

# Investigate of SCC of Low Carbon Steel in 0.1M Sulfuric Acid Solution Using Tension Test Method

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**Abstract:** *The aim of this work is to investigate the effect of applied stress on the commercial Low Carbon Steel corrosion process using tension test method. A suitable metal frame was used to build the testing system. The applied stress effect was obtained by using a flexible calibrated spring.*

*A comparison between specimen's behavior under stress effect and without was studied. The weight loss and polarization methods were used for corrosion rate calculation. A necessary optical photo to the fracture surfaces was taken and graphs were plotted according to the obtained data. The metals used were found to be susceptible to SCC. The stress effect and acidic solution accelerate the failure or fracture points because of increasing cracks propagation which may occurs through machining or from corrosion process effect. The Sulfuric Acid solution is a high corrosive media for Low Carbon Steel materials.*

**Keywords:** *Stress Corrosion Cracking, Corrosion rate, Acid Environments, Low Carbon Steel, Weight Loss Method.*

## 1. Introduction

The corrosion can be defined as the destructive attack of a material by reaction with its environment. Corrosion is the damage to metal caused by reaction with its environment and can lead to failures in factories infrastructure and machines [1, 2]. Nearly all of the environments in which metals serve are potentially hostile and their successful use in commercial applications and engineering depends on protective mechanisms [3].

Corrosion cannot be defined without a reference to environment. All environments are corrosive to some degree. It may, therefore, be observed that corrosion is a potent force which destroys economy, depletes resources and causes costly and untimely failures of plants, equipment and components [4].

Corrosion of Metals in aqueous environments is electrochemical in nature involving two or more electrochemical reactions taking place on the metal surface [5].

The metals are extracted from these ores after supplying a large amount of energy. Metals in the

uncombined condition have a higher energy and are in an unstable state. It is their natural tendency to go back to the low energy state. This is the main reason for corrosion [6].

To understand corrosion reactions one needs to study electrochemical thermodynamics and electrochemical kinetics of the partial reactions. In other words, corrosion is essentially an electrical circuit wherein electrons flow from the anode to the cathode. When electrons flow from the anode, the anode oxidizes to form metallic ions. These metallic ions then detach from the metal surface to flow toward the cathode through the electrolyte. Throughout this process the metallic ions may also react with elements in the electrolyte to form other compounds [7, 8].

### 1.1 Stress Corrosion Cracking

Stress corrosion cracking (SCC) is a process by which cracks propagate in a metal or alloy by the concurrent action of a tensile stress (residual and/or applied) and a specific corrosive environment. Environmental species are often specific to the alloy system and may not have an effect on other alloys of different type. Not all environments cause cracking of any particular alloy, but new alloy-environment combinations resulting in SCC are being discovered on a regular basis [9].

Stresses on the metal increase its corrosion, these stresses create stress cells of course, but more importantly they disrupt the surface film that has protected the metal to some extent.

Material variables are uniquely important in determining SCC resistance [10]. In metals, slip bands consisting of innumerable dislocations make the surface near a crack tip rough with micro-cracks. Consequently, the passive thin layer is broken, exposing the material to the corrosive atmosphere. In fact, the size of the plastic zone is not important because only the plastic deformation near the crack tip is influenced by the corrosion. Thus, the rate of crack growth does not depend much on increasing stress intensity factor with the advancement of the crack length [11].

The crack surface in the vicinity of a crack-tip, becomes the anode of the electrolytic cell formed.

If the crack surface of steel component is wet with water, the iron ions in the vicinity of the crack tip dissolve, leaving excess electron on the surface. Thus the crack surface near the tip starts acting as the anode of the electrolytic cell as modeled in Figure 1. Far away from the crack tip, the passive layer works as the cathode of the cell. The excess electrons on the anode pass through the conducting body of the component to the cathode surfaces. These electrons then reduce the hydrogen ions of the surrounding water, resulting into the evolution of hydrogen gas. Some of these hydrogen atoms may diffuse into the component to cause hydrogen embrittlement.

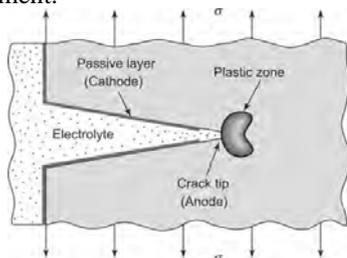


Figure 1. Electrolytic cell formed near the crack tip

A range of test methods is available in the form of international and national standards and procedures, comprising a wide variety of test methods for assessing SCC susceptibility and also a variety of specimen configurations [12].

The applicable types of tests could be classified, according to the stressing modes applied to the test specimens, in to: (*Constant total strain, Constant load and Constant strain rate Stress Corrosion Cracking*).

Specimens used to determine tensile properties in air are well suited and easily adapted to SCC, as discussed in ASTM G49. When uniaxially loaded in tension, the stress pattern is simple and uniform. The magnitude of the applied stress can be accurately determined. Specimens can be quantitatively stressed by using equipment for application of either a constant load, a constant

strain, or an increasing load or strain. This type of test is one of the most versatile methods of SCC testing because of the flexibility permitted in the type and size of the test specimen, the stressing procedures, and the range of stress level. It allows the simultaneous exposure of unstressed specimens (no applied load) with stressed specimens and subsequent tension testing to distinguish between the effects of true SCC and mechanical overload [13]. A constant-load system can be modified by the use of a calibrated spring, such as that shown in Figure 2.

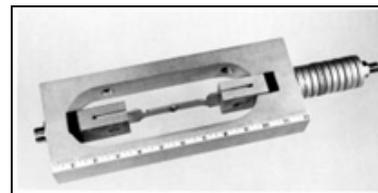


Figure 2. Spring-Loaded Stressing Frame [14].

## 2. Experimental Work

### 2.1. Materials and Specimen's Preparation

The materials used in this work are: (Commercial Low Carbon Steel, Sulfuric Acid with concentration equal to 0.1M, 10% HCL solution and distilled water and Natal solution with concentration 2%).

The specimens were prepared by cutting a standard rectangular sheet of Low Carbon Steel and make grinding and polishing process before machining with Silicon Carbide (SiC) emery paper with grit size of 240, 320, 400, 800, 1000 and 1200, then polished it to obtain a mirror finish surface, free from scratches. The specimen chemical composition was founded using XRF test which illustrated in Table 1. The specimen's net weight was determined using sensitive balance with 0.0001 g accuracy.

The final specimen shape for this work was shown in Figure 3.

Table 1 Chemical Composition of Low Carbon Steel Using XRF test

Materials	Fe wt%	Cr wt%	Mn wt%	Cu wt%	Si wt%	C wt%
L.C.S.	Bal.	0.14	0.48	0.19	0.48	0.14

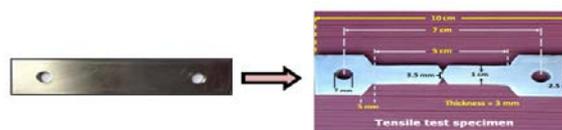


Figure 3. Rectangular shape metal plate and the final specimen tensile test.

In this work a similar frame was machined using metal square frame with dimension (22x22x2 cm) as shown in the Figure 4.

A suitable metal clamps are machined to use it to fixed the specimen's ends. In the upper clamp we use screw with 7in. length and 1.2cm as a

diameter. A suitable spring with high flexibility with 11cm length and 4.5 cm as a diameter to use it for applied stress by make the screw passed through it and a suitable nut to fix this spring.

The electrolyte solution placed in plastic cylindrical container with 11cm as a diameter fixed

on circular wooden base and the specimen passed this plastic container and electrolyte solution.

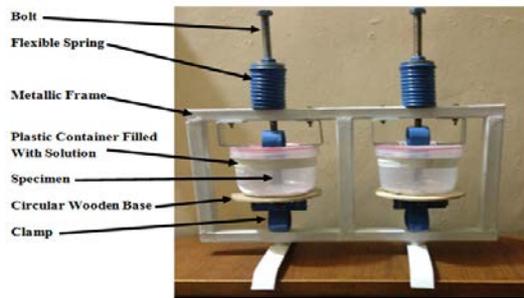


Figure 4. Machined spring-loaded stressing frame

Before using the spring in the frame test, a calibration must be done to study its flexibility and to know what the value of ultimate load can this spring applied when we screw it, in other word testing its mechanical properties by tensile test using Instron 1122 machine must be done. Figure 5 showed this spring and plots of its calibration test and its geometrical properties are illustrates in Table 2. From this test the maximum load which applied to the specimen equal to 2341 N.

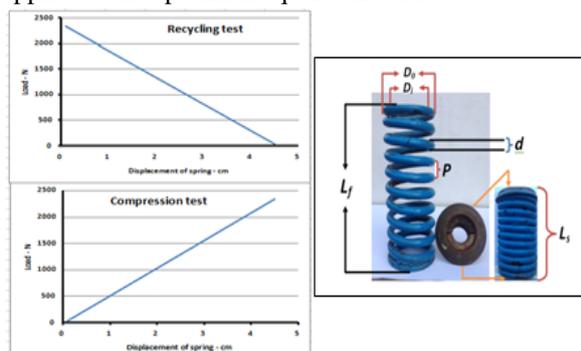


Figure 5. Compression and Recycling test plots of the spring using Instron 1122 machine.

## 2.2. Experimental Procedure

### 2.2.1. Weight Loss Method

In order to know the failure or fracture time of the different specimens with variant notch dimension and the same metal type, three specimens were chosen with different notch width 2.5, 3.5 and 4.5mm to make this test with 0.1M of Sulfuric Acid as an environment using the system shown in Figure 4, and another three specimens from the same metal type and with 2.5, 3.5, and 4.5 mm too as a notch width were used but without applied stress in order to make a comparison between these two cases (with stress and without stress). The initial weights for all specimens ( $W_1$ ) were determined and then fixed it in the system test using maximum applied load (2341N) for the first three specimens, and fixed the other three without load.

The observation of these specimens behavior at any time as possible must be done to notice any changes on it.

After fracture process happened, and the specimens separates into two parts, we get out its parts, then cleaned it using HCL solution and distilled water with soft brush to remove the corrosion contamination, then drying it and determine its final weights ( $W_2$ ), and make the etching process using Natal Solution in order to take the optical photos.

The relation for corrosion rate calculation using this method is:

$$C.R. (mmpy) = \frac{K \Delta W}{D A T} \dots\dots\dots 1$$

Where:

$$K=8.76 \times 10^4, \Delta W = \text{weight loss in (g)},$$

$$D = \text{metal density in (g/cm}^3\text{)},$$

$$A = \text{exposure area to corrosion in (cm}^2\text{)},$$

$$T = \text{time of exposure in (hours)}.$$

### 2.2.2. Electrochemical testing method

In order to make a comparison between the weight loss method and polarization method, the potentiostatic test cell was used which shown in Figure 6 for one specimen which notch width equal to 3.5 mm. This test was done with two conditions too. The first is under stress effect and the second is without. After test end the Tafel Plot was obtained and the corrosion rate was determined using it. The data of this test was illustrated in Table 3. The corrosion rate in (mm/y) was calculated from the relation 2.

$$CR = K \frac{i_{cor}}{\rho} EW \dots\dots\dots 2$$

Where:

$$K = 0.00327, i_{cor} \text{ is corrosion current density in } (\mu A/cm^2), EW = \text{equivalent weight}, \rho = \text{density in (g/cm}^3\text{)}.$$

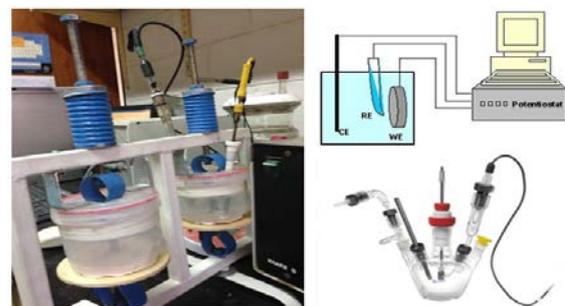


Figure 6. Polarization testing cell

## 3. Results and Discussion

### 3.1. Weight Loss Method

After 19 days, the specimen (1) was fractured because of its notch width was the lowest one between specimens used. The other two (2 and 3) were fractured after 26.25 and 38.5 days respectively. The other three specimens which used

without stress were removed after 48 days without fracture. The data of this test were illustrated in Table 3-a and b.

When applied stress affected on the specimen's ends, the surfaces near the notch edge will be as a region of stress concentration, so the micro cracks will occurred and with the sulfuric acid solution effects on this region by corrosion process phenomena will cause cracks propagation near any pits or local defects in the direction perpendicular to the stress effect axis, although there are residual stresses near these micro cracks or pits because of the components difference which will increase the cracks propagation under acid solution effects.

The first three specimens were split into two pieces or there is a fracture process was happened, and this is an evidence of stress effect on cracks propagation acceleration until fracture. The corrosion in the notch of specimens under stress was happened in the direction of the notch angel end more than other regions which will cause the fracture process, but in the other specimens without stress effects this process was occurred in many directions randomly, that's caused the specimen not to be fractured at the same time in the others.

The optical photos of these specimens were shown in Figures (7, 8, 9, 10, 11, and 12).

The corrosion process affected the mechanical properties of the specimen metal. The corrosion will make it weak or the strength of the specimen will decrease. The corrosion rate and the failure time of the specimen increased with increasing of notch width because of increasing of the surfaces area which affected from corrosion.

In general, corrosion rate and weight loss for the two groups of specimen was increased with notch width increasing because of increasing of the corrosive area. In other hand the corrosion rate of the first group under stress effect was greater than

the second without stress effect because the stress accelerate failure process when it make cracks propagation quicker and initiate more cracks and then the corrosion process will cause pits initiation and will be as a cracks source which will cause a failure process.

### 3.2. Polarization Method

Using relation 2, corrosion rate was calculated from corrosion current density ( $i_{corr}$ ) which obtained from Tafel Plot as shown in Figure 15 and 16. These values were illustrated in Table 4. The corrosion rate of specimen under stress effect was greater than the other without stress effect because of increasing of corrosion current density which depends on corrosion velocity. The difference between the two methods for corrosion rate calculation was illustrated in Table 5. There are small difference between them because the polarization cell was not standard which we can used in chemical libraries which shown in Figure 6.

### 4. Conclusion

All the results can be concluded as follows:

The corrosion process is activity in defects region in some areas more than other areas and this is depending on the type of defects and the value of stress. So, in this region the cracks will began to create, and will represent the source of failure of the metal under continuity of the stress effect and corrosion process.

The sulfuric acid solution is a high and more effectiveness corrosive media for low carbon steel metals because the value of the highest Iron percentage value in the specimens metals instead of the lowest (Cr) and (Mo) elements percentage. The stress effects accelerate the failure or fracture point because it's increasing the cracks propagation and growth, and is the same effect for the acidic solution.

**Table 2 Main parameters of spring which used in the metal frame of test**

Parameter	Meaning	Value
$D_o$	Outer diameter of spring coil	4.11 cm
$D_i$	Inner diameter of spring coil	2.98 cm
$D = (D_o + D_i)/2$	Mean diameter	3.545 cm
$L_f$	Free Length - Maximum length of spring without applied stress	11 cm
$L_s = d(N_a + 2)$	Solid Length - Minimum length of spring under maximum applied stress.	6.6 cm
$d$	Wire diameter	0.6 cm
$N_t$	Number of total coils	11 coil
$N_a = N_t - 2$	Number of active coils	9 coil
$P$	Pitch - distance from center to center of the wire.	1.09 cm
$F$	Maximum load	2341 N

**Table 3-a Weights of specimens and fracture times with maximum applied stress.**

Specimen No.	Notch Width (mm)	Weight $W_1$ (g)	Weight $W_2$ (g)	$\Delta W$ (g)	F. T.* (hour)	C.R. mm/y
1	2.5	38.8601	38.1098	0.7503	456	2.5084

2	3.5	40.3730	39.1526	1.2204	630	2.9531
3	4.5	43.8904	41.6575	2.2329	924	3.6840

**Table 3-b Weights of specimen and removing time without applied stress.**

Specimen No.	Notch Width (mm)	Weight $W_1$ (g)	Weight $W_2$ (g)	$\Delta W$ (g)	R. T.* (hour)	C.R. mm/y
1	2.5	39.4159	39.0271	0.3888	1152	0.5145
2	3.5	39.1204	38.5286	0.5918	1152	0.7832
3	4.5	43.5681	42.8691	0.6990	1152	0.925

\* *F. T. Failure Time or Fracture Time.*

\* *R. T. Removing Time.*

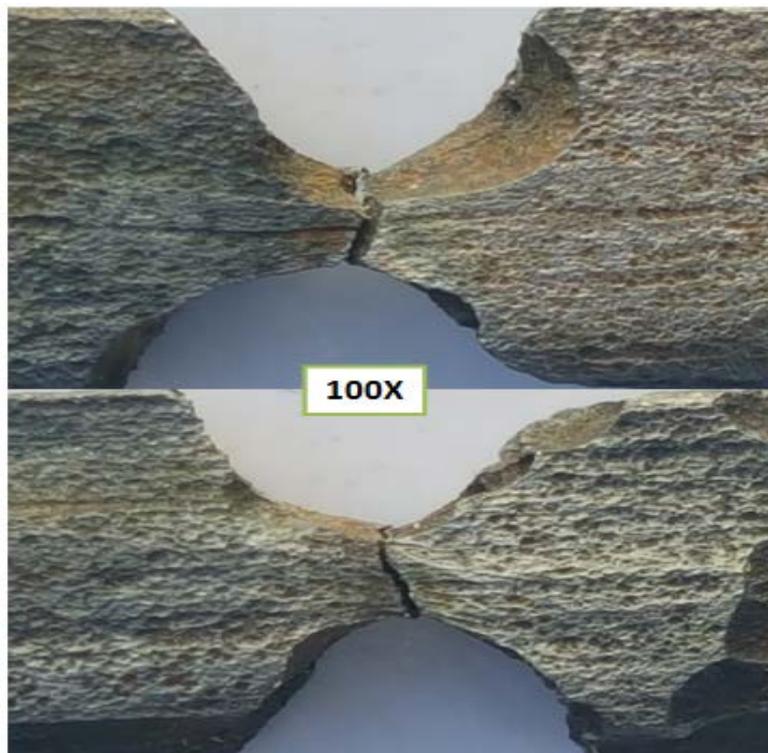


Figure 7-a 2.5 mm notch width specimen fractured after 19 days

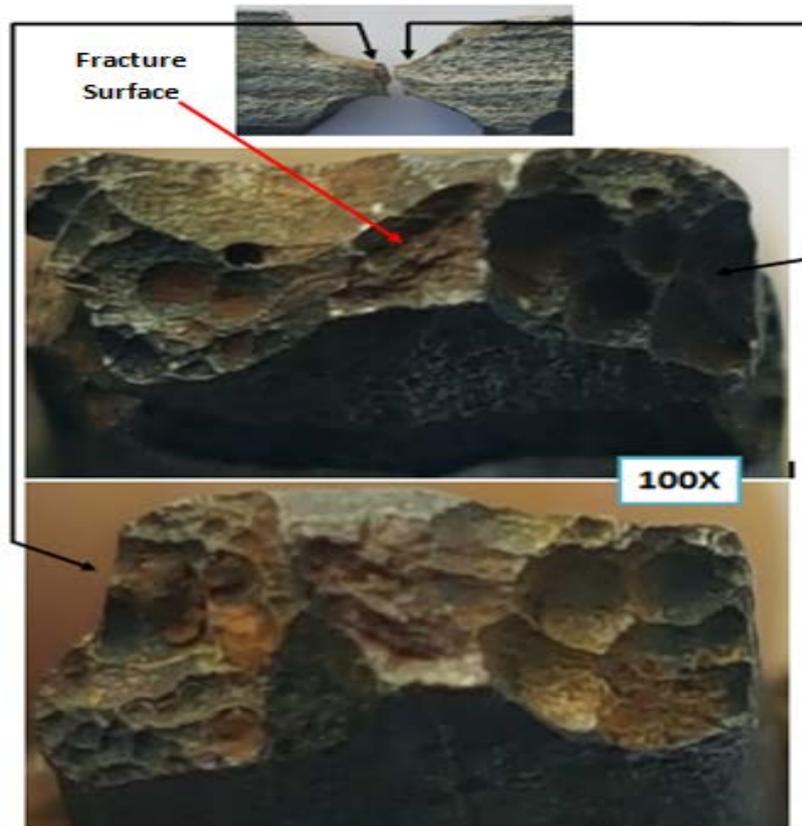


Figure 7-b Surfaces of fracture of 2.5 mm notch width specimen after fracture

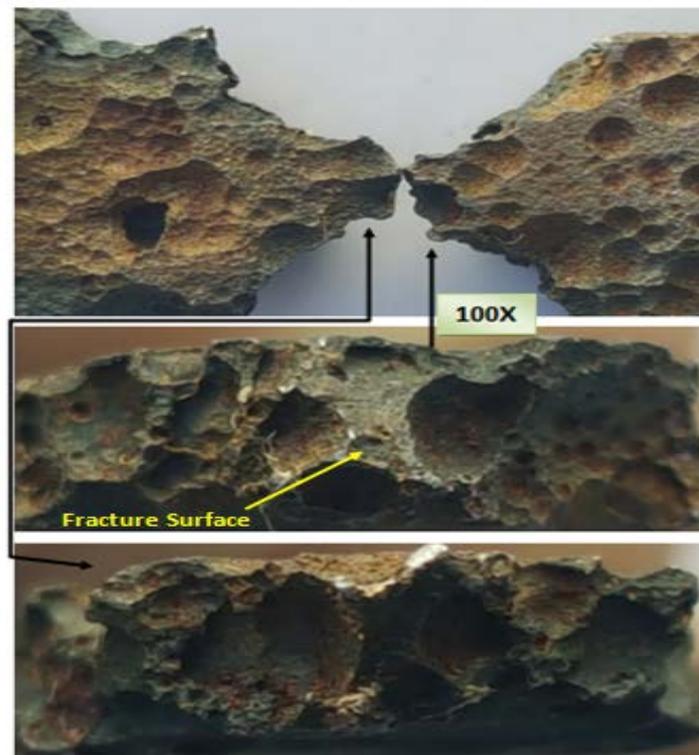


Figure 8. 3.5 mm notch width specimen fractured after 26.25 days

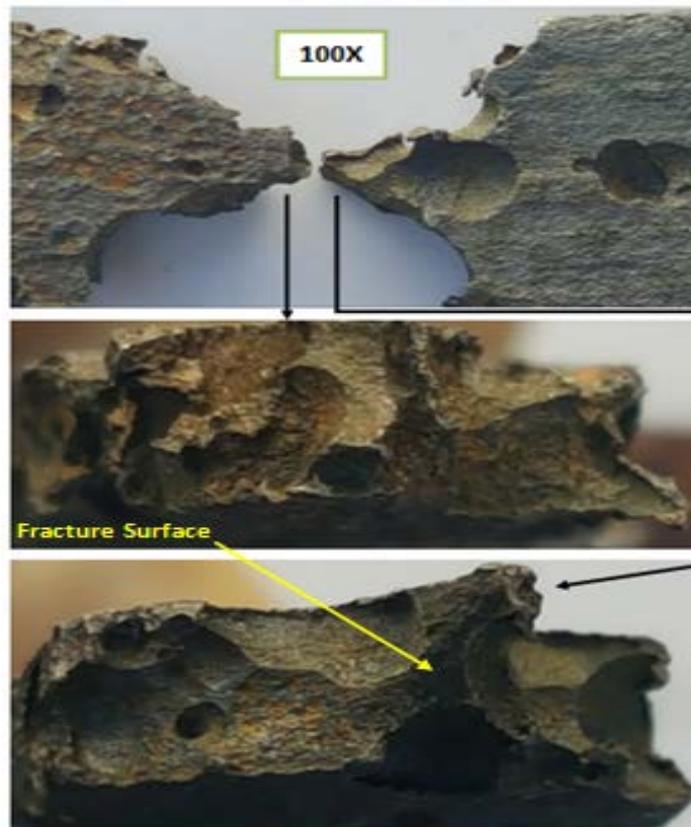


Figure 9. 4.5 mm notch width specimen fractured after 38.5 days

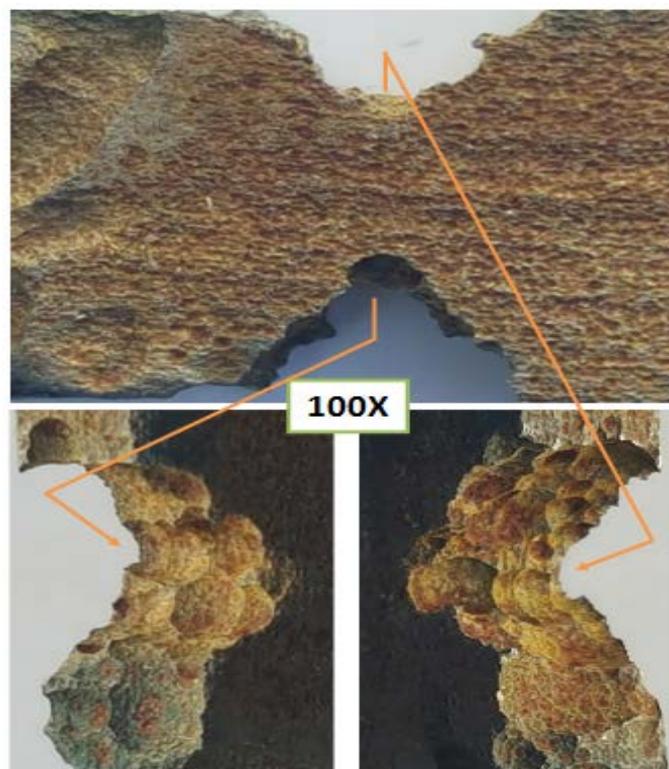


Figure 10. Corrosion region of the 2.5 mm notch width specimen *without applied stress effect* after 48 days

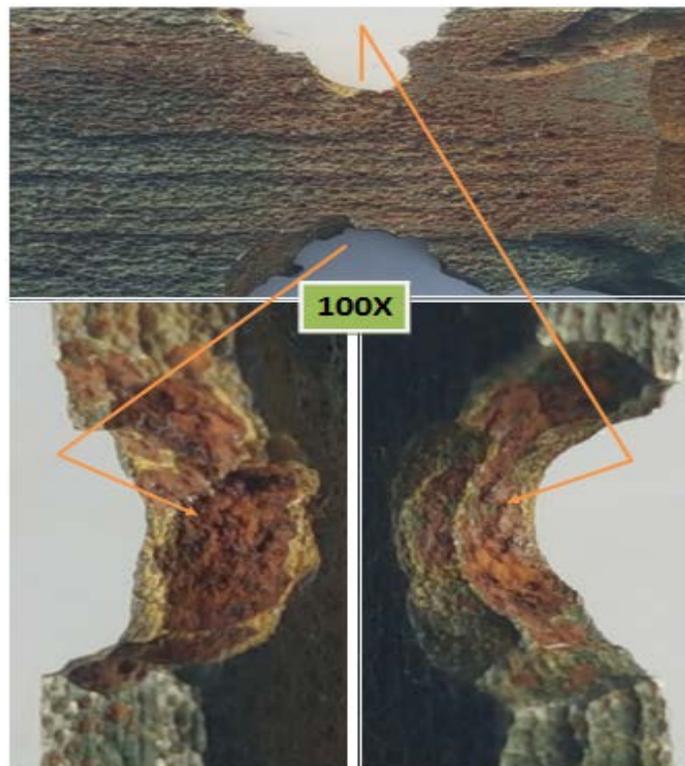


Figure 11. Corrosion region of the 3.5 mm notch width specimen *without applied stress effect after 48 days*

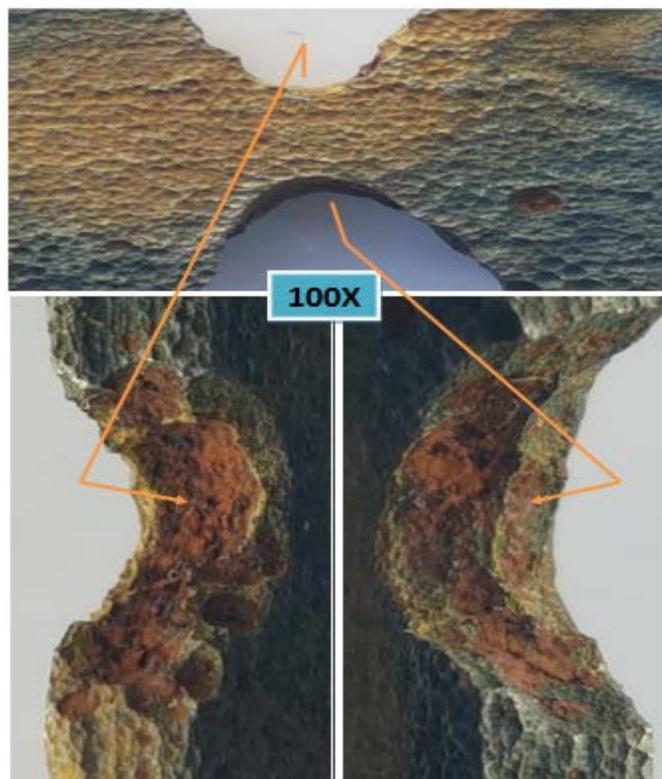
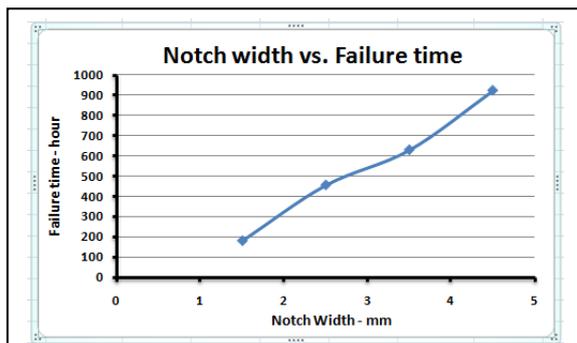
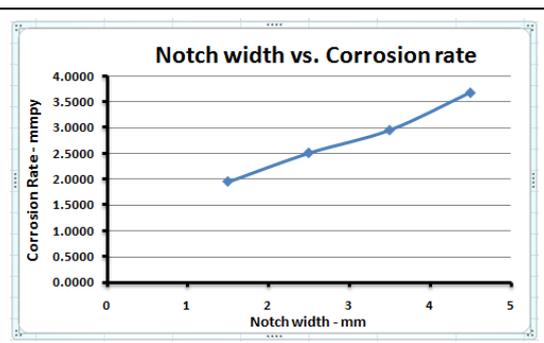


Figure 12. Corrosion region of the 4.5 mm notch width specimen *without applied stress effect after 48 days*

The relation between notch width and failure time was shown in Figure 13 and between this width and corrosion rate in Figure 14.



**Figure 13 the relation between notch widths vs. failure time**



**Figure 14 the relation between notch widths vs. corrosion rate**

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