

ANKARA YILDIRIM BEYAZIT UNIVERSITY

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**INVESTIGATION OF PITTING CORROSION MORPHOLOGY OF
ST 37 MATERIAL IN DIFFERENT CORROSIVE ENVIRONMENTS**

M.Sc. Thesis by

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April, 2018

ANKARA

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CORROSIVE ENVIRONMENTS**

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M.Sc. THESIS EXAMINATION RESULT FORM

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INVESTIGATION OF PITTING CORROSION MORPHOLOGY OF ST 37 MATERIAL IN DIFFERENT CORROSIVE ENVIRONMENTS

ABSTRACT

According to the terminology, corrosion is defined as “the chemical or electrochemical reaction between a material, usually a metal, and its environment that produces a deterioration of material and its properties.” Corrosion also produces pits or holes in the surface of reinforcing steel. These pits reduce the strength of the steel. If corrosion is not controlled, it can affect the integrity of the structure. Steel in concrete is generally not worn in passive conditions. However, steel- reinforced concrete is often used in severe environments where sea water or deicing salts are present. When chloride moves into the concrete, it disrupts the passive layer protecting the steel, causing it to rust and pit. Pitting corrosion of metals is one of the most important electrochemical corrosion phenomena. At the site of the pitting, where the metal is unprotected, corrosion will develop if the pit does not re-passivate.

Generally steel material known as St 37, is preferred in reinforced concrete and St 37 steel is exposed to pitting corrosion due to using in steel salt water. In this study, pitting morphology of St 37 material was investigated at different sodium chloride solution concentrations (1.8 percent and 3.5 percent). At the same time, the microstructures of the steel and the pit associations were also investigated as part of the study. The main objective of this thesis is to observe pit morphology and pit growth and also to observe the effect of corrosion to the mechanical properties of St 37 steel.

The specimens immersed to the 1.8 percent sodium chloride solution showed lower strength than the specimens put to the 3.5 percent sodium chloride solution as unexpected, but did not cause any significant change in elongation values. The value of mass loss formed by the corrosion result is too low. In the prepared solution there

was no significant change in the pH measurements before and after corrosion which are shown that the reason is based on the pH value in short period.

Keywords : Pit corrosion, Passive Film, St 37, SEM



ST 37 MALZEMENİN FARKLI KORUZİF ORTAMLARDA ÇUKUR MORFOLOJİSİNİN İNCELENMESİ

ÖZ

Terminolojiye göre korozyon “bir malzeme ile genellikle bir metal ile çevresi arasında gerçekleşen, madde ve özelliklerinde bozulmaya neden olan kimyasal ya da elektrokimyasal bir reaksiyon” olarak tanımlanır. Korozyon takviye çelik yüzeyinde çukur ve delikler oluşmasına sebep olur. Oluşan bu çukurlar çeliğin mukavemetini azaltır. Eğer korozyon kontrol altında tutulmazsa yapının bütünlüğünü etkileyebilir. Beton içindeki çelik pasif durumlarda genellikle aşınmaz. Ancak çelik takviyeli betonlar genellikle deniz suyu veya buz çözücü tuzlar gibi ağır ortamlarda kullanılır. Klorür beton içine taşındığında, koruyucu pasif tabakayı bozar ve pas ve çukura neden olur. Çukur korozyonu metallerde en önemli elektrokimyasal korozyonlardan biridir. Metalin korunmadığı çukurlaşma yerinde çukur tekrar pasif hale getirilmezse korozyon gelişecektir.

Takviyeli betonlarda kullanılan donatılarda genellikle St 37 olarak bilinen çelik malzeme tercih edilmektedir ve tuzlu suda kullanılması nedeniyle çukur korozyonuna maruz kalmaktadır. Bu çalışmada St 37 malzemenin farklı sodyum klorür çözeltileri derişimlerdeki (yüzde 1.8 ve yüzde 3.5) çukur morfolojisi incelenmiştir. Aynı zamanda çeliğin mikro yapıları ve çukur ilişkileri de çalışmanın bir parçası olarak araştırılmıştır. Bu tezin temel amacı, çukur morfolojisi ve çukur büyümesini gözlemek ayrıca korozyonun St 37 çeliğinin mekanik özelliklerine etkisini gözlemlemektir.

Yüzde 1.8 sodyum klorür çözeltilerine daldırılan numuneler, beklenmedik bir şekilde yüzde 3.5 sodyum klorür çözeltilerine konan örneklerden daha düşük bir mukavemet gösterdi, ancak yüzde uzama değerlerinde anlamlı bir değişikliğe neden olmadı. Korozyon sonucu meydana gelen kütle kaybı göz ardı edilecek kadar düşüktür. Hazırlanan çözeltilerde korozyondan önce ve korozyondan sonra yapılan pH

ölçümlerinde önemli bir deęişiklik olmamıştır, bunun sebebinin kısa periyotlarda alınan pH deęerleri olduęu düşünölmektedir.

Anahtar sözcükler: Çukur Korozyonu, Pasif Film, St 37, SEM



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NOMENCLATURE

Abbreviations

μm	Micrometer
MPa	Mega Pascal
N	Newton
nm	Nanometer
rpm	Rounds per minute
E	Elastic Modulus
UTS	Ultimate Tensile Strength

Acronyms

SCC	Stress Corrosion Cracking
CPT	Critical Pitting Temperature
ASTM	American Society for the Testing of Materials
SEM	Scanning Electron Microscope

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CHAPTER 1

INTRODUCTION

1.1 Corrosion

Corrosion is known generally as rust and it is as old as the earth. Mostly it destroys the brightness, and beauty of objects and shortens their service life [1-2].

Corrosion is mainly used to describe the loss of metallic properties by reacting with the environment of the metals. Corrosion is very important for the country's economy. Each year, a quarter of annual steel production is caused a loss. Urban air, rain water and seawater are the main factors of corrosion. Corrosion commonly occurs in metal materials because of their high tendency to electrochemical reactions [3].

Several definitions of corrosion have been given below:

- Corrosion is the surface wastage that occurs when metals are exposed to reactive environments.
- Corrosion is the result of interaction between a metal and environments which results in its gradual destruction.
- Corrosion is an aspect of the decay of materials by chemical or biological agents.
- Corrosion is an extractive metallurgy in reverse. For instance, iron is made from hematite by heating with carbon. Iron corrodes and reverts to rust, thus completing its life cycle.
- Corrosion is the deterioration of materials as a result of reaction with its environment.
- Corrosion is the destructive attack of a metal by chemical or electrochemical reaction with the environment [2].

Although different description, it can be observed that corrosion basically happens at different rates with different metals and in different environments [2].

If we expose metals such as iron or steel to air and water we can expect to see rust form in a short time. The color of red-brown iron oxide occurs on the surface of metals and the rust may develop in minutes. Corrosion takes place with other metals such as copper, brass, zinc, aluminum, and stainless steel, but it might take longer to develop. However, nonmetals are not included in this definition of corrosion such as ceramics, polymers, composites and semiconductors [4].

Corrosion involves chemical change, so principles of chemistry are very important to understand corrosion reactions. Understanding of electrochemistry is also significant because corrosion processes are most often electrochemical [4]. Essentially all corrosion reactions are electrochemical in nature. Electrochemical corrosion occurs due to the existence of separate ‘anodic’ and ‘cathodic’ areas / peaks between which current flows through the conducting solution [5]. One is the anodic reaction, in which iron atoms are ionized and go into solution, leaving their electrons within the original metal surface. The other is cathodic reaction, in which the free electrons within the metal are taken up by chemical species such as O_2 and H_2O in reduction reactions [6].

Hence, the overall corrosion reaction is;



The above reaction can be divided in to two partial reactions:

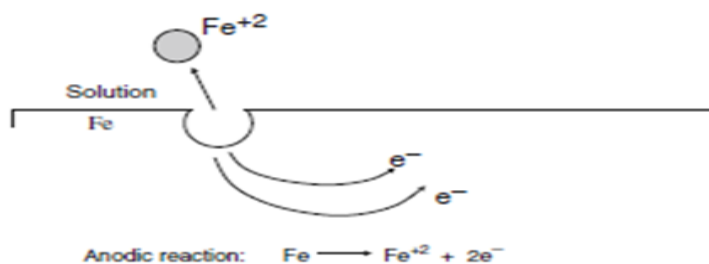
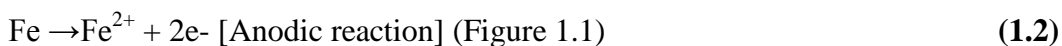


Figure 1.1 Anodic Reaction [3].

The reaction in equation (1.2) is an anodic reaction because of the following:

- A given species undergoes oxidation, i.e., there is an increase in its oxidation number.
- There is a loss of electrons at the anodic site (electrons are produced by the reaction) [7].

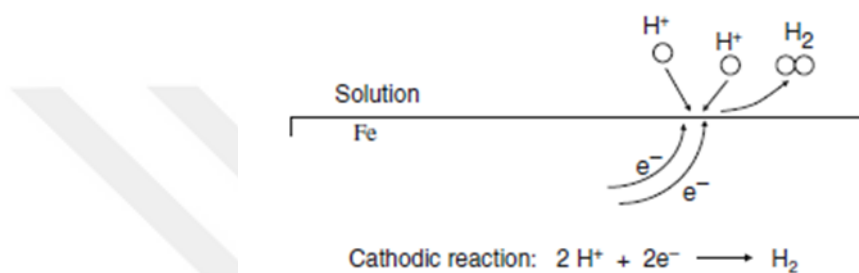
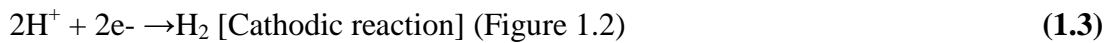


Figure 1.2 Cathodic Reaction [3].

The reaction in equation (1.3) is a cathodic reaction because of the following:

- A given species undergoes reduction, i.e., there is a decrease in its oxidation number.
- There is a gain of electrons at the cathodic site (electrons are consumed by the reaction) [7].

The destruction of metal by electrochemical reactions causes many effects on the safe, reliable and efficient operation of equipment or structures. Although the amount of metal destroyed is quite small, Failures of various kinds and the need for expensive replacements may occur [6].

Some of the important destructive effects of corrosion can be summarized as follows:

1. Reduction of metal thickness leading to loss of mechanical strength [6].

2. When the metal is lost in localized zones, very significant structural failure or breakdown occurs.
3. Structural failure or breakdown causes some hazards or injuries for people (e.g. bridges, cars and aircraft) [6].
4. Corrosion affects the global supply of metals by removing components or structures from service so that their replacement consumes a portion of the total supply of the earth's material resources [7].
5. Goods of value reduced because of breakdown of appearance.
6. Possible harm to the surroundings occurs when vessels and pipes have important perforation.
7. Loss of technically important surface properties of a metallic component.
8. Mechanical damage to valves, pumps, etc, or blockage of pipes by solid corrosion products.
9. Reducing of the service life of a metal product or component causes additional manufacturing or processing, thus emissions of greenhouse gases increase [6-7].

1.2 Classification of Corrosion

Corrosion can be classified into direct chemical (dry) corrosion and electrochemical (wet) corrosion. Dry corrosion occurs when there is no water or moisture to aid the corrosion, and the metal oxides with the atmosphere alone. Wet corrosion of metals occurs through electron transfer, involving two processes, oxidation and reduction. In oxidation, the metal atoms lose electrons, then the surrounding environment gains the electrons in reduction. The metal, where electrons are lost, is called the anode. The other metal, liquid or gas which gains the electrons is called the cathode. [8-10]. Corrosion is based on the material, environment, or the morphology of the corrosion damage. Based on morphology of the damage, corrosion can be classified into 14 different modes of attack:

a) Uniform Corrosion

This is also called general corrosion. Uniform corrosion is the most common type of corrosion and is caused by a chemical or electrochemical reaction that results in the deterioration of the entire exposed surface of a metal (Figure 1.3). Uniform etching of metal occurs when the chemical directly attacks on metal surfaces.

The use of chemical-resistant protective coatings or more resistant materials will control this type of corrosion [5].



Figure 1.3 Uniform Corrosion [5].

General attack corrosion causes for the vast amount of metal destruction by corrosion. Due to the fact that it is foreseeable, manageable and often preventable. It is considered as a safe form of corrosion [10].

b) Galvanic Corrosion

Galvanic corrosion is an electrochemical action of two different metals, they are located together in a corrosive electrolyte. It occurs when dissimilar metals are in contact (Figure 1.4) [5]. In this process, the more active metal (with more negative electrode potential) acts as an anode while the less active metal (with less negative

electrode potential) acts as cathode [11]. Control of galvanic corrosion is achieved by using metals closer to each other in the galvanic series [5].

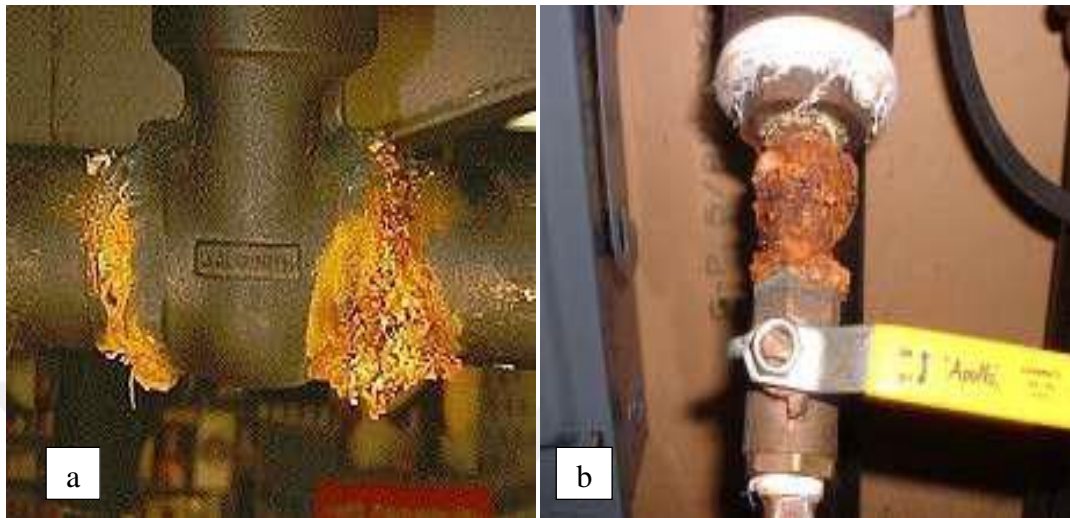


Figure 1.4 Galvanic corrosion in pipe joints (a and b) [5].

c) Concentration Cell Corrosion

Concentration cell corrosion occurs when two or more areas of a metal surface are in contact with different concentrations of the same solution. Concentration cell corrosion is involved with gaskets, joints, scale, debris, loose protective films, etc [5].

When the oxygen concentration is least, corrosion attack is accelerated. Low oxygen availability at the area of metal becomes anodic to other areas. The cathodic area is large compared to the anodic area so the intensity of attack is usually more severe on surrounding areas of the same surface [5].

d) Pitting Corrosion

Pitting corrosion occurs in materials that have protective films and corrosion attacks with localized holes on the metal's surface so it can penetrate the metal very rapidly [5].

Pitting results small holes, or cavity on the metal surface, and generally small holes can spread very quickly (Figure 1.5) [12]. Then the holes lead to unexpected

breakdown and destructive system failure in a very short period of time [13]. Sometimes pitting corrosion can be quite small on the surface but it can be very large below the surface [5].

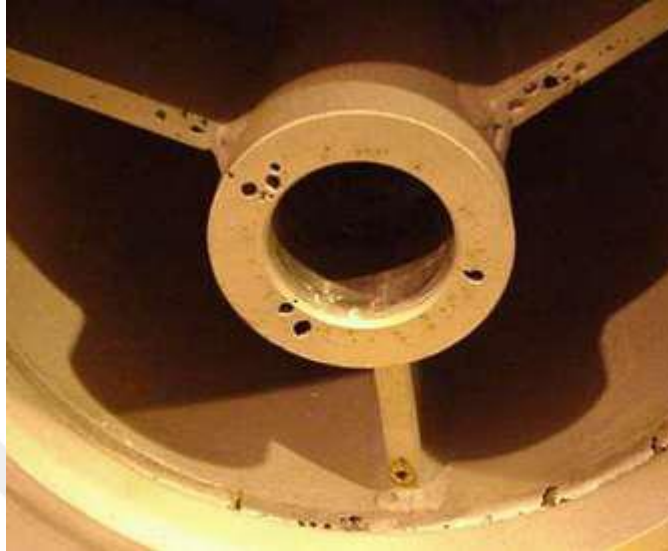


Figure 1.5 The 316L type stainless steel pump, there is a local pitting corrosion [5].

When the solution on the metal surface contains chloride, hypochlorite or bromide ions, pitting is vigorous. Generally stainless steel, chromium, passive iron, cobalt, aluminum, copper and associated alloys are all prone to pitting corrosion [13].

Pitting corrosion can be controlled by maintaining clean surfaces, application of a protective coating, and using of inhibitors or cathodic protection [5].

e) Crevice Corrosion

Crevice or contact corrosion is the corrosion produced at the region of contact of between metallic and non-metallic material (Figure 1.6). It may occur at washers, under barnacles, at sand grains, under applied protective films, and at pockets formed by threaded joints. Cleanliness, the proper use of sealants and protective coatings can also be used to control corrosion effects [5-11].



Figure 1.6 It is a corrosion that occurs on the six covered surfaces such as rivets, bolts, gaskets and in intervals [11].

f) Filiform Corrosion

It is a corrosion phenomenon that runs under the paint or coating layer on the metal surface (Figure 1.7). Filiform corrosion is seen when the air humidity is between 78-90% and the surface is slightly acidic. Protective coating of metal surfaces can deteriorate when long branching filaments of corrosion product extend out from the original corrosion pit. Filiform corrosion is minimized by coating surface carefully [5].

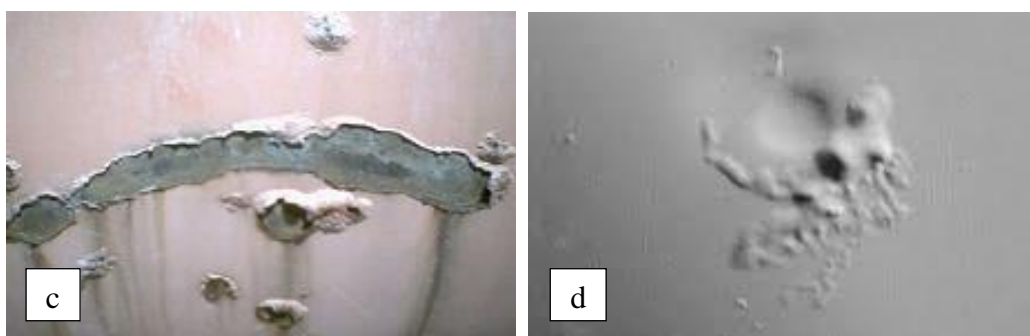


Figure 1.7 Filiform corrosion occurs under the paint or coating layer on the metal surface (c and d) [5].

g) Intergranular Corrosion

Intergranular corrosion is the selective attack on or close to the grain boundaries of a metal or alloy [5]. Intergranular corrosion is usually seen in austenitic stainless steels, Al-Cu and Ni-Cr alloys. Intergranular corrosion is the most dangerous type of corrosion because of not being determined by naked eyes [13]. The type of damage occurs only at grain boundaries beneath the surface and it causes to exfoliate of metal surfaces (Figure 1.8) [5].

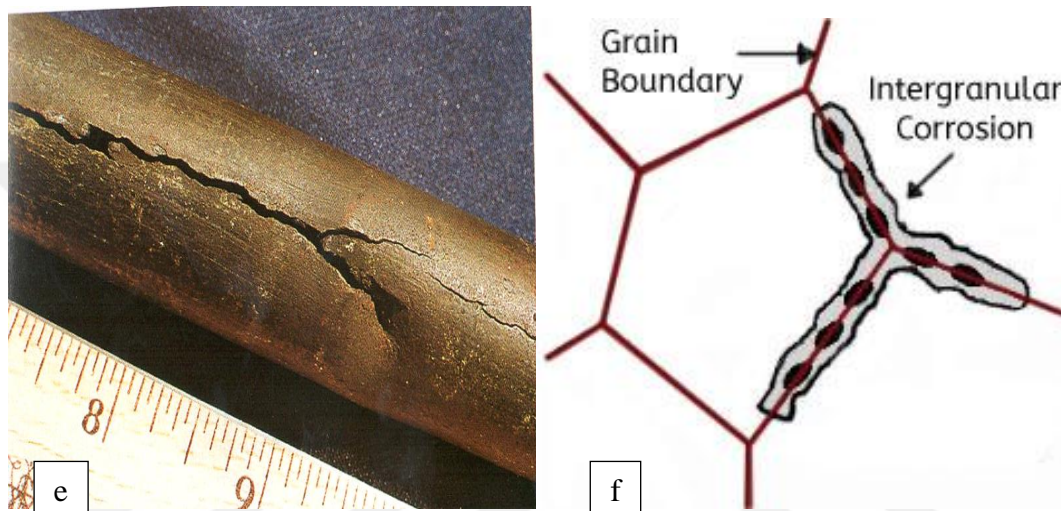


Figure 1.8 Intergranular Corrosion (e and f) [13].

h) Stress Corrosion Cracking

Stress corrosion is a special kind of intergranular corrosion. Stress corrosion cracking (SCC) is caused by the simultaneous presence of tensile stress and a specific corrosive environment [13].

Stress corrosion occurs only after tensile stresses. For example stress corrosion is seen between riveted surfaces in airplanes. It can be avoided by using appropriate heat treatment, selecting the proper alloy for a given environment, putting the equipment in service in a stress free condition, or using protective coatings [5].

i) Corrosion Fatigue

Corrosion fatigue occurs by the combined dynamic load and corrosive environment. Materials under dynamic loads can crack when the influence of smaller stresses. This type of corrosion is found especially on train wheels. Control of corrosion fatigue can be accomplished by either lowering the cyclic stresses or by corrosion control using protection methods [5].

j) Fretting Corrosion

Fretting corrosion that occurs at the interface between two surfaces in contact by a stick-slip action causing breakdown of protective films or welding of the contact areas that allows other corrosion mechanisms to operate [10].

k) Erosion Corrosion

Erosion corrosion is a result of a combination of an aggressive chemical environment and high fluid-surface velocities [5]. Metal is removed from the surface as dissolved ions and corrosion products are mechanically swept away from the metal surface (Figure 1.9)[13]. Erosion corrosion can be prevented by using a more corrosion resistant alloy [7].



Figure 1.9 Erosion Corrosion in Solder Joints [13].

l) Dealloying

When the alloy loses the active component of the metal, dealloying occurs. A corrosive medium specifically attacks one component of the alloy. Control is achieved by the use of more resistant alloys-inhibited brasses and malleable or nodular cast iron [5-13].

m) Hydrogen Damage

Corrosion reactions can result in hydrogen atoms on the surface of the metal. They are adsorbed on the metal surface and these hydrogen atoms turn into molecules causing a large volume increase. Therefore hydrogen molecules create a large pressure in the metal cavities and this pressure causes the metal to crack [13]. Hydrogen embrittlement is a problem especially on high-strength steels, titanium, and some other metals. Hydrogen damage is controlled by eliminating hydrogen from the environment or by the use of resistant alloys [10].

n) Microbial Corrosion

During the development of microorganisms, acids and sulfides occur. These components increase the corrosion rate [5]. Microbiological corrosion is very common in crude oil storage tanks, sewage and sludge, cooling water systems, especially in stagnant regions [13].

CHAPTER 2

PITTING CORROSION

2.1 Introduction to Pitting Corrosion

Passive films are very useful for many engineering alloys such as aluminum alloys and stainless steel. The passive films are thin oxide layers that form naturally on the metal surface and greatly decrease the rate of corrosion of the alloys. However, such passive films are often sensitive to localized breakdown resulting in accelerated dissolution of the underlying metal. Especially chloride ions cause that breakdown of a passive film [14]. The chloride ion has a special significance in pitting corrosion. Chloride ions especially exist in seawater, brackish waters, de-icing salts, and airborne salts [7]. The chloride ions causes the attack confined to a small fixed area of the metal surface, it is called pitting corrosion and this mechanism is illustrated in Figure 2.1 [14-10].

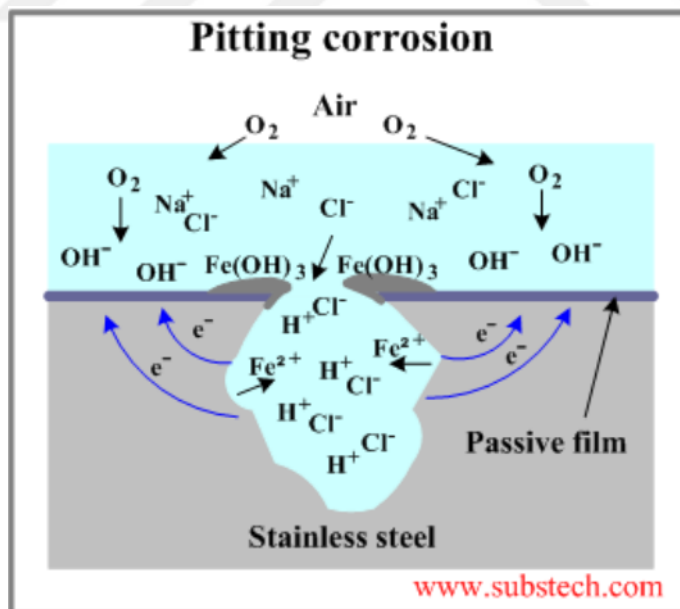


Figure 2.1 Pitting Mechanism [10].

Pitting corrosion generally can be described as localized corrosion process that results in the formation of microscopic holes in metallic alloys. Corrosion pits form

at microscopic sizes. If there are defects, scratches, breakages and cracks on the passive film, pit formation especially occurs in these regions [15-16].

Pitting occurs when an electrolyte begins to allow the transport of ions between an anode and cathode. Only a small amount of metal is corroded and this is usually referred to as localized attack [17]. The corrosion propagates penetrating the metal insidiously thus pitting starts at the surface in different forms and sizes depending on the metallurgical property of the alloy and different forms of pits are illustrated in Figure 2.2 [15].

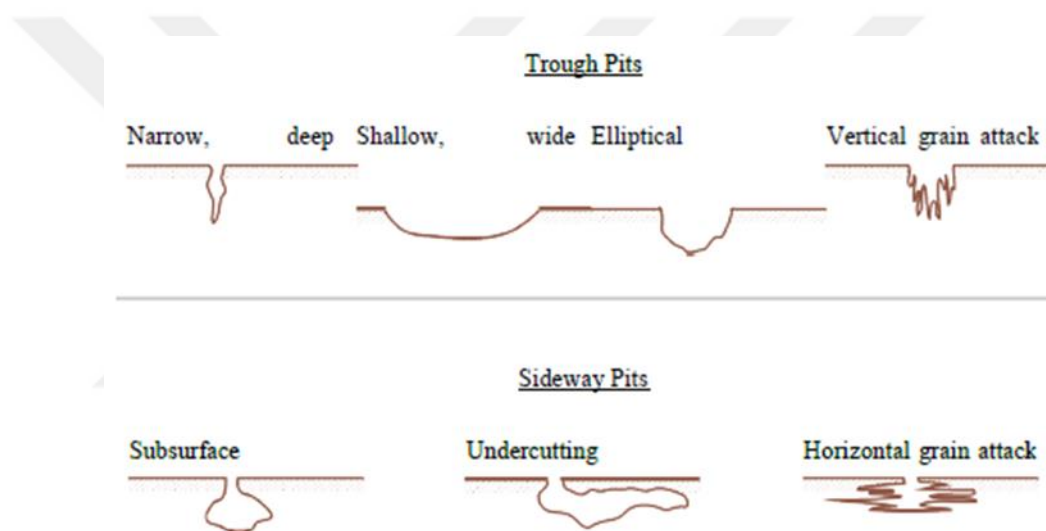


Figure 2.2 Types of Pitting Corrosion [15].

Pitting is a dangerous form of corrosion attack for several reasons:

- Pits can result in the perforation of a metal surface [7],
- Pits cause material loss and economic loss [16],
- It is more difficult to detect and it's harder to get rid of.

An unimportant small pit cause the complete mechanic structure damage. The metal loss is minimal but it can lead to the failure of an engineering system [15].

2.2 Stages of Pitting

Pitting can be considered of two stages: pit initiation and pit growth [15]. Each of these stages is very important. Once the passive film breaks down and a pit initiates, easily there is a stable pit will grow [18].

2.2.1. Pit Initiation

Many corrosion scientists have studied the processes of breakdown of passivity. The mechanism of pit initiation and the breakdown of the passive film are not known with great certainty. The passive film is blocking entry of the environment to the metal. When aggressive chemicals such as chlorides remove through the oxide film, particularly damaging occurs on the metal surface. The breakdown of passive layer is the reason for the initiation of pitting corrosion. As a result, anodic reaction starts on the metal surface and passivated metal surface behave as the cathode [16].

According to some researchers these particles precipitate along the grains boundaries and serve as local anodes so formation of initial pits occur on the metal surface [15]. Pitting mechanism is illustrated in Figure 2.3.

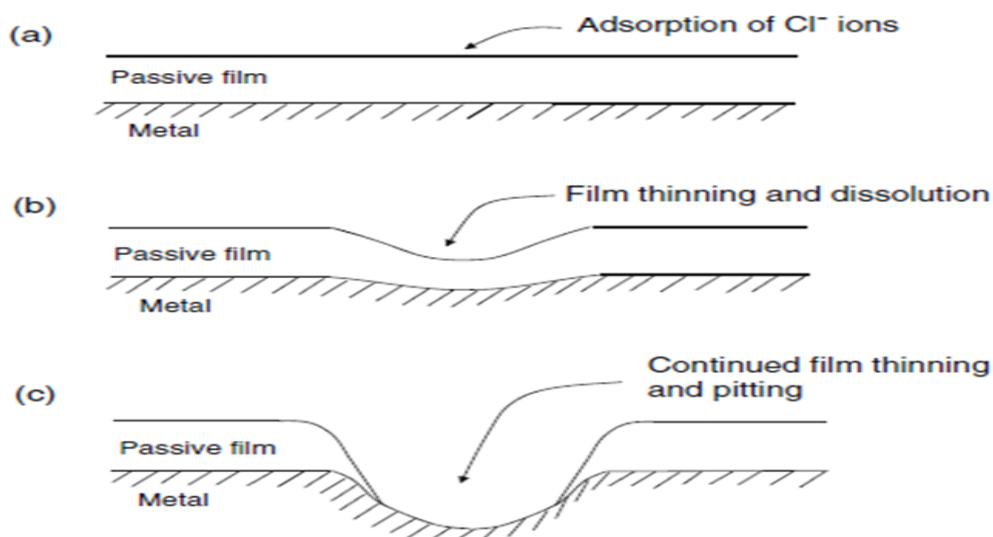


Figure 2.3 The film thinning mechanism of pitting in which chloride adsorption (a) initiates the process of film thinning (b) leading to pitting (c) [7].

2.2.2. Pit Growth

The growth of corrosion pits is depends on some factors such as the composition of the alloy, electrolytic composition and acidity (rate of pH) and potential within the pit. Pits often develop with a porous cover. However, this cover is extremely difficult to notice so that the awareness of the severity of attack can be overlooked. The electrolytic diffusion phenomenon within the pit is strongly associated with the kinetics of the growth process [16-18].

Pit growth is relevant with

- the formation of a highly corrosive internal electrolyte which is acidic,
- concentration of chloride ions,
- dissolved cations of the metal or alloy [7].

CHAPTER 3

St 37 STEEL AND CORROSION

3.1 Introduction

St 37 steel is very popular and has good ductile properties as well as excellent weldability. Hundreds of tons of St 37 steel are used in numerous applications: for example, the fabrication of water vessels and shelters (Hangars). St 37 steel is produced in many shapes and thickness such as steel plates, sheets, strips, angles and different other geometric shapes [19].

St 37 is a hot rolled non-alloy low carbon structural steel. This material is intended for structural purposes where mechanical test values are required. The calculated mass of this steel is determined using a volumetric mass of 7.85 kg/dm^3 .

Steel-reinforced concrete composites are the most commonly used structural materials due to the low cost and availability of materials. The most important parts of these composite systems are reinforced steel rebars because of their load carrying capabilities. The steel is selected from structural steels and the most used one is St 37. The mechanical properties of steel rebars are strong, but the service problems influence negatively to the permanency of them. For example, due to the wear of concrete, steel rebars may contact with air and they can corrode. Similarly, storing of steel rebars for a long time may result corrosion before servicing of them. According to the ASTM, chemical or electrochemical reaction between a metal and its environment produces a deterioration of material and its properties and this is called corrosion [20]. Pitting corrosion is localized accelerated dissolution of the metal which occurs as result of a breakdown of the protective passive film on the metal surface. The metal is not corroded uniformly, but deep pits are produced and these pits reduce the surface area and enhance the stress concentration. The most destructive environments for the pitting corrosion of the structural steels are coastal

areas because the structures are subjected to extreme exposure to saline water and this results in intrinsic tensions and crackings [14-22]. However, due to the high alkaline properties and passive film layer on metal surface, reinforced steel can bear to the corrosion for a long time. Furthermore, a well pressed concrete has lower permeability and the compounds which are the reason of the corrosion (oxygen, water and chloride) cannot penetrate into the concrete and so corrosive matters cannot reach to the rebars.

3.2 Relationship Between St 37 Steel and Corrosion

The corrosion pits decrease the effective area of the surface that sustain the loads and due to the stress concentration at the bottom of the pits, the cracks will possibly form from these regions. The volume of the corroded rebar expands and makes the reason for the parallel cracks on the rebars inside the concrete. Once these cracks and voids form, concrete is more exposed to atmospheric conditions and the structural member complete its life very rapidly [23-25].

The most important reason of the corrosion of the steel rebar is the penetration of the chloride ions to the concrete. The chloride ions can penetrate to the concrete with the salt inside the sea water or the salt that concrete contains. Although the material loss is very low comparing to the other homogenous corrosion types, the parts of the material may become unusable very rapidly. [19-26]. Pitting corrosion is one of the most dangerous detrimental mechanisms for the reinforcing rebar because it is widespread, difficult to control and it has degrading effects in the material. For these reasons, the pitting corrosion behavior of St 37 is investigated with respect to the salinity of the Black Sea and the Mediterranean Sea to observe the influence of salt to the reinforcing rebars of buildings used in these regions.

3.3 Chemical Compound (According to ASTM) of St 37 Steel

The chemical composition of St 37 is presented in Table 3.1 and the product analysis were given in % weight.

Table 3.1 Chemical Composition of St 37 Steel (BS EN 10025, 1993) [19].

Analysis	C % max for nominal product thickness t			Mn % max	Si % max	P % max	S % max	N % max
	t ≤ 16 mm	16 < t ≤ 40 mm	t > 40 mm					
Product analysis	0.21	0.25	-	1.5	-	0.055	0.055	0.011

3.4 Mechanical Properties of St 37 Steel

The mechanical properties of St 37 are presented in Table 3.2 as the minimum values required for the yield strength, tensile strength as well as for the elongation. These values are also classified according to different thickness of steel sheets.

St 37 is constructional steel having maximum 0.17 % carbon ratio, and its fracture strength is in between 360-510 MPa. The steels were commercially available and obtained from a local company in the form of sheet. The carbon content of St 37 is higher than those of stainless steels, 0.17%, but it is still considered as low carbon steel. It is used to increase the mechanical properties of rebar material in composite concrete systems.

Table 3.2 Mechanical Properties of St 37 Steel [19].

Minimum yield strength R_{eH} [MPa] nominal thickness t [mm]		Minimum tensile strength R_m [MPa] nominal thickness t [mm]		Minimum percent elongation [%]					
				$L_0 = 80$ mm				$L_0 = 5.65\sqrt{S_0}$	
				Nominal thickness t [mm]					
t ≤ 16	16 < t ≤ 40	t < 3	3 ≤ t ≤ 100	t ≤ 1	1 < t ≤ 1.5	1.5 < t ≤ 2	2 < t ≤ 2.5	2.5 < t < 3	3 ≤ t ≤ 40
235	225	360-510	340-470	17	18	19	20	21	26

3.5 Mechanical Testing

Mechanical testing plays an important role in evaluating fundamental properties of engineering materials as well as in developing new materials and in controlling the quality of materials for use in design and construction.

The results of tensile tests are used in selecting materials for engineering applications. Tensile properties often are measured during development of new materials and processes, so that different materials or processes can be compared. Tensile properties often are used to predict the behavior of a material under forms of loading other than uniaxial tension. The major parameters that describe the stress-strain curve obtained during the tension test are the tensile strength (UTS), yield strength or yield point (σ_y), elastic modulus (E) and percent elongation ($\Delta L\%$). Universal tensile test machine was used which had 2500 N capacity and 0.1 N precision. A tensile test involves mounting the specimen in a machine and subjecting it to tension. A tensile load was applied to the specimens until it fractured. During the test, the load required to make a certain elongation on the material was recorded. A load elongation curve was plotted by an x-y recorder, so that the tensile behavior of the material could be obtained. An engineering stress-strain curve can be constructed from this load-elongation curve by making the required calculations. The tensile force is recorded as a function of the increase in gage length.

Engineering stress, or nominal stress, σ , is defined as

$$\sigma = P/A_0 \quad (3.1)$$

where P is the tensile force and A_0 is the initial cross-sectional area of the gage section.

Engineering strain, or nominal strain, e , is defined as

$$e = \Delta L/L_0 \quad (3.2)$$

where L_0 is the initial gage length and ΔL is the change in gage length ($L - L_0$).

Tensile Strength is the maximum stress that the material can support, is defined as

$$\sigma_{UTS} = P_{max}/A_0 \quad (3.3)$$

where σ_{UTS} is the maximum point of the stress-strain diagram, necking starts.

When force-elongation data are converted to engineering stress and strain, a stress-strain curve for ductile materials like St 37 (Figure 3.1) that is identical in shape to the force-elongation curve can be plotted [27].

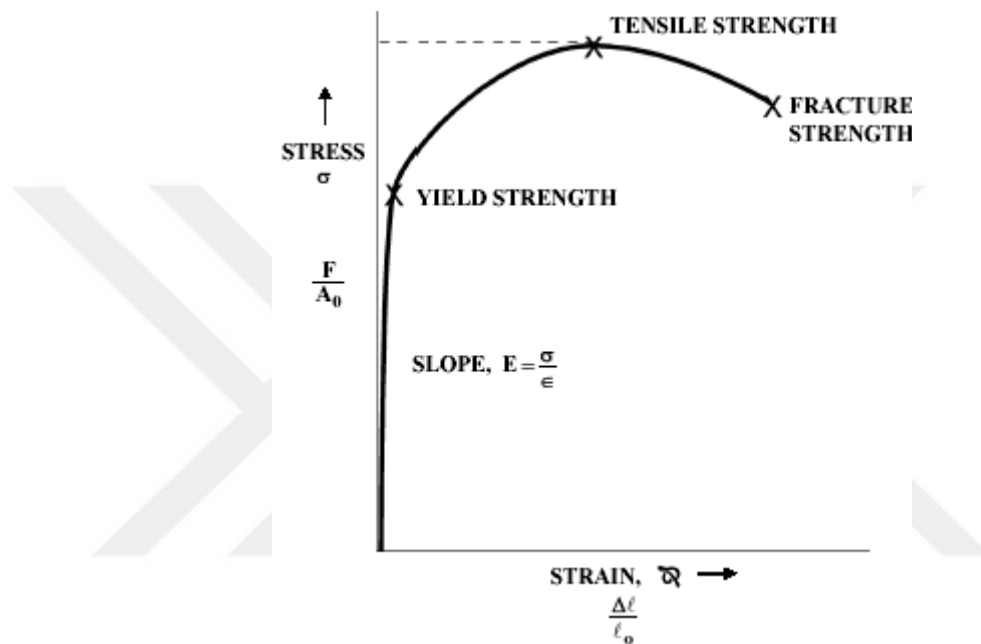


Figure 3.1 Corresponding Engineering Stress-Strain Curve [27].

Stress-strain curves and elongation (%) were measured during the test. Figure 3.2 shows the typical engineering stress-strain behavior [27].

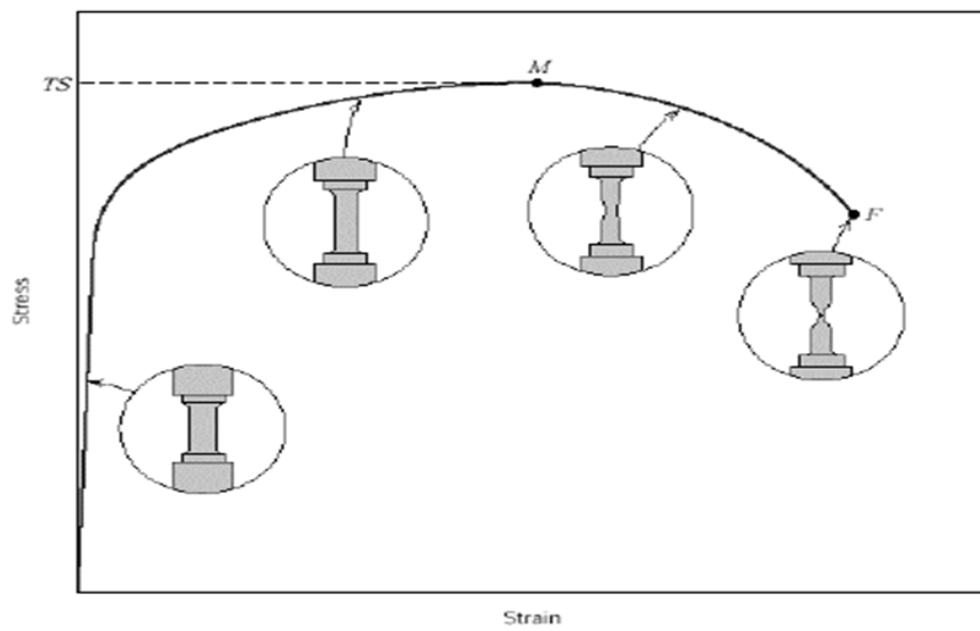


Figure 3.2 Typical Engineering Stress-Strain Behavior to Fracture, Point F. The Tensile Strength is Indicated at Point M. The circular insets represent the geometry of the deformed specimen at various points along the curve [27].

CHAPTER 4

EXPERIMENTAL PROCEDURE

4.1 Materials

In this study, specimens shown in Figure 4.1 were used. The specimens were machined in laser cutting machine factory. St 37 sheet metal was obtained and cut according to the ASTM standards as dog bone specimen shape. Dog bone specimens were used in this research because of the combined effect of stress concentration close to center and the pitting corrosion.

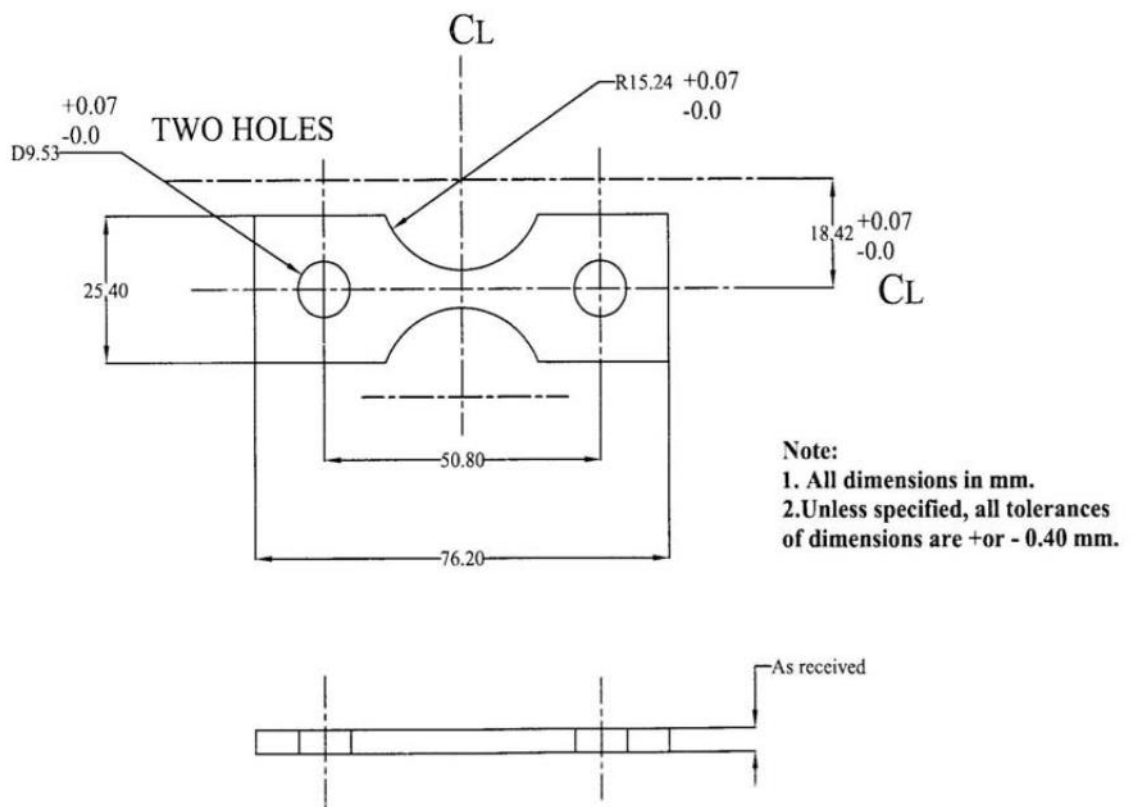


Figure 4.1 Specimen Used in This Study. Adapted from [28].

4.2 Methods

In the scope of this research project, some specimens are subjected to NaCl solutions with different concentration levels and some pristine specimens are used for comparison. The solutions used in this study include 1.8 % NaCl and 3.5 % NaCl concentration. The 1.8 % NaCl concentration represents the salinity of Black Sea and the 3.5 % NaCl concentration represents the salinity of Mediterranean Sea. After specimens immersed to the NaCl solutions, the specimens are taken out to investigate pitting morphology of them in optical microscope and SEM. Then, tensile tests are applied to compare the strength of the corroded specimens with the pristine specimens. Fractured specimens are observed under SEM to detect the crack initiation points and elemental analysis (EDS) is used to identify the particular elements on the surface and fractured points.

4.2.1 Preparation of Solutions

First a solution of 3.5% NaCl was prepared using 100 mL of distilled water with 3.5 g of NaCl. This solution was stored in two different beakers for two different specimens. In order to observe corrosion behavior of St 37 steels in Mediterranean Sea, they were put into 3.5% NaCl solution. Another solution of 1.8% NaCl was prepared using 100 mL of distilled water with 1.8 g of NaCl. Corrosion behavior of St 37 steels in Black Sea were investigated in 1.8% NaCl solution.

Pits on the specimen's surfaces were not observed approximately 24 hours therefore to induce the corrosion pitting and keeping the time shorter, experiments were performed by adding H₂O₂ to the NaCl solutions. The solutions with 1.8% NaCl and 3.5% NaCl were prepared using distilled water with 30 % H₂O₂. Figure 4.2 shows those specimens in solution.

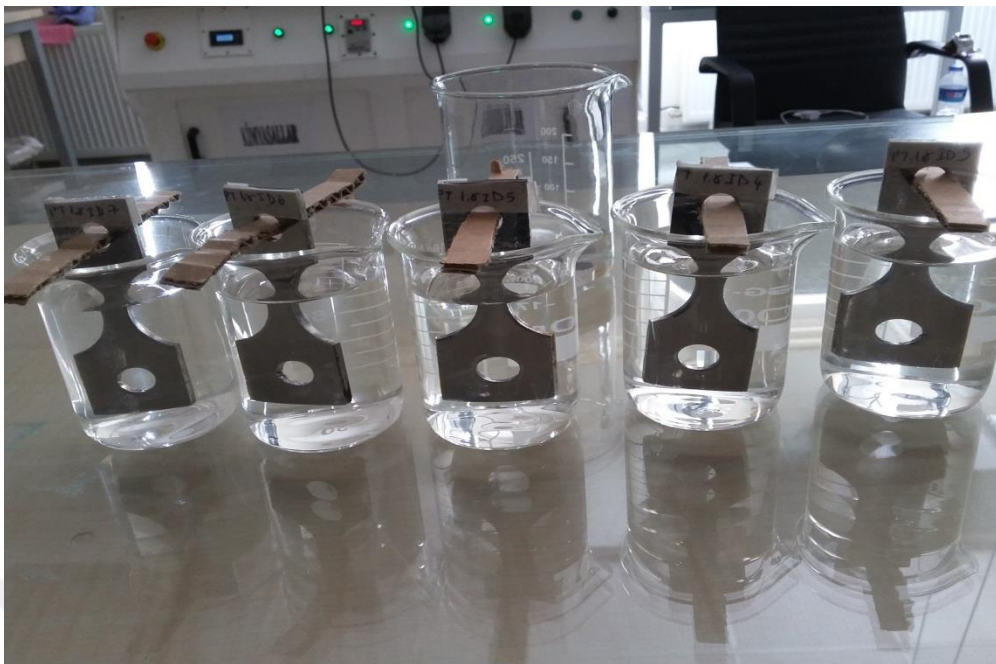


Figure 4.2 Specimens in Solution

Two solutions were prepared to immerse twelve specimens in different beakers, six of them with 3.5 % NaCl- 13 mL of 30 % H₂O₂ and the other six with 1.8 % NaCl - 13 mL of 30 % H₂O₂. Two of them were used to perform pilot experiments.

4.2.2 Preparation of Specimens

In this study, specimens shown in Figure 4.1 were used. After the specimens were machined in accordance with the drawing in Figure 4.1, both sides of the specimens were polished according to the procedure shown in Figure 4.3. Firstly the specimens were polished with abrasive grit size 120 paper. But it was very difficult to clean the surface of the samples. So it was decided to use thicker sandpaper which is grit size 80. Then the sharp corners of the specimens were polished with abrasive grit size 1000 paper. Polishing machine used in the experiments is shown in Figure 4.4. After this step only one side of the specimens was polished to 1 micron and 0.3 micron finish.

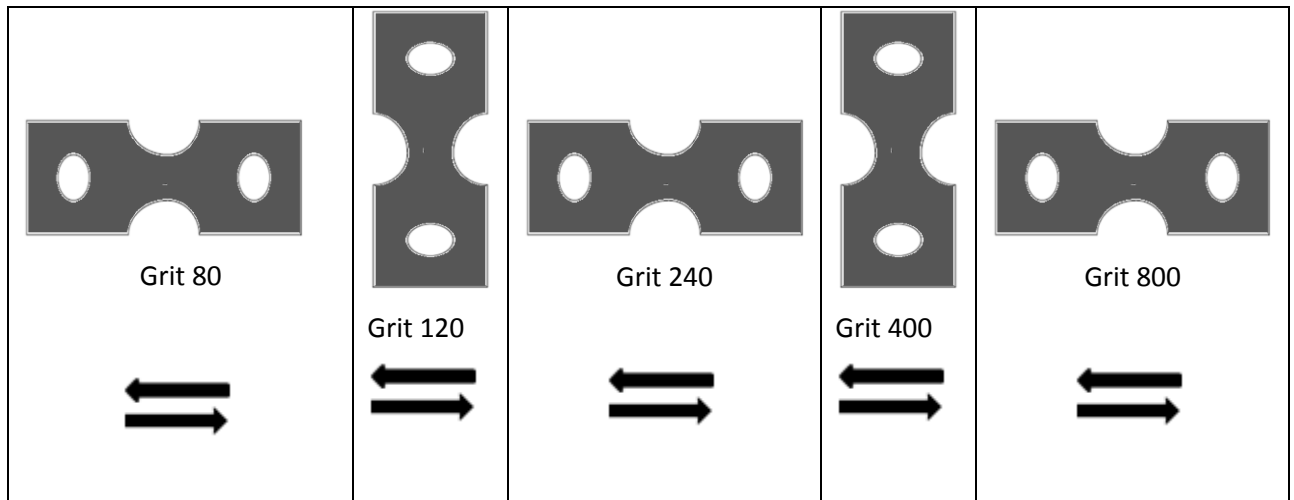


Figure 4.3 Schematic View of the Specimen Polishing with the 80, 120, 240, 400, 800 Grit Sandpaper.



Figure 4.4 Polishing Machine

Scratches, discontinuities, surface faults and residues, etc. were minimized by polishing. In this way, polishing process decreases the probability of the specimen deformity during testing.

After that, they were cleaned with the acetone in the ultrasonic cleaner and designated with ID numbers. Figure 4.5 shows that ultrasonic cleaner. In Figure 4.6

(a) and Figure 4.6 (b), specimens treated by ultrasonic bath and the designations of the specimens with the ID numbers are illustrated respectively.



Figure 4.5 Ultrasonic Cleaner



Figure 4.6 (a) Sanded and Ultrasonic Bathed Specimens (b) ID Numbered Specimens For 3.5 % Salinity Solution

In advanced work the area to be corroded was an area in the center of the specimen 1.5 mm x 1 mm in dimension across the width of the specimen. But this area was very large to examine pitting corrosion. So it was decided the area to be corroded was an area in the center of the specimen 1 mm x 1 mm in dimension across the width of the specimen. This area should be the narrowest section of the dog bone, as

shown in Figure 4.7. Before a specimen was put into the solution, the specimen was painted by fingernail polish except the area at the center of the dog bone, which is an area of 1 mm x 1mm. The reason is to have the corrosion only at the center for examining easily. Fingernail polish was chosen to protect the rest of the area as it contained no metallic substances that could affect the ability to protect the covered surface. Polish was applied three layers with 20 minutes drying periods. Then the samples were stored in a dry environment until tested. Drying of the specimens is illustrated in Figure 4.8. (a and b)

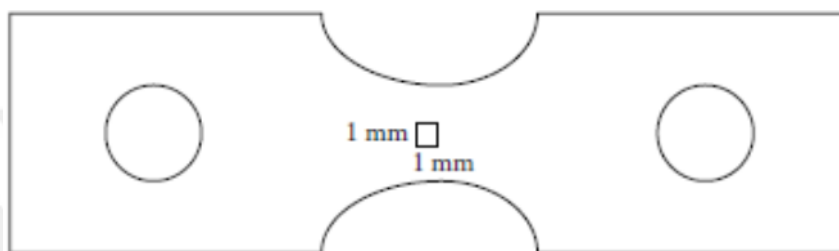


Figure 4.7 Representation of Specimen and 1 mm² Area at the Center

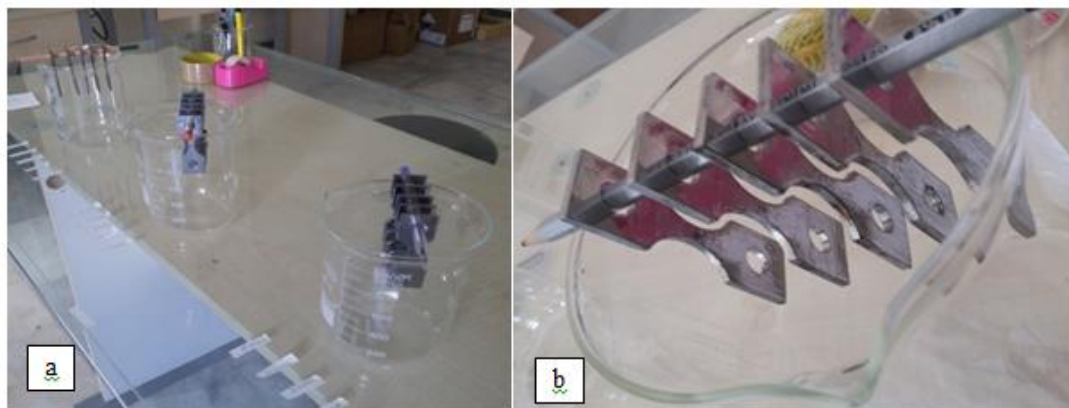


Figure 4.8 (a) Polished With Nail Polish and Dried Specimens (b) Zoom in Image

4.2.3 Corrosion Experiments

To make this research some standards are obtained such as TS 5731 EN ISO 8044 (corrosion of metals and alloys - basic terms and definitions), TS EN ISO 11130 (corrosion of metals and alloys - alternate immersion test in salt solution), TS EN

ISO 11463 (corrosion of metals and alloys - evaluation of pitting corrosion) and TS ISO 11845 (corrosion of metals and alloys- general principles for corrosion testing). If an experimental set up according to standards would be established the cost would be very high so in this research in house developed protocols have been established with the decision of the author to save time and labor savings. Therefore, the corrosion procedure of this study was developed during this study.

4.2.3.1 Testing Procedure

The main reason to carry out the pilot test was to establish the corrosion protocol and to see the effect of time, pH and weight loss. Before the main test, pilot testing was necessary because one of the main reasons of this research is to observe the pit morphology. After corrosion protocol was established the main tests were carried out.

Some pilot tests were done before the final protocol was established. Sixteen specimens were used totally, four for 1.8 % salinity, four for 3.5% salinity, four specimens as pristine and four for pilot tests.

The weight of specimens for pilot experiments designated with ID numbers, was measured and recorded to see the corrosion effect on weights.

Then, the specimens were measured with caliper and 1 mm² areas were marked at the center of the specimens and fingernail polish was used to protect the rest of the area because it contained no metallic substances that could affect the ability to protect the covered surface.

A few alternatives have been investigated instead of fingernail polish. Aluminum tape was supplied from the sample market. However, it was observed that this alternative was not useful in experiments. Because it was very difficult to clean the adhesive from the specimens surfaces.

Afterward, the solutions were prepared to immerse four specimens in different beakers, four solutions were prepared, two of them with 3.5% NaCl and the other two with 1.8% NaCl.

Firstly specimens stayed in the solution for 3.5 hours. Then the specimens were removed from the solution and cleaned using ultrasonic bath. After the corrosion process, each specimen was investigated by optical microscope for pitting characterization and measurements were taken and recorded to a computer with the help of digital images. The corroded area are illustrated in Figure 4.9 (a and b).

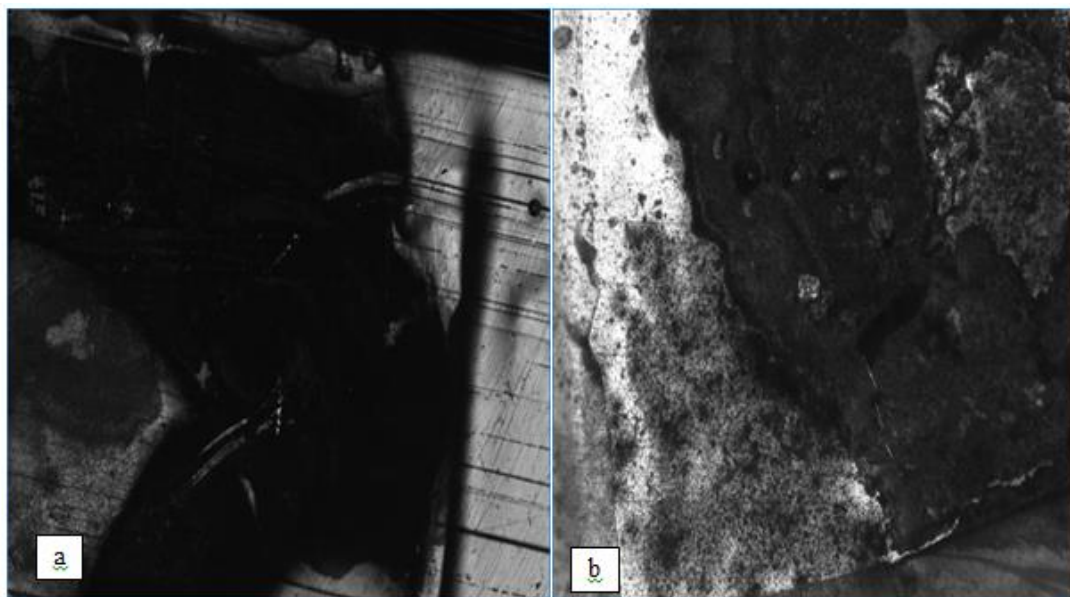


Figure 4.9 (a and b) Pilot Specimens of Corroded Area With 3.5 % NaCl in 3.5 Hours

The pits were combined with each other so desired pitting corrosion was not observed on the surface of samples. For this reason it was decided to add H_2O_2 to decrease pH of the solution resulting faster corrosion. Therefore, corrosion progress could be observed in a clear way [28].

In order to investigate the effect of pH to corrosion, the pH values of the solutions were measured before and after the specimens were immersed to solutions. pH meter used in the experiments is shown in Figure 4.10.



Figure 4.10 pH Meter

The specimens were put into these solutions for 15 minutes periods, after each period they were observed for pit morphology with optical microscope and the process was repeated three times. The immersion procedure and the corroded area are showed in Figure 4.11 (a and b). End of the process, the pH values of solutions were measured.

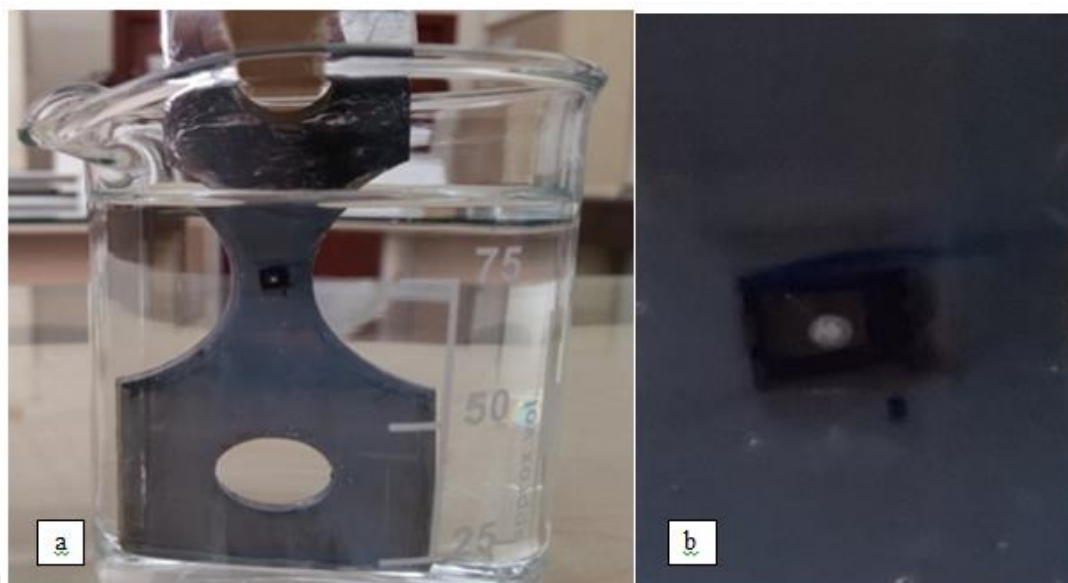


Figure 4.11 (a) An Immersed Specimen in a Prepared Solution (b) Zoom of the Corroded Area

Then, the specimens put to the ultrasonic cleaner and the nail polish was removed from the specimens and weighed after process. Cleaned and weighed four specimens were observed with optical microscope. Then, the four fractured specimens were investigated with Scanning Electron Microscope. The relation between the pits and the fracture points were examined by this way. A summary of the experimental procedure for pilot experiments is showed in Table 4.1.

Table 4.1 A Summary of the Experimental Procedure for pilot experiments

Experiments	Specimen ID	Explanations
Pit Morphology Observation	1.8 ID 1	Pitting Morphologies Observed with Optical Microscope in the First Part of The Study.
	1.8 ID 2	
	3.5 ID 1	
	3.5 ID 2	

After specimens (1.8 ID 1, 1.8 ID 2, and 3.5 ID 1, 3.5 ID 2) were immersed to the solutions, they were taken out each 15 minutes period and observed under optical microscope whether pits occurred or not. Then, for each period images were captured and the specimens cleaned with acetone. Lastly, the cleaned specimens again observed with optical microscope to detect whether the pits continued or not. Hence, the pitting morphology figures include the pits at 15 minutes, 30 minutes, and 45 minutes. They were zoomed to a pit and the growing of this pit was observed.

4.2.3.2 Corrosion Procedure (The main test)

Corrosion experiments were performed with eight specimens in 1.8% and 3.5% NaCl solution in the same way according to corrosion protocol established with pilot test as stated in 4.2.3.1 section. The main purpose of this study is to observe pit morphology and the effect of morphology to mechanical properties. Therefore, after corrosion experiments the mechanical properties are also studied for main tests. A summary of the experimental procedure for main test experiments is showed in Table 4.2.

Table 4.2 A Summary of the Experimental Procedure for Main Test Experiments

Experiments	Specimen ID	Explanations
Tensile Test	PS ID 1	Eight Corroded and Four Pristine Specimens were Applied Tensile Tests. The Reason is to Investigate The Effect of Corrosion to the Strength.
	PS ID 2	
	PS ID 3	
	PS ID 4	
	1.8 ID 3	
	1.8 ID 4	
	1.8 ID 5	
	1.8 ID 6	
	3.5 ID 3	
	3.5 ID 4	
	3.5 ID 5	
	3.5 ID 6	
Scanning Electron Microscope Observation	1.8 ID 3	Four Corroded Specimens were Observed with Scanning Electron Microscope. The reason is to Investigate the Relation of Fracture Points with the Pits.
	1.8 ID 6	
	3.5 ID 3	
	3.5 ID 4	

4.2.4 Microstructural Analysis

Microstructure photographs of corroded samples are taken by NIKON Eclipse MA100 inverted optical microscope in 1000X magnification to illustrate the pit structure, orientation, and size.

In addition, pitting corrosion images of samples are taken in 100X magnification to show the effect of NaCl and time on surface structure.

Also, average pitting sizes of samples are calculated by Clemex Vision Lite software.

4.2.5 Elemental Analysis

Elemental analysis is examined by HITACHI TM 4000 EDS (energy dispersive spectroscope) analysis system attached to SEM (scanning electron microscope) in Ankara Yıldırım Beyazıt University. Examined areas are chosen randomly in 10000X magnification. Results are illustrated via EDS software suite.

4.2.6 Mechanical Testing

After optical microscope examination process, pristine specimens, 1.8 ID 3, 1.8 ID 4, 1.8 ID 5 and 1.8 ID 6 and 3.5 ID 3, 3.5 ID 4, 3.5 ID 5 and 3.5 ID 6 specimens were applied tensile tests. Tensile test is carried out to obtain the stress-strain diagram, to determine the tensile properties and hence to get valuable information about the mechanical behavior and the engineering performance of the material.

The tensile test was also carried out at 23 ° C and 50% relative humidity. The specimens were pulled with 1 mm/min velocity and the data were recorded. The load and elongation were measured continuously and recorded until failure. Figure 4.12 shows tensile test and Figure 4.13 shows zoom of the breaking area.

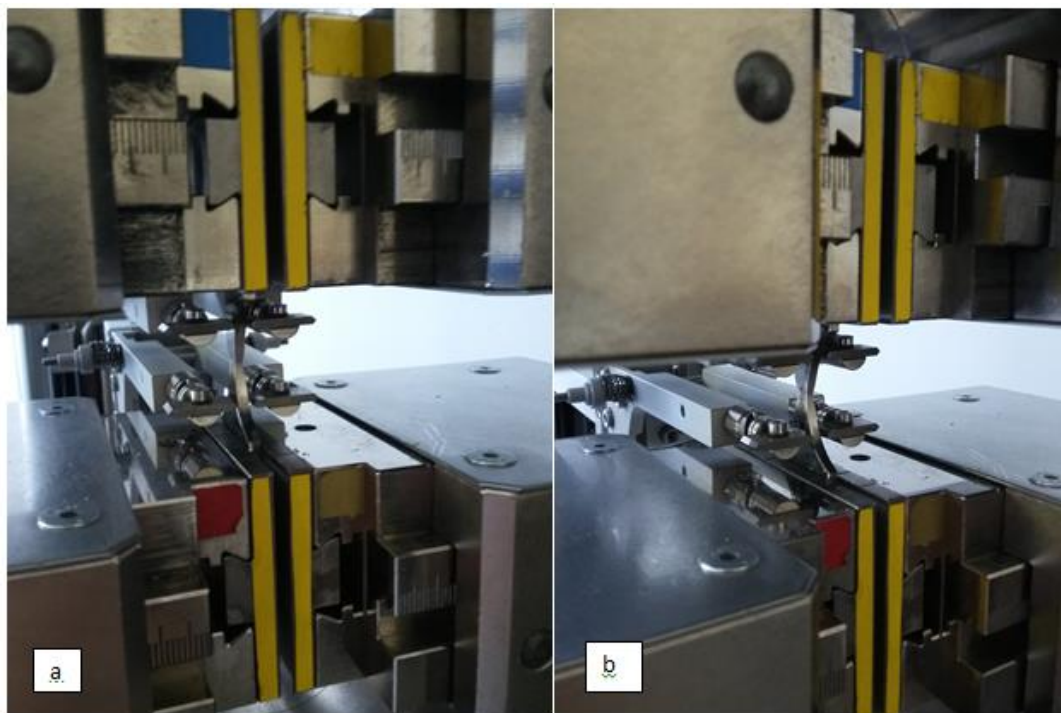


Figure 4.12 (a) Sample Placed in the Device (b) The Breaking Moment of the Specimen

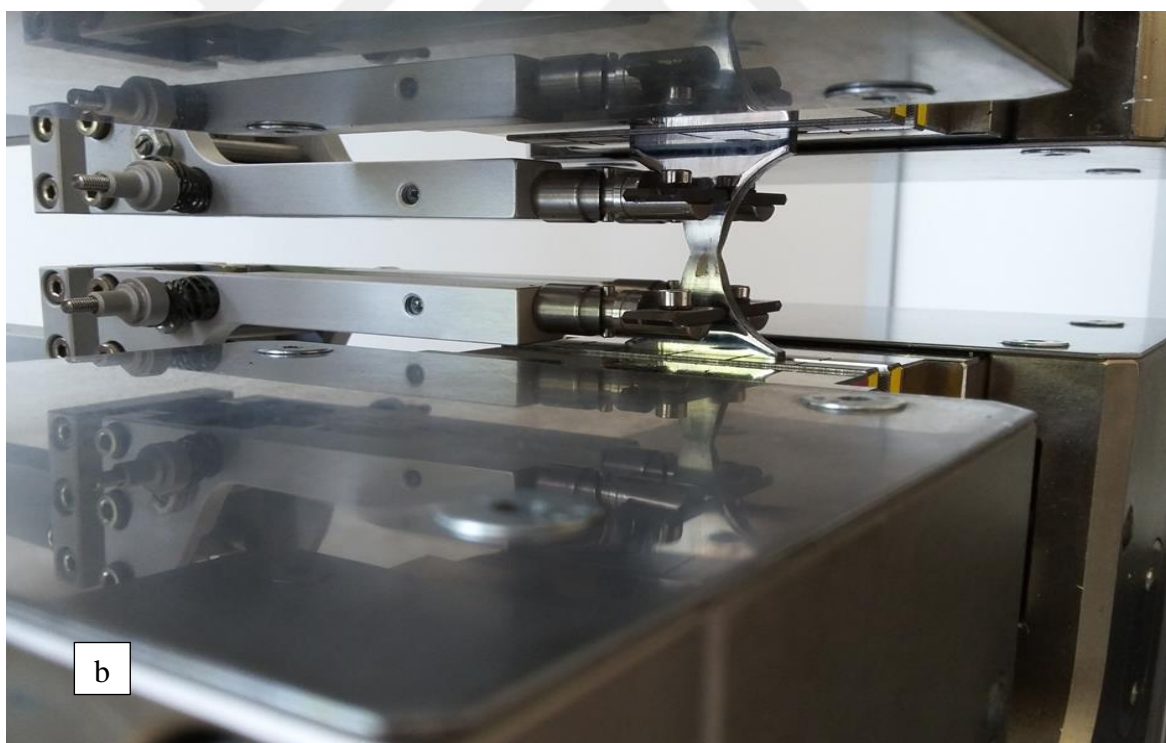
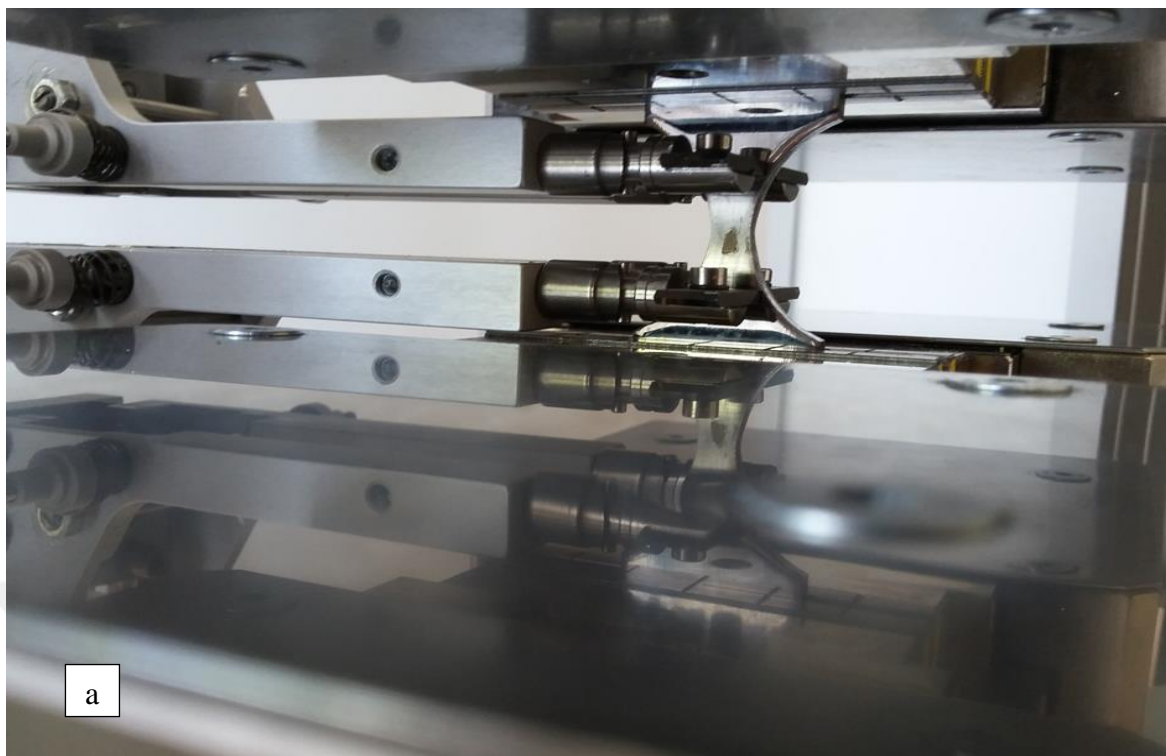


Figure 4.13 Zoom of the Breaking Area (a and b)

4.2.7 Scanning Electron Microscope

HITACHI TM 4000 Scanning Electron Microscope (SEM) was used for investigation after removing the corrosion product. The internal structures of surfaces were analyzed by SEM. SEM analysis was performed both on fractured surfaces and on polished surfaces.



CHAPTER 5

RESULTS AND DISCUSSION

5.1 Effect of Pitting Corrosion on Weight

The effects of the pitting corrosion on the reinforced steel St 37 were investigated. The pitting corrosion is a localized corrosion and they are seen on the optical microscope as small hollows. These hollows are progressed to vertical direction to the material inside. Pitting corrosion can propagate until out of service and it may not be even noticed. As mentioned in methods section, there were twelve specimens for solutions and four specimens for pristine. Only four specimens material loss was investigated for pilot tests. The Table 5.1 shows this material average loss study. According to the Table 5.1, the material loss is almost zero, which means very low material loss was observed. Rahbar Ranji A., et al. [29] and Yüzer N. et. al. [30] suggested that weight loss has a low tendency. Thus, there is no possibility to comment this result for pitting corrosion formation. However, in literature survey, researches related with time and weight loss have different results. Muhirwa A. C. et. al. [31] suggested that generally, there was an linear trend between weight loss and increased duration of exposure.

Table 5.1 Material Weight Losses of Specimens as Percentage for Pilot Tests

Specimen	% Loss
1.8 ID 1	0,0009
1.8 ID 2	0,0006
3.5 ID 1	0,0006
3.5 ID 2	0,0025

5.2 Effect of Pitting Corrosion on Solution pH

One of another factor that can affect the pitting corrosion is the pH value. The structural metal St 37 was immersed to the solutions and pH value difference of the solutions were observed after the specimens were corroded. There is no significant change of the pH values as seen in Table 5.2 and in Figure 5.1. Some researchers have reported that the inhibitive properties of the concrete cannot be expressed only by the OH⁻ concentration because also a lot of other factors such as alkaline reserves affect to the concrete [32]. Moreover, other research also stated that the chloride relation with pH is not a reliable parameter since the effect of the other factors may change the pH [33].

Table 5.2 pH Value before Corrosion and after Corrosion for Pilot Test

Specimen	Before Corrosion	After Corrosion
1.8 Specimens	3,94	3,96
3.5 Specimens	3,97	4,02

Hence, the pH change may not be a significant parameter on the pitting corrosion. However, the reason of the little change may be the short immersion time of specimens.

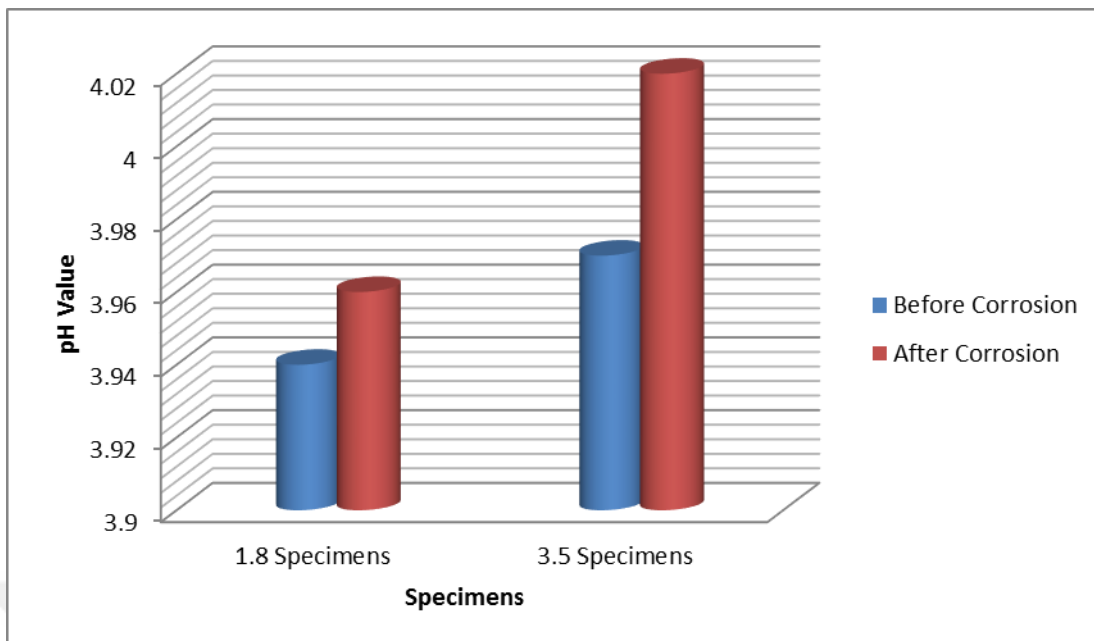


Figure 5.1 pH Value before Corrosion and after Corrosion

5.3 Pitting Morphology Observations

After specimens immersed to the solutions, they were taken out each 15 minutes period including the pits at 15 minutes, 30 minutes, and 45 minutes and cleaned with acetone. Then specimens were observed with optical microscope. The pitting morphology Figures 1.8 ID 1, 1.8 ID 2 and 3.5 ID 1, 3.5 ID 2 (pilot test) were zoomed to a pit and the growing of this pit was observed. The change in the pit morphology of specimen illustrated in Figure 5.2 and Figure 5.3.

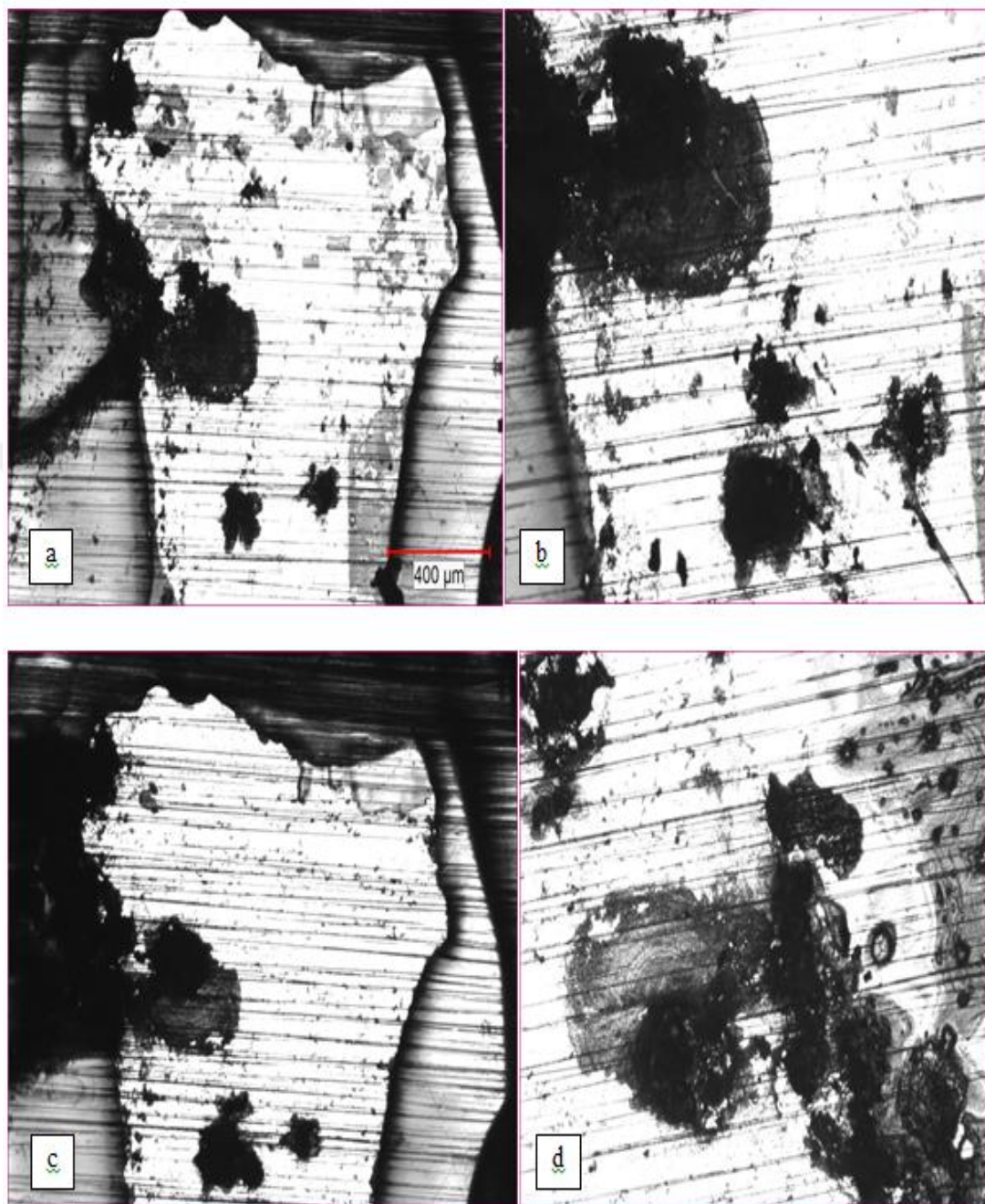
Specimen 1.8 ID 2

Figure 5.2 The Change in the Pit Morphology of Specimen 1.8 ID 2 at (a) 15 min. (b) 30 min. (c) 45 min. (d) Cleaned with Acetone

Specimen 3.5 ID 1

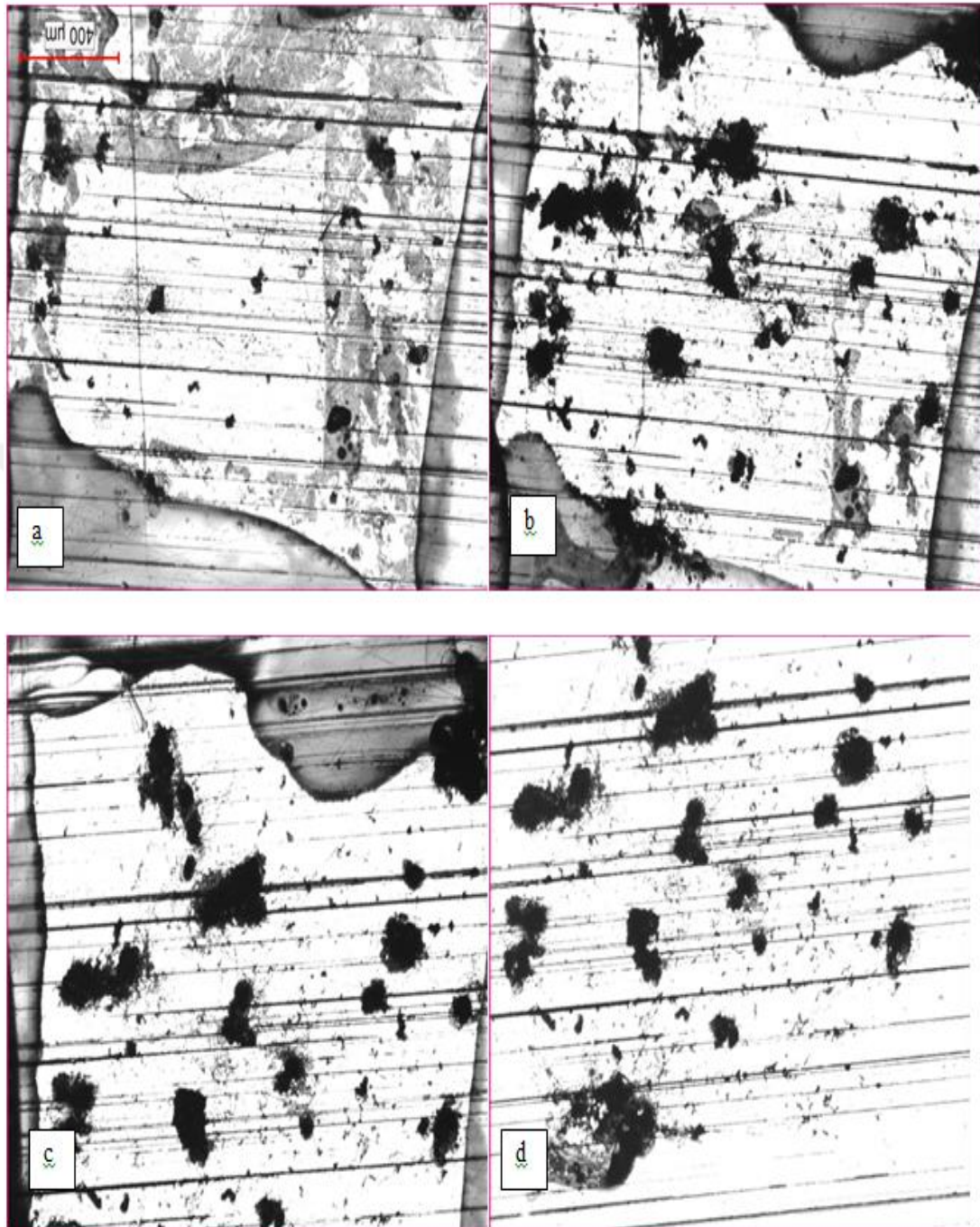


Figure 5.3 The Change in the Pit Morphology of Specimen 3.5 ID 1 at (a) 15 min. (b) 30 min. (c) 45 min. (d) 45 min (Cleaned with Acetone)

Some pits were noticed within the structures of the samples as the Chloride ion concentration increased.

The sizes of pits in Figure 5.4, Figure 5.5 and Figure 5.6 showed the pit morphologies of the specimens. Each 15 minutes the pits in the corroded area grew. These figures were cropped from the whole images to show the pits only. Especially pits grew in the first 15 minutes for all specimens. However, after first 15 minutes the depths of pits increased instead of the size of them. This meant that the sizes of the pits were almost same after 15 minutes. In addition, the pits were tending to be more circular.

The size of the pits 1.8 ID 1 in (15 min.) was $7096 \mu\text{m}^2$, in (30 min.) was $10752 \mu\text{m}^2$, in (45 min.) was $12584 \mu\text{m}^2$ and in (45 min. with acetone) was $11650 \mu\text{m}^2$. The reason of decrease was due to the acetone addition.

The size of the pits 1.8 ID 2 in (15 min) was $12150 \mu\text{m}^2$, in (30 min.) was $12520 \mu\text{m}^2$, in (45 min.) was $12864 \mu\text{m}^2$ and in (45 min. with acetone) was $13650 \mu\text{m}^2$. Thus, the size of pit developed with time.

The size of the pits 3.5 ID 1 in (15 min) was $6315 \mu\text{m}^2$, in (30 min.) was $7562 \mu\text{m}^2$, in (45 min.) was $8624 \mu\text{m}^2$ and in (45 min. with acetone) was $9150 \mu\text{m}^2$. In this pit, these size belong Figure 5.6 (a). Figure 5.6 (b), Figure 5.6 (c) and Figure 5.6 (d) had two more circular pits. This showed that these two new pits were formed and they had grown.

The size of the pits 3.5 ID 2 in (15 min) was $8132 \mu\text{m}^2$, in (30 min.) was $9545 \mu\text{m}^2$, in (45 min.) was $10584 \mu\text{m}^2$ and in (d) was $10225 \mu\text{m}^2$. The reason of decrease in (45 min. with acetone) was due to the acetone addition.

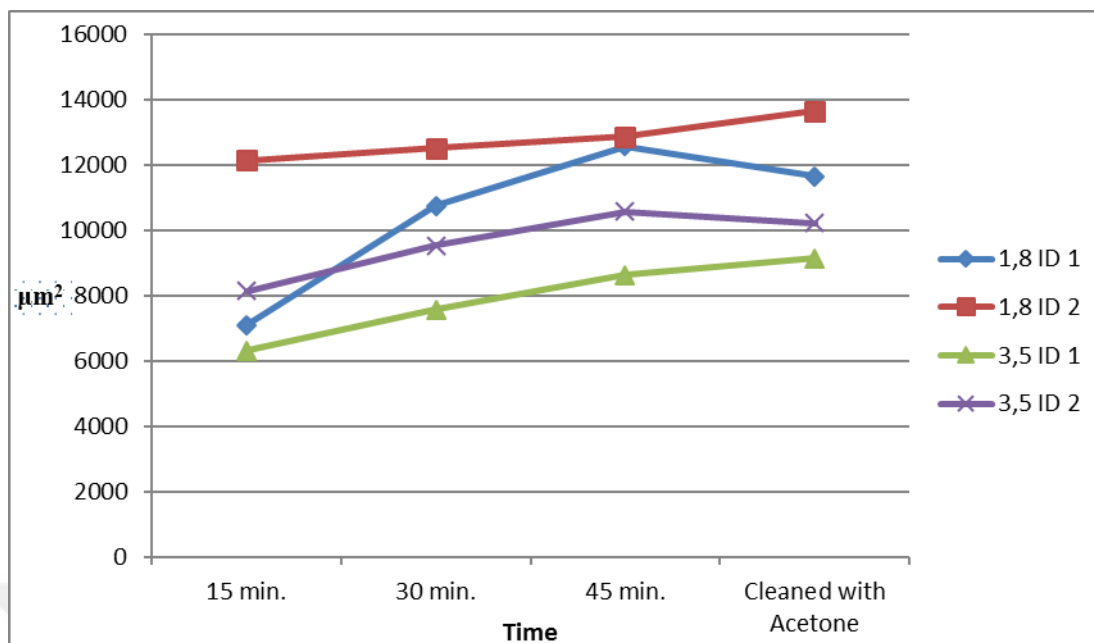


Figure 5.4 The size of the pit

Specimen 1.8 ID 2

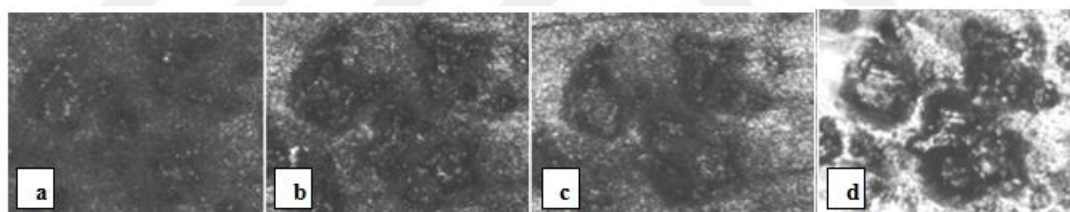


Figure 5.5 The Change in the Pit Morphology of Specimen 1.8 ID 2 at (a) 15 min. (b) 30 min. (c) 45 min. (d) Cleaned with Acetone

Specimen 3.5 ID 1

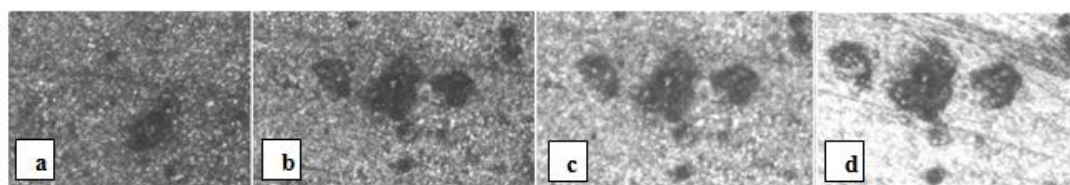


Figure 5.6 The Change in the Pit Morphology of Specimen 3.5 ID 1 at (a) 15 min. (b) 30 min. (c) 45 min. (d) Cleaned with Acetone

5.4 Tensile Test Results

Tensile test was used to observe the durability of the material under static forces and other mechanical properties. The studies showed that the tensile test applied on metallic materials, the physical detriments such as scratches or cracks of the materials affected negatively the strength [34].

According to the results, the specimens immersed to the 1.8 % NaCl solution showed lower strength than the specimens put to the 3.5 % NaCl solution as unexpected (Figure 5.7 to 5.18). The surface area of the pits for the specimens that were immersed inside 1.8% NaCl solution showed higher values than that of the specimens immersed inside 3.5% NaCl solution. Because of the higher pit area of the specimens in 1.8% NaCl solution, effective area of these specimens decreased in a higher value than effective area of specimens in 3.5% NaCl solution, lowering the tensile properties of these specimens. But pristine specimens's strength higher than other specimens which immersed to the solutions as expected (Table 5.3 and Figure 5.19). The residual strength of a material is related with the effective area of material. If the effective area of the material decreases by means of corrosion pits, defects, voids, etc. then the residual strength of the material also decreases. Because the tensile property of a material is as follows.

$$\sigma = F/A \tag{5.1}$$

where F is the force applied to the cross section and A is the cross sectional area.

During the testing F doesn't change, as the A decreases F/A ratio increases.

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ϵ_B Kopma Uz. %	ϵ_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	486,28	6291,5	19,020	-	26,7	11,1	429	35300

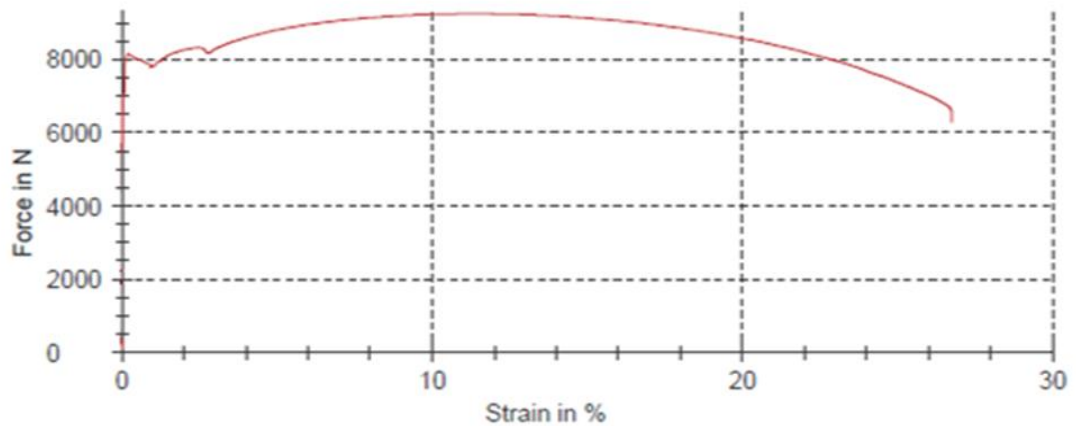
Series graph:

Figure 5.7 The Tensile Test Graph of PS ID 1 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ϵ_B Kopma Uz. %	ϵ_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	521,60	6853,4	18,320	-	25,8	10,9	477	74200

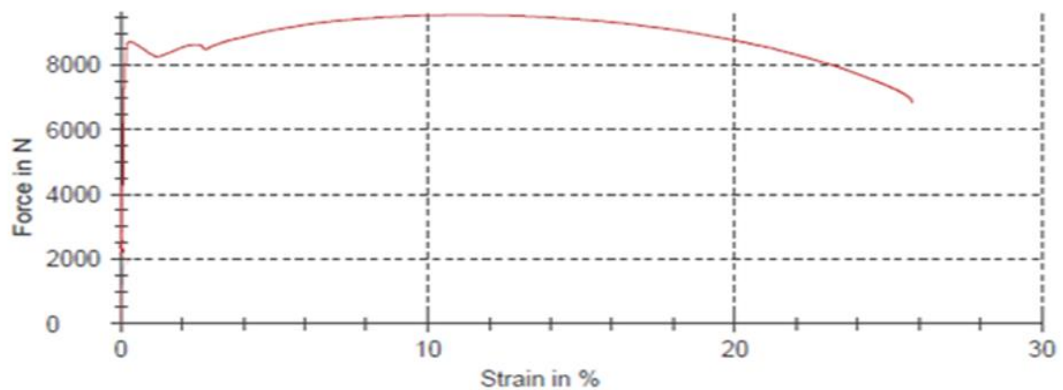
Series graph:

Figure 5.8 The Tensile Test Graph of PS ID 2 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ε_B Kopma Uz. %	ε_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	491,36	6310,9	18,790	-	26,3	11,3	491	28400

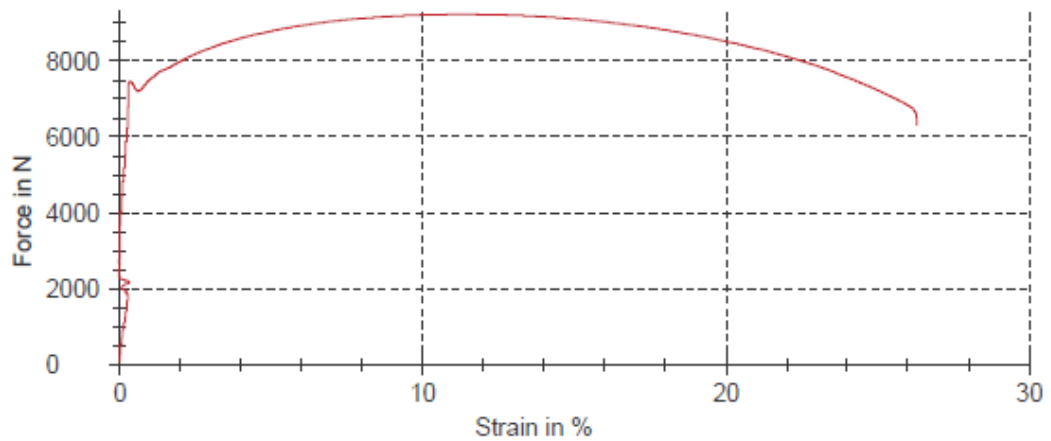
Series graph:

Figure 5.9 The Tensile Test Graph of PS ID 3 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ε_B Kopma Uz. %	ε_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	492,42	6272,4	18,610	-	26,5	11,8	492	19700

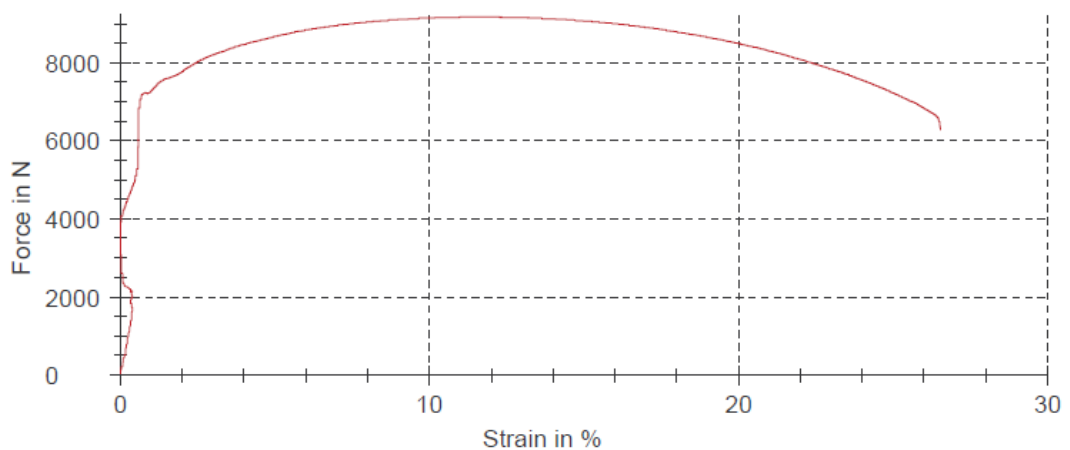
Series graph:

Figure 5.10 The Tensile Test Graph of PS ID 4 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ϵ_B Kopma Uz. %	ϵ_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	367,89	5008,0	18,930	-	21,7	9,9	301	151000

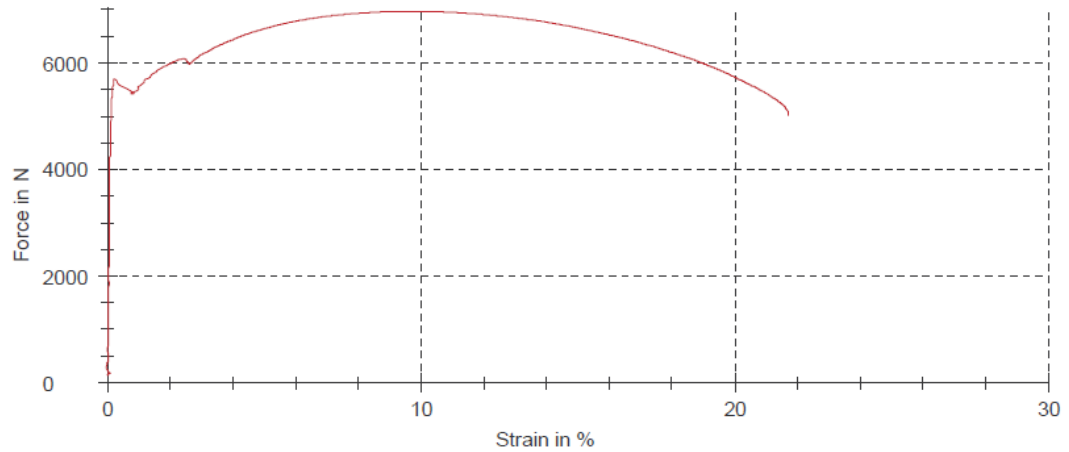
Series graph:

Figure 5.11 The Tensile Test Graph of 1.8 ID 3 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ϵ_B Kopma Uz. %	ϵ_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	372,41	4519,0	17,720	-	19,5	8,4	306	96400

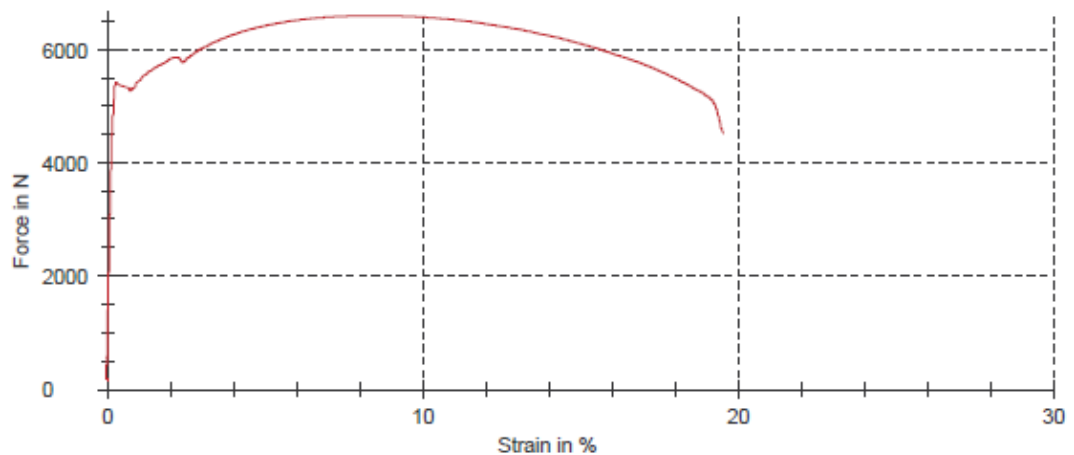
Series graph:

Figure 5.12 The Tensile Test Graph of 1.8 ID 4 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ϵ_B Kopma Uz. %	ϵ_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	385,10	4212,3	18,150	-	16,5	8,7	308	133000

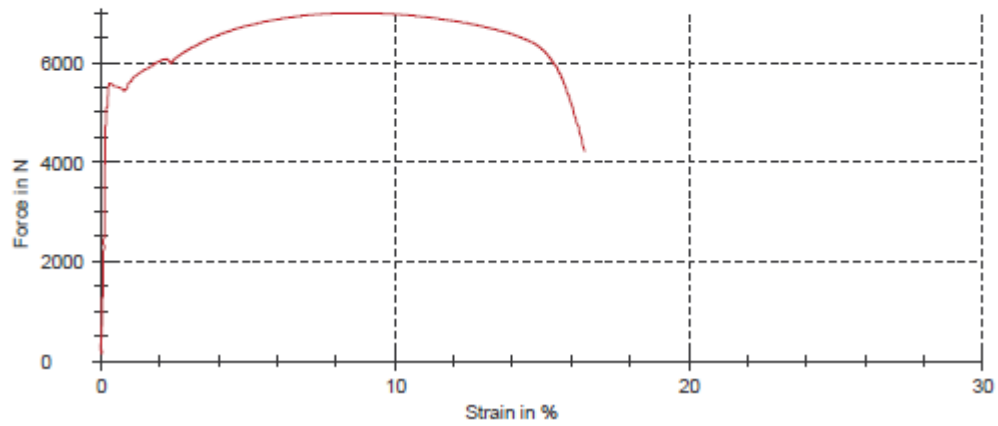
Series graph:

Figure 5.13 The Tensile Test Graph of 1.8 ID 5 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ϵ_B Kopma Uz. %	ϵ_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	367,79	3530,3	18,840	-	16,2	8,4	297	143000

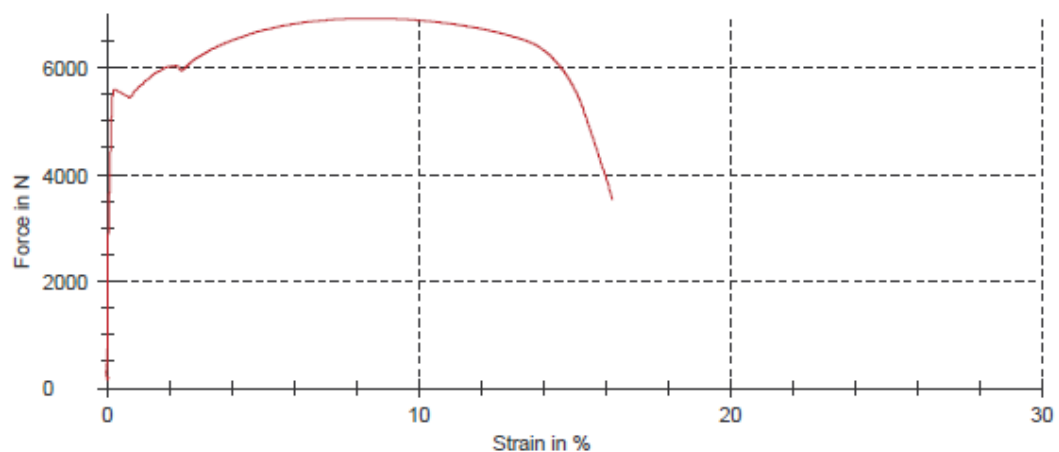
Series graph:

Figure 5.14 The Tensile Test Graph of 1.8 ID 6 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ϵ_B Kopma Uz. %	ϵ_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	386,84	4810,3	17,710	-	22,7	10,7	335	82300

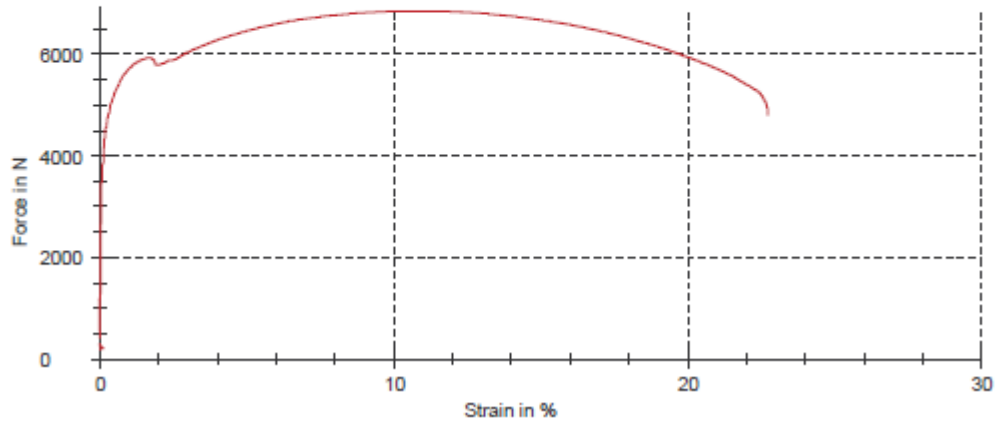
Series graph:

Figure 5.15 The Tensile Test Graph of 3.5 ID 3 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ϵ_B Kopma Uz. %	ϵ_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	378,57	5075,1	18,620	-	20,5	9,7	307	119000

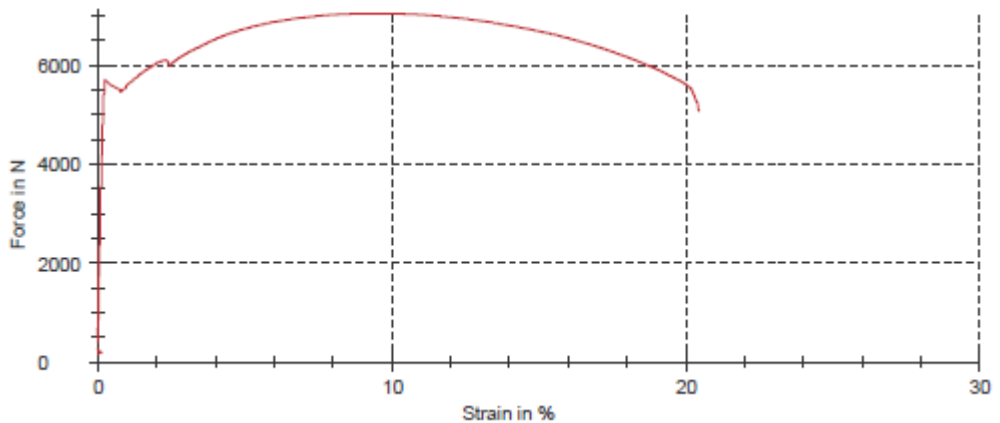
Series graph:

Figure 5.16 The Tensile Test Graph of 3.5 ID 4 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ϵ_B Kopma Uz. %	ϵ_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	378,06	4903,7	17,550	-	21,9	10,8	306	134000

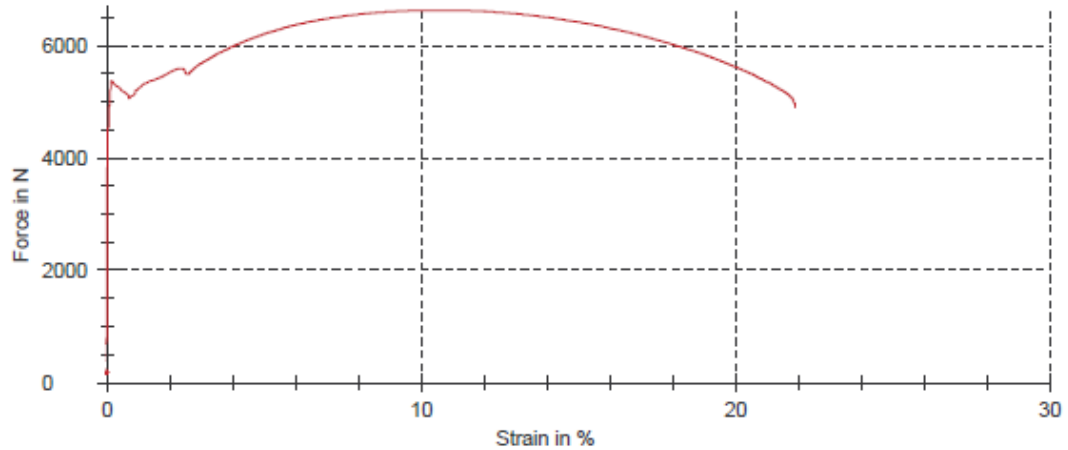
Series graph:

Figure 5.17 The Tensile Test Graph of 3.5 ID 5 as Force (N) to Strain (%).

Test results:

Nr	σ_M Cekme Muk. N/mm ²	σ_B Kopma Muk. N	A_0 Kesit mm ²	F (30 %) N/mm ²	ϵ_B Kopma Uz. %	ϵ_M Cekme Uz. %	σ_Y Akma N/mm ²	EModulu N/mm ²
1.1	377,08	4773,8	18,270	-	21,0	9,7	308	103000

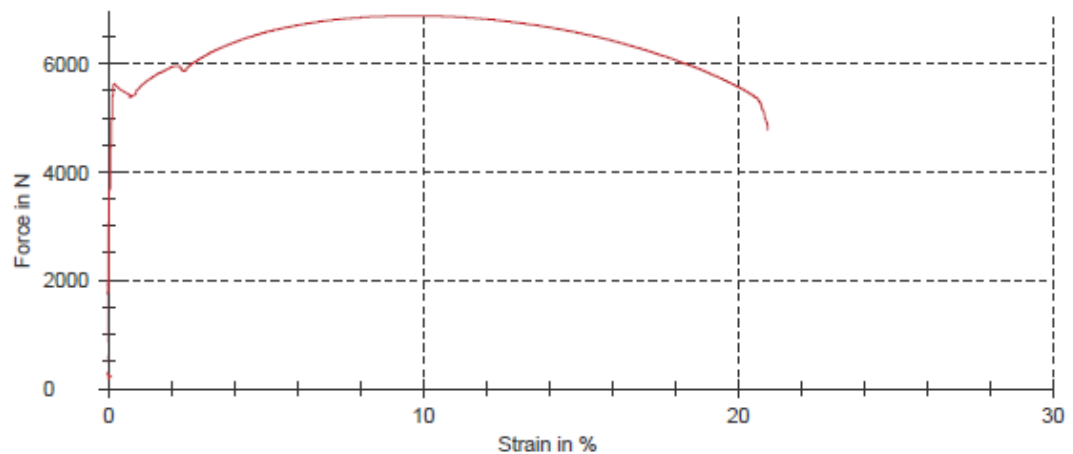
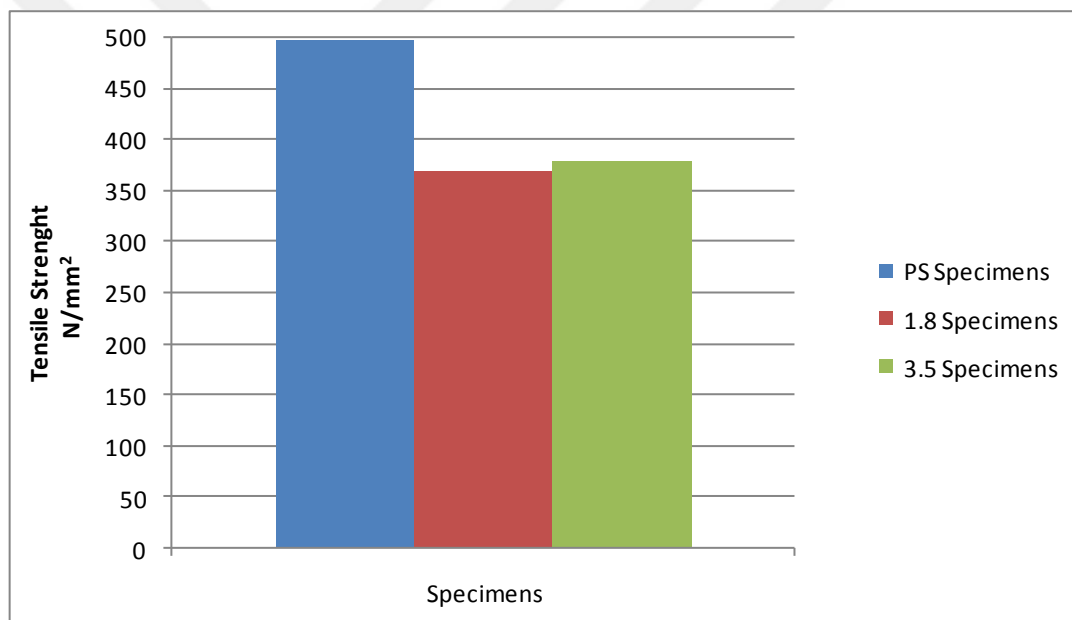
Series graph:

Figure 5.18 The Tensile Test Graph of 3.5 ID 6 as Force (N) to Strain (%).

Table 5.3 Results of the Tensile Tests Average

Specimen	Tensile Strength [N/mm ²]	Fracture Strength [N]	Strain Rate [%]
PS Specimen	498	6432	26
1.8 Specimen	369	4352	19
3.5 Specimen	378	4917	21

**Figure 5.19** Results of the Tensile Tests

5.5 Scanning Electron Microscope (SEM) Results

The SEM analysis before and after fracture were carried out. The aim of the SEM analysis was to determine whether the fracture came out from the corroded area or not. Then, if fracture was due to corrosion pits, the purpose was to determine the origin of the fracture (from which pits). The elemental analysis by using SEM EDS

was also performed. The fracture came from the corroded area at the same time corrosion pits could be observed clearly (Figure 5.20 and Figure 5.21).

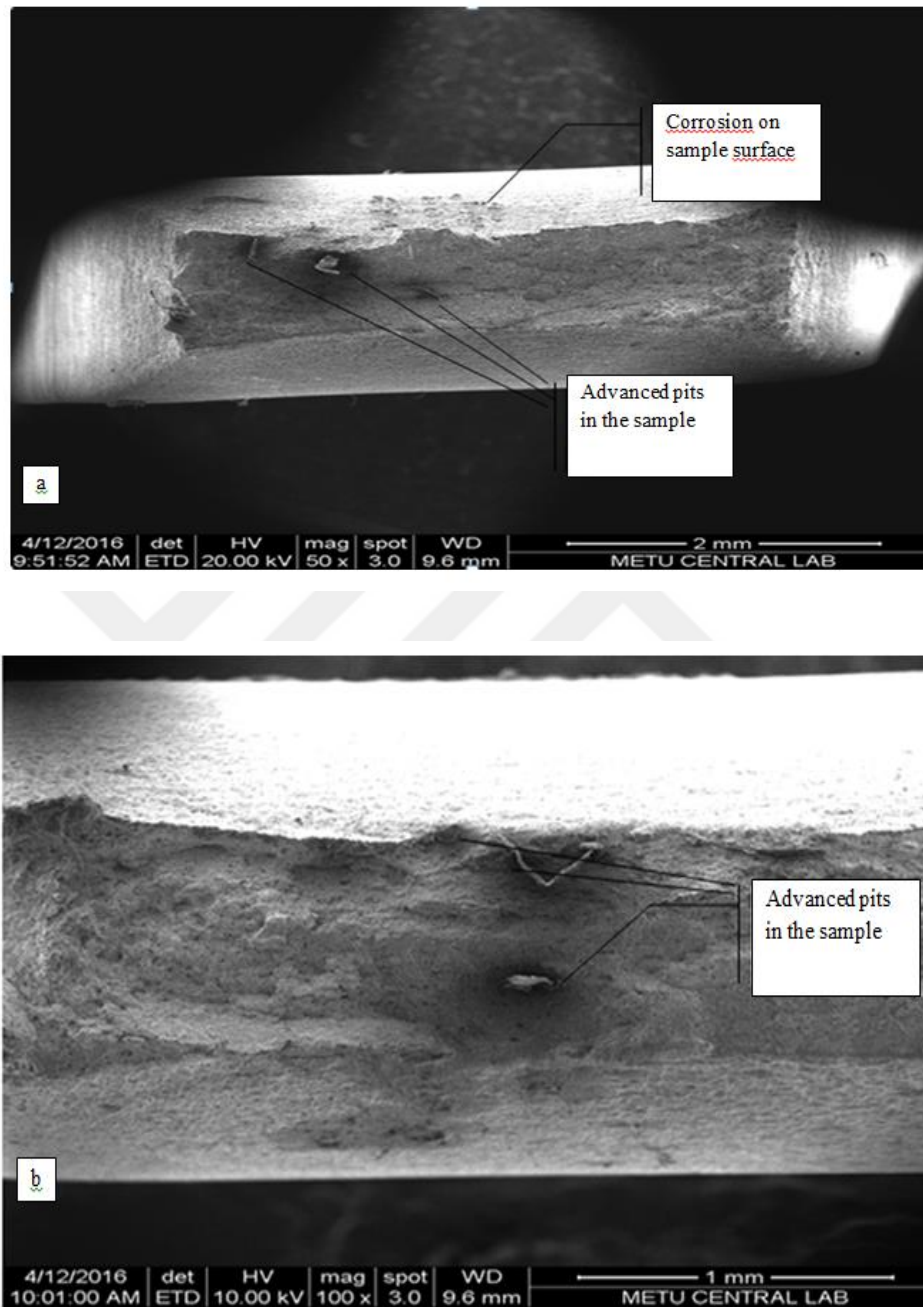


Figure 5.20 From Surface View SEM Micrographs of Specimens Immersed to the (a)&(b) 3.5% NaCl Solution

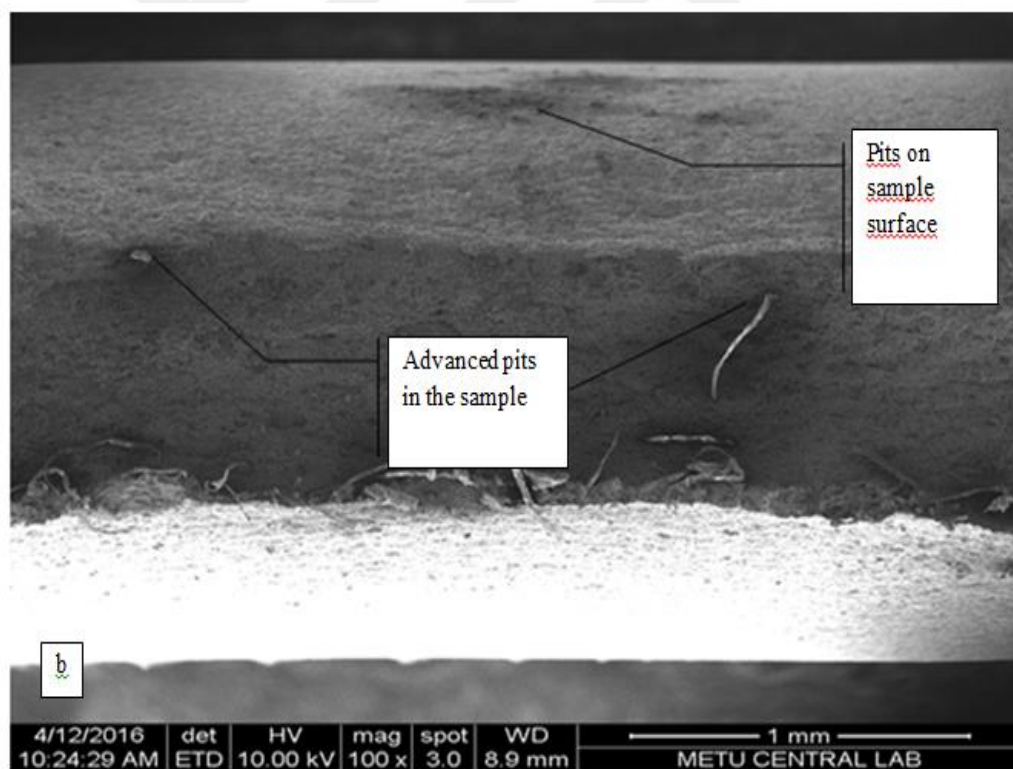
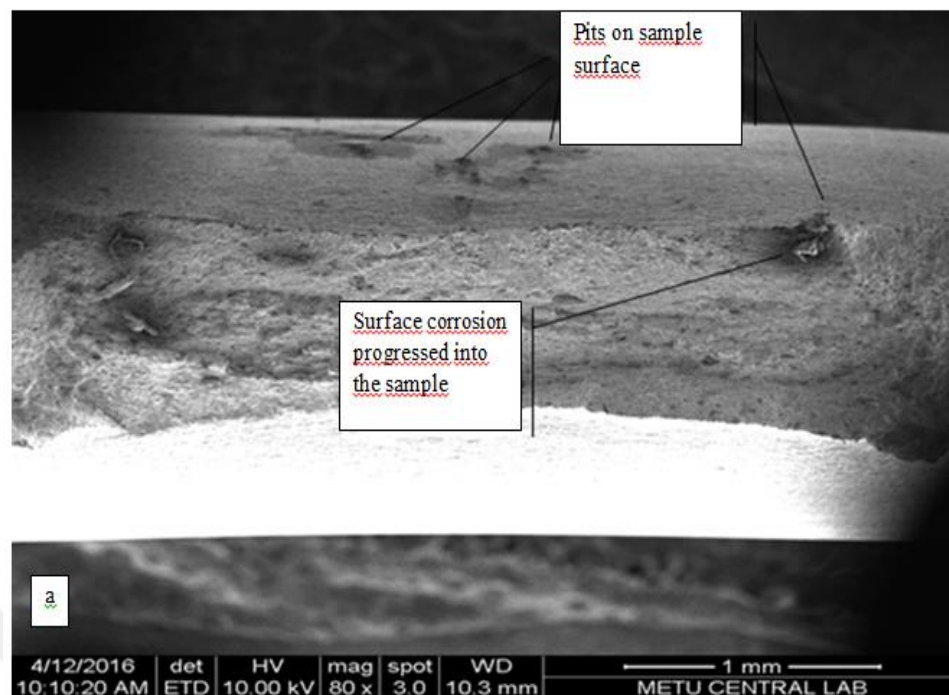


Figure 5.21 From Surface View SEM Micrographs of Specimens Immersed to the (a)&(b) 1.8 % NaCl Solution

Figure 5.22 (a and b) and Figure 5.23 (a and b) shows the SEM images of corroded surfaces of specimens in 1.8% NaCl and 3.5% NaCl solution before tensile testing. According to the results, the specimens immersed to the 1.8 % NaCl solution showed lower strength than the specimens put to the 3.5 % NaCl solution as unexpected. The reason of this result was that the specimens immersed to the 1.8 % solution had large number of pits on their surfaces after they were corroded and the pits average size were nearly 65-75 μm . But, the specimens immersed to 3.5% solution had few number of pits on their surfaces after they were corroded and the pits average size were nearly 90-100 μm and this results showed that the small and large number of pit reduced strengths unexpectedly. Therefore, the stress rates were different from each other and this was an expected situation. Some researchers stated that increase pitting corrosion decreased the tensile strength and total elongation drastically [35].

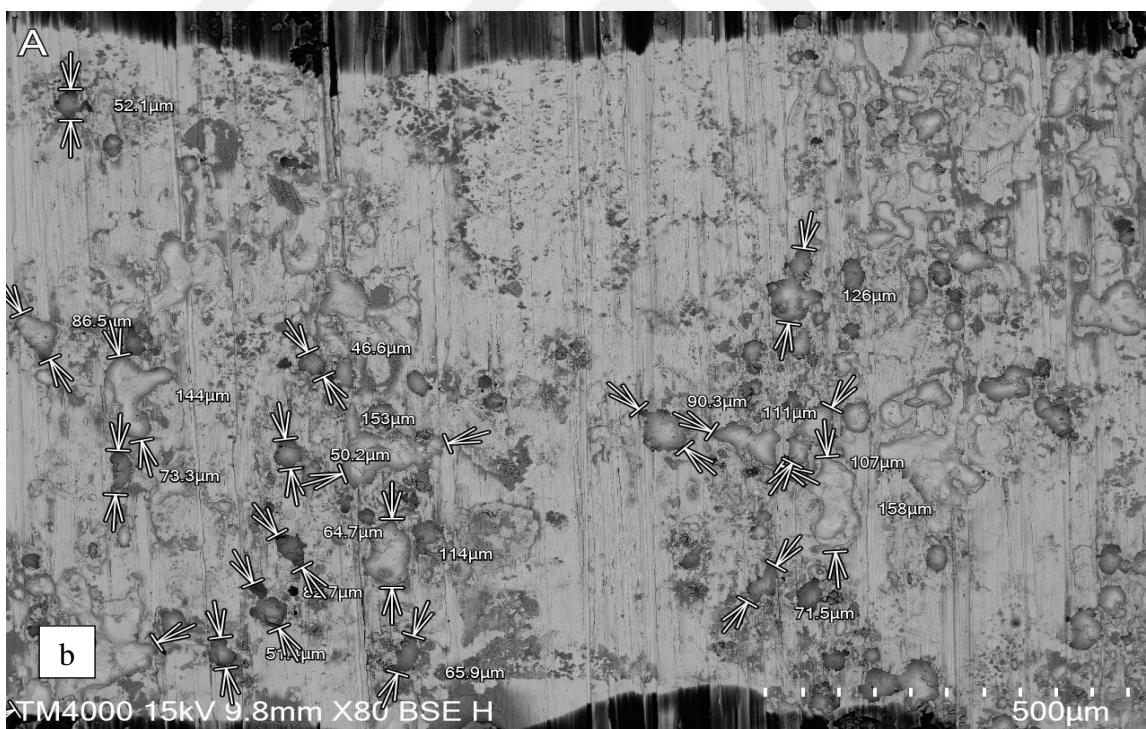
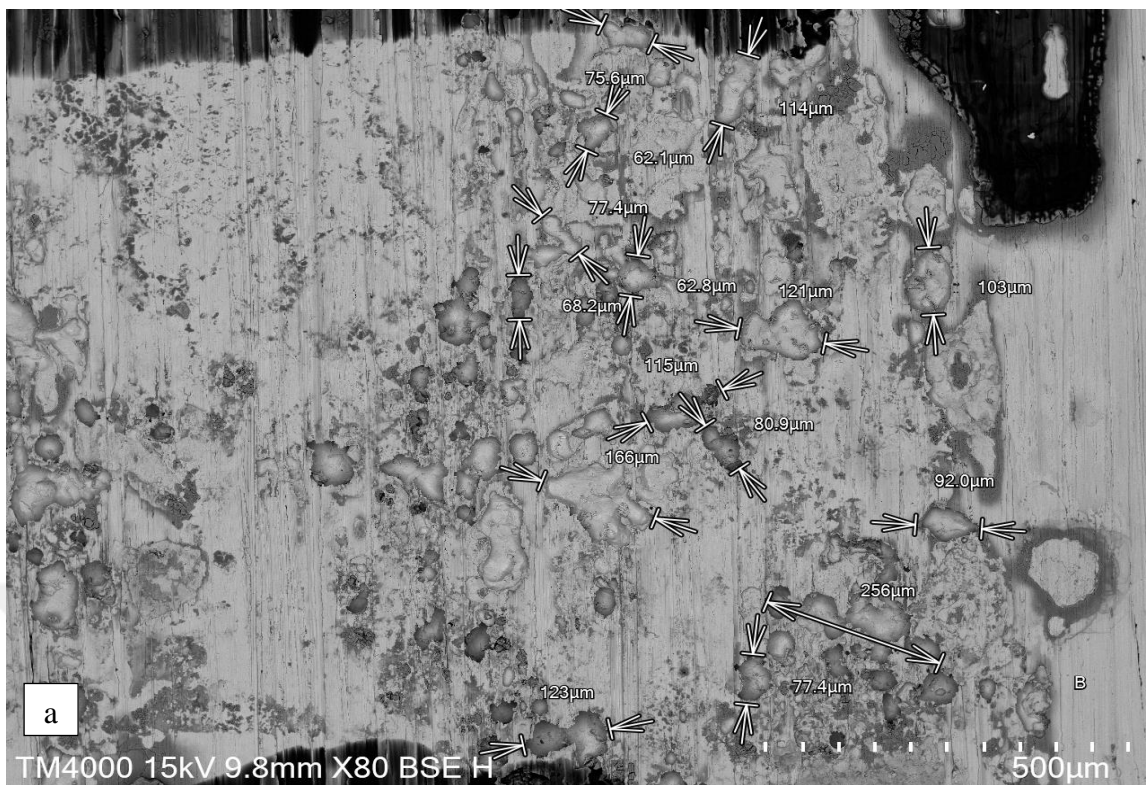


Figure 5.22 Left (a) and Right (b) Surface View SEM Micrographs of Specimens 1.8 ID 3 Immersed to the 1.8% NaCl Solution.

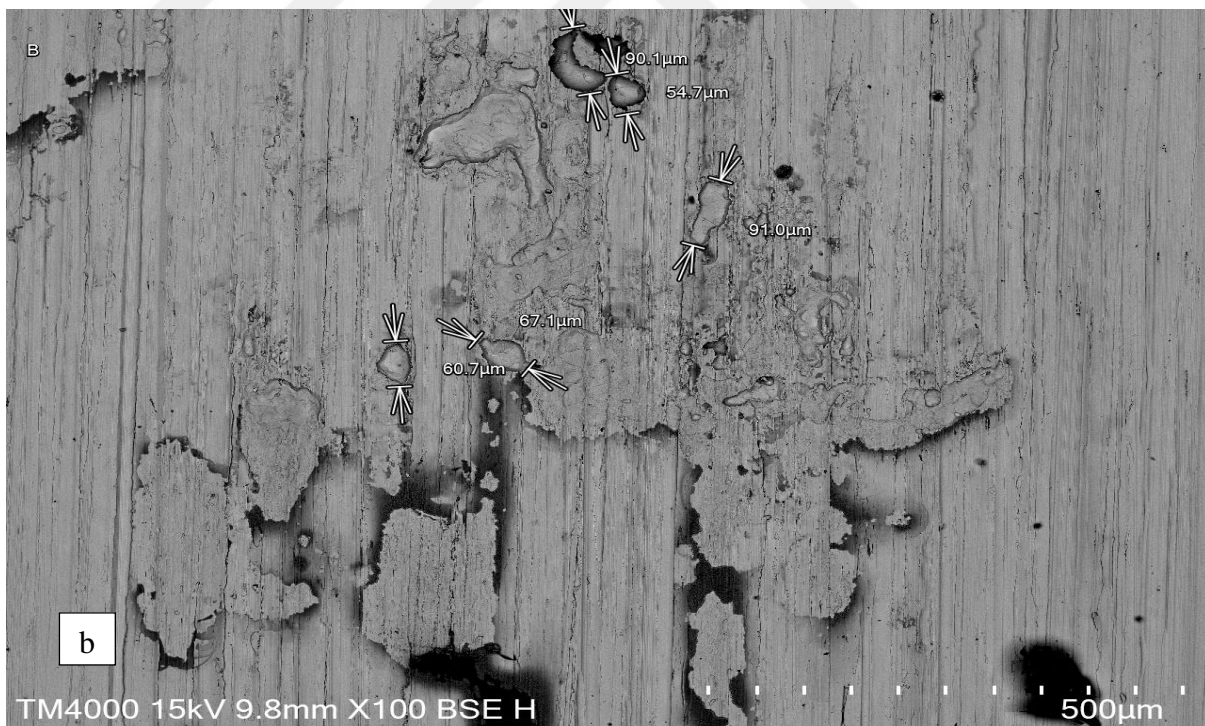
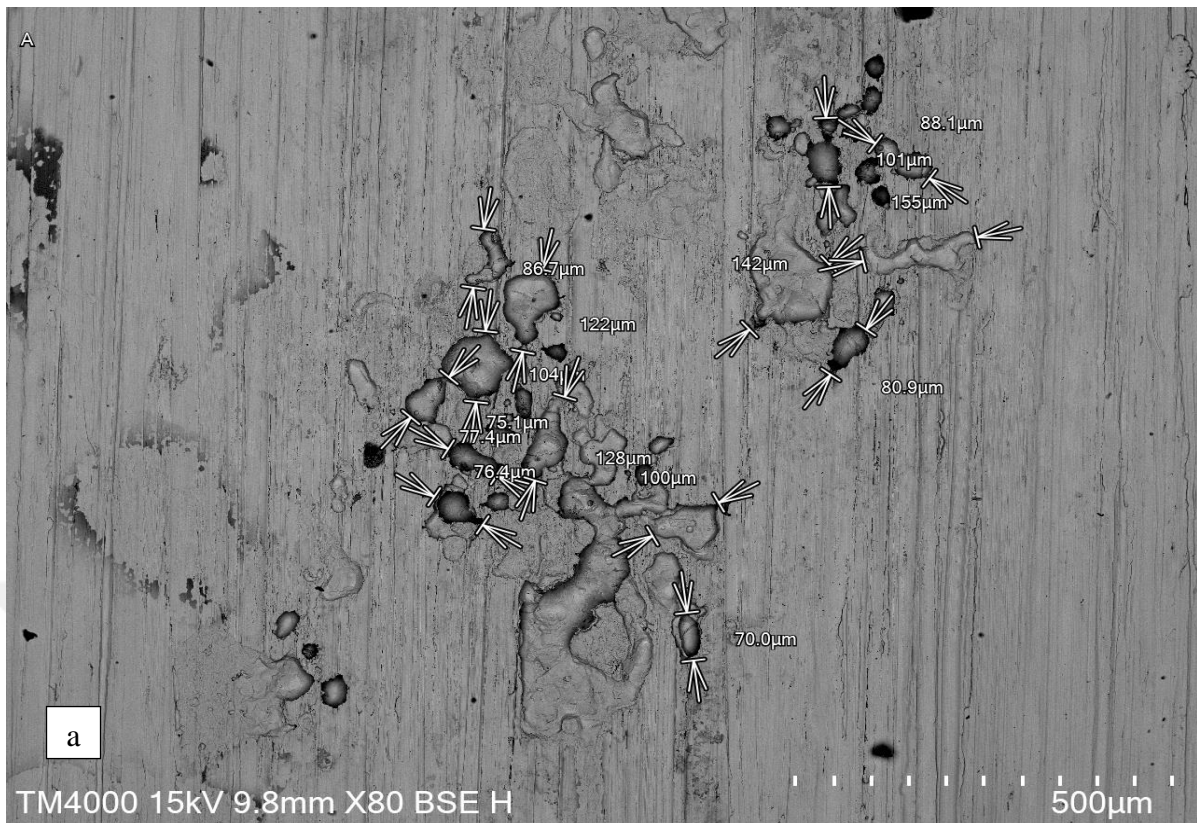


Figure 5.23 Left (a) and Right (b) Surface View SEM Micrographs Of Specimens 3.5 ID 3 Immersed to the 3.5 % NaCl Solution.

It could not be detected where the fracture came from which pit due to the coalescence of the corrosion pits and hence we observed that several pits combined and formed a single big pit. As it seen from Figure 5.24 and Figure 5.25, for both specimens corroded in 1.8% and 3.5% NaCl solution, the fracture came from the corroded area however it could not detected the origin of the fracture.

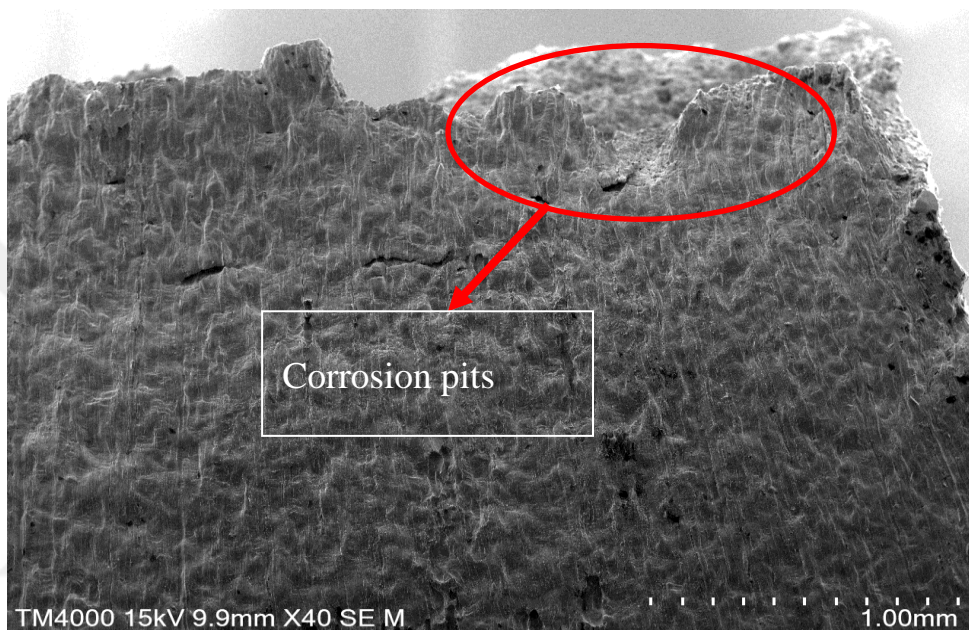


Figure 5.24 From Surface View SEM Micrographs of Specimens 1.8 ID 3



Figure 5.25 From Surface View SEM Micrographs of Specimens 3.5 ID 3

Figure 5.26 shows the SEM and EDS images of cross sectional view of specimen 3.5 ID 4. EDS analysis made on cross sectional area illustrated that there were no C elements on fractured region. Because of the lack of C according to EDS analysis it was concluded that the studied material was a low C steel. On the other hand EDS analysis detected C elements on the surface of corroded specimens (Figure 5.27). Because there could be some finger nail polish residue, contains C elements, on the surface.

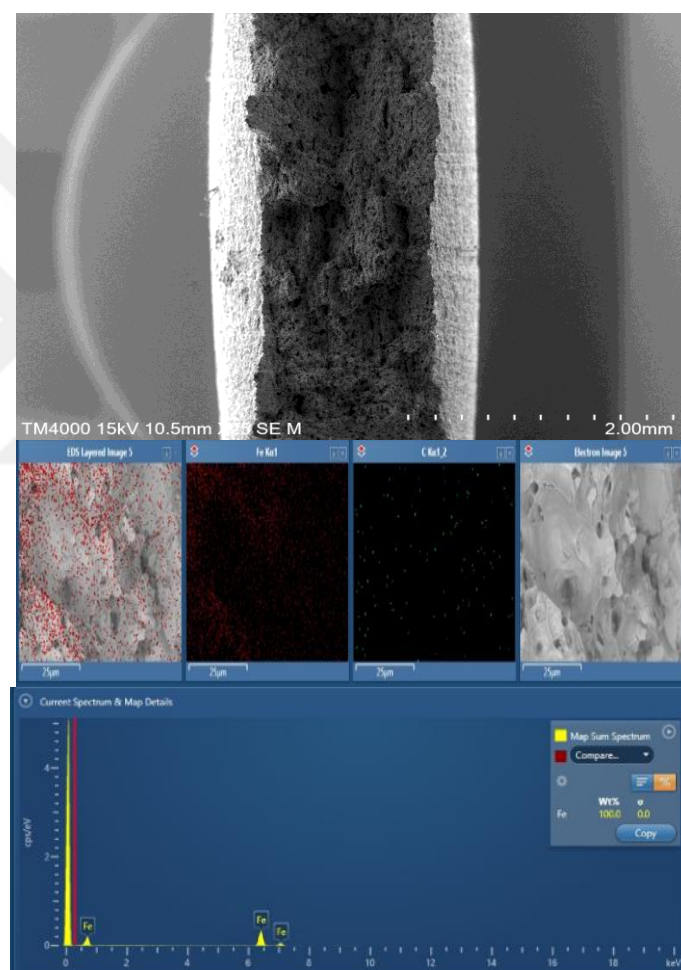


Figure 5.26 EDS Cross Sectional Views of Specimens 3.5 ID 4

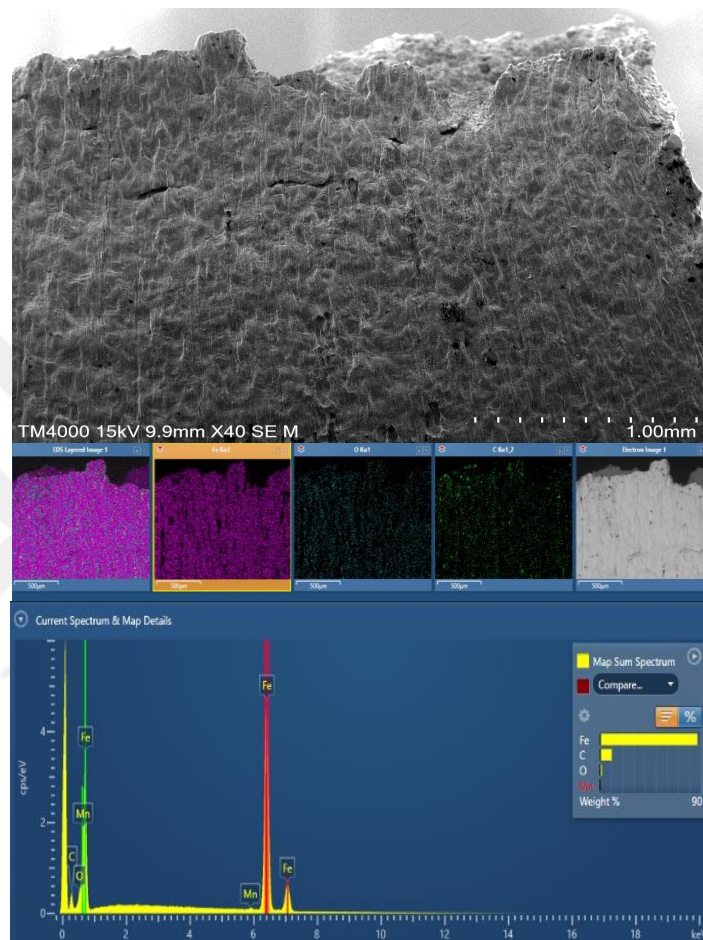


Figure 5.27 EDS Surface Views of Specimens 3.5 ID 4

In this study, the purpose of using the Scanning Electron Microscope is to determine whether the fractures started from the pits or not.

However, the corroded pits were formed in a narrower area for the specimens immersed to 1.8% NaCl solution. Moreover, they did not propagate along cross-section.

CHAPTER 6

CONCLUSIONS AND FUTURE WORK

6.1 Conclusions

From the present study the following points can be concluded:

1. Pitting corrosion behavior of structural steel St 37 with several corrosive environments have been investigated. Mainly, the effects of salinity rates were observed using two different solutions. According to this study, to detect the weight difference, a significant amount of pitting corrosion should be occurred. Thus, the weight of specimens did not change enough. But this low level of loss, which is the general characteristic of pitting corrosion, can quickly disable the material.
2. As seen in the optical microscope images we obtained in this type of corrosion, small cavities and pits were formed on the surface of the material. It showed that these cavities are proceeding almost perpendicular to the metal surface.
3. The pH value of solutions did not change with Cl^- concentration. Thus, pH value doesn't be a significant parameter on the pitting corrosion.
4. The increase in chloride ion concentration changed the morphology of pits.
5. The tensile test results did not decrease with increase of salinity as unexpected. Because of the higher pit area of the specimens in 1.8% NaCl solution, effective area of these specimens decreased in a higher value than effective area of specimens in 3.5% NaCl solution, lowering the tensile properties of these specimens.
6. The specimens immersed to the 1.8 % solution had large number of pits on their surfaces after they were corroded and the pits average sizes were nearly 65-75 μm . But, the specimens immersed to 3.5% solution had few number of pits on their surfaces after they were corroded and the pits average size were nearly 90-100 μm and this results showed that the small and large number of pit reduced strengths unexpectedly.

6.2 Future Work

1. St 37 metal, used for several structures, was investigated according to the corrosion resistance with experiments and to determine the most suitable environmental conditions was the main purpose. This purpose was tried to explain with some studies and supported with literature searches.

2. Thus, selecting the material and environment conditions properly causes to increase the lifetime that results to decrease the effect of corrosion to the structural metals. Therefore, these precautions will contribute to the national economy.

3. Similarly, avoiding the expensive repairs, economy can make savings from the labor force. To investigate pitting corrosion resistance, more specimens should be used; the pitting morphology of these specimens should be observed carefully.

4. The depth of the pits is important to evaluate the pit morphology and to predict tensile properties of the material. Therefore in order to determine the depths of the pits, more advanced instruments like profilometer is needed for further studies.

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- Reinforced Concrete

