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Effect of the Heat Treatments on Corrosion and Erosion-Corrosion for Carbon Steel

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Abstract

The aim of present work is to study the effect of different heat treatments [full annealing ,normalizing ,and hardening and tempering at (300C° ,600C°)] on general corrosion and erosion-corrosion for a carbon steel type (C35) in 3.5wt% NaCl solution as corrosive medium for general corrosion while in erosion-corrosion ,slurry with 1wt% SiO_2 sand was added to 3.5wt% NaCl solution as the erodent. The experimental work tests were done using special device which was designed and manufactured according to (G73 ASTM) using traditional weight loss technique to measure weight loss rate or corrosion rate in (gmd) unit.

It was found that the general corrosion rates for all heat treated specimens were lower than erosion-corrosion rate. The erosion-corrosion rate of specimen which was quenched and tempered at 600C° was found smaller than other heat treated specimens The normalizing heat treatment of low carbon steel improves the general pure corrosion resistance after immersion 20 day in 3.5 wt% NaCl solution.

Keywords: corrosion ,heat treatments ,erosion-corrosion ,carbon steel

تأثير المعلاملات الحرارية على التأكل العام والتأكل بالتعرية لفولاذ كاربوني

الخلاصة

يهدف البحث الى دراسة تأثير المعاملات الحرارية المختلفة (التلدين التام، المعادلة، التقسية والمراجعة عند درجة حرارة (300C°, 600C°)) على التأكل العام والتأكل بالتعرية لفولاذ كاربوني نوع (C35) في وسط ملحي من (3.5wt% NaCl) في حالة التأكل العام ومحلول عالق من رمل السليكا بنسبة (1wt%) كمادة تعرية في محلول كلوريد الصوديوم (3.5wt%) في حالة التأكل بالتعرية. في حالة التأكل بالتعرية أجريت التجارب المختبرية بأستخدام جهاز خاص تم تصميمه وتصنيعهُ أستناداً الى المعيار العالمي (ASTM G73) وبأستخدام تقنية فرق الوزن لقياس معدل فرق الوزن أو معدل التأكل بوحدة (gmd). كذلك درست تأثير هذهِ المعاملات الحرارية على البنية المجهرية والخواص الميكانيكية(الصلادة ومقاومة الصدمة) لهذهِ العينات.

وقد وجد أنه معدل التأكل العام لجميع العينات أقل من معدل التأكل بالتعرية كذلك أبدت العينات المعاملة حرارياً (المعادلة) أقل وزن مفقود وأفضل مقاومة للتأكل العام بعد مرور 20 يوم من غمرها في ماء البحر (3.5% NaCl). كذلك وجد أن العينات المعاملة حرارياً بالتقسية والمراجعة في 600 ^{o}م يكون معدل التأكل بالتعرية أقل من بقية العينات المعاملة حراريا .

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Introduction

Erosion-Corrosion is the acceleration or increase in the rate of deterioration or attack on a metal because of relative motion between a corrosive fluid and the metal surface, this movement is quite rapid ,and mechanical erosion effect or abrasion are involved. All types of equipments exposed to moving fluids are subjected to erosion-corrosion. Examples are piping systems particularly, valves, pumps, blowers, impellers , heat exchanger tubing such as heaters, condensers ,measuring devices such as an orifice turbine blades, ducts ,nozzles, and vapor lines, and equipment subject to spray [1,2.3]. If the removal of oxide layer occurs in a corrosive liquid, the corrosion will be enhanced because a bare metal surface will exposed to the liquid for a while until the oxide layer is healed. This is called erosion-corrosion.

W.M.M. Huijbegts (1982)[4] studied the effect of chemical composition of steel on erosion-corrosion in wet stream. He showed that minute quantities of Cr, Cu, and Mo proven to increase the resistance against erosion-corrosion in the laboratory test. A.Toro et al (1999)[5] studied erosion-corrosion resistance of martensitic stainless steel AISI 410S (13%Cr , 0-0.08%C max) specimens which were nitrided at 1473 K under 0.25 Mpa high purity nitrogen atmosphere; and then oil quenched and tempered at 473K . Slurry wear testes were performed by placing the specimens in st.st vessel containing ocean water and

20% quartz particles with 0.3 – 0.5 mm diameter. They found that erosion-corrosion resistance of the nitrided and 473K tempered AISI 410S was better than that of the AISI 410 steels.

Ramarkrishna Malka (2005) [6] indicated that erosion- corrosion due to sand is an increasingly significant problem in petroleum protection , erosion and corrosion interact synergistically in a complex manner where predication of metal loss becomes difficult. Erosion- Corrosion experiments were done by using sand and CO_2 saturated water. In this work synergism obtained by comparing these erosion and corrosion components with the pure erosion and pure corrosion measurements. Steel was AISI 1018 steel.

Barker Hughes (2006)[7] indicated that metal loss in production equipment due to erosion – corrosion occurred when sand and /or particles caused abrasion. Studies show that damage caused by a combination of corrosion and erosion in greater than either problem alone.

The aim of this work is to study the effect of different heat treatments on corrosion and erosion-corrosion (with slurry and with out slurry) of a carbon steel.

Experimental Work

1- Material

The material used in this work is a carbon steel type C35 according to DIN standard (structural steel). It is machined and formed to pieces have square cross section(1x1 cm^2) and 55mm long .Table 1 shows the chemical composition analysis of that work piece.

2- Experimental Media

The pure corrosion and erosion-corrosion tests conditions are shown in the **Tables 2 and 3** under the effect of some parameters such as flow rate and PH at constant temperature (25C°) and media pressure at 1 bar**.**

3- **Corrosive media**

Two media were used in this work, first was seawater of 3.5wt% NaCl solution for general corrosion test at room temperature, 25C°,second media was mixture of 1wt%SiO_2 sand in 3.5wt%NaCl solution for erosion-corrosion test with slurry at 25C°.

Table 4 shows the chemical composition of the sand type AL-Ardhemah (Iraqi sand), particle size ranges are 53µm-350µm as shown in **Table 5** .

4- **Tests Apparatus Used**

Tests apparatus used in this study was similar to that proposed by Mksim Antonov and Margaret Stack. The principal scheme is shown in F**igure 1.**

Figure 2 shows schematic diagram of corrosion, erosion and erosion-corrosion device .

The apparatus was designed and manufactured in accordance with the test apparatus explained in American Standard (ASTM G73) and has many modifications were carried out by the researcher Mohammed A. Ahmed[8].

5- **Specimens Preparation for Corrosion Test**

The specimens were machined and formed to pieces have squire cross section (1x1 cm^2) and 55mm length for immersion test or weight loss method.

The specimens were ground or abraded in sequence on 180, 220 , 320 , 500 , and 1000 grades with emery paper of SiC and water for cooling the specimens . Then the specimens were polished using special cloth with alumina (Al_2O_3)suspension of particles size of 0.5µm in solution. The samples were washed with water and acetone , then dried with hot air and kept in a desecrater for 1 hr.

6- **Heat treatments**

The different heat treatments are used shown in **Table 6 .** All heat treatments were carried out in an electric furnace type Carbolite.

7- Microstructure and Hardness Tests

Similar procedures as mentioned in paragraph (5) for specimens preparation were carried out for microstructure and hardness tests. After the samples were washed with water and acetone, then dried with hot air, etching process was carried out using Nital etchant solution (2%HNO_3 acid and 98% alcohol) and then washed with water and alcohol and dried. The microstructure was analyzed by optical microscopy type Olympus provided with camera and computer.

Vickers hardness test was made by using Vickers hardness tester type (Einsingenbei U/M, Model Z323) under a static load of (1.0Kg) for(10-15)sec..

Three to five readings were taken on each sample and the average hardness (VHN) was found.

8- Impact Test

The Charpy V-notch impact test is an ASTM E23 standard [9].The notch is located in the center of the test specimen which is support horizontally at two points. The specimen receives an impact from a pendulum of a specific weight on the side opposite that of the notch as shown in **Figure 3**. The specimen fails in flexure under impact. The energy absorbed by the specimen when it receives the impact from the hammer denote to the impact energy.

9- Weight loss method

Weight loss method is used to find corrosion rate of general corrosion and erosion-corrosion rate in gmd (gram / meter square x day) unit as following:-

1. Cleaning the specimen carefully with drying paper.
2. Weight the specimen with using digital balance with accuracy ± 0.1 mg, type {DENVER Instrument}, certified according to ISO 9001, Germany.
3. Immersion the specimens in chamber of the test apparatus mentioned in section with test media for each type of test. These media are indicated in section (Corrosive Media).
4. Test media is injected toward the specimen surface for (t =10 min.), for erosion-corrosion test the flow rate is(Q = 36 L / min.) and injection pressure is (P = 1 bar) .This test is carried out for 1.5 hr .
5. The specimen is weighted again after the specified (10 min.) of exposure to the media. The weight is carried out after cleaning the sample with distilled water and a brush and then with alcohol, and drying the specimens using drying paper and specimen drier.
6. This process is repeated several times to get several readings. Corrosion rate and erosion-corrosion rate are obtained from weight loss law.

Results and Discussion

1 – Microstructure Results

Microstructure plays an important role in assessing metal behavior in different media such as corrosive medium, erosive medium, or erosive corrosive medium. The results of microstructure for different with and without heat treatment are shown in **Figures 4,5,6,7 and 8**

Figure 4 shows the microstructure of as received sample (without heat treatments), dark areas are pearlite; light areas are ferrite ;that structure .

Figure 5 shows the full annealed sample microstructure, subsequent furnace cooling will result in large grains of ferrite and small areas of coarse lamellar pearlite. That heat treatment produces the coarse grains.

Figure 6 shows the microstructure of normalized sample; the white ferrite network surrounding the dark pearlite areas. The net effect is that normalizing produces a finer and more abundant pearlite structure than is obtained by full annealing.

Figure 7 shows the microstructure of quenched sample and tempered at 300C°, the structure etches dark and unknown and black or not clear that is tempered martensite.

Figure 8 shows the microstructure of quenched sample and tempered at 600C°, the structure etches dark and unknown and black or not clear that is tempered martensite.

2- Impact Test and Hardness Results

The results of impact energy and Vickers hardness tests for different heat treatments are shown in **Table 7** The mechanical properties of a carbon steel after full annealing were 119HV hardness and (7.67 kgm) impact energy. The normalizing heat treatment increases the hardness and reduces the impact energy. This is due to refine the grains of ferrite and pearlite The mechanical properties of quenched sample and tempered at 300C° better than quenched and tempered at 600C°.

3- General Corrosion Results

Corrosion in fresh or salt water is always the result of an electrochemical reaction. The electrochemical reaction seems to denote a complicated phenomenon. As used to describe the corrosion of metals, the application of electrochemical theory allows one to separate the relatively complicated corrosion reaction into two simple parts: the anode reaction where the metal is oxidized, and the cathode reaction where the oxidizer is reduced. Neither of these reactions will occur without the other. Writing them as separate reactions is done only to describe how the overall corrosion reaction takes place [10].

3 –1 Weight Loss Rate Results

From **Figure 9** indicates the results of general pure corrosion of carbon steel in 3.5wt% NaCl after different heat treatment. It was seen that weight loss rate increases continuously with the immersion time in 3.5 % NaCl solution for all samples which are heat treated in different conditions. The normalized sample has the lowest weight loss rate after immersion times (5-20 day) as comparing with other heat treated samples. This is due to refine grain and better microstructure and lower residual stresses in sample. The normalizing heat treatment is used to improve microstructure and mechanical properties of carbon steel [11]. While annealing heat treatment produces coarse grain of pearlite and ferrite which is more corrosion than pearlite because it is the soft phase in steel. The metallurgical difference of phase in steel will be induce electrochemical potential difference which results galvanic corrosion between soft and hard phase in steel, thus this is steel easily affected by external media such as corrosion, erosion and erosion –corrosion[12] .

Figure 10 shows the relationship between the loss weight rate or corrosion rate in unit (gmd) with immersion time in 3.5wt % NaCl for different heat treatments , it is seen the weight loss rate increases with time because of removed of surface layer and oxide and formation of new surface exposed tocorrosion.

It has been shown that the samples with hardening and tempering have weight loss rate higher than the normalized sample but less than full annealed and as received sample This is due to formation hard and brittle phase (martensite) and little affected by corrosion (3.5wt % NaCl solution) media. The tempered sample at 600C° has weight loss rate lower than that of tempered 300C° because of martensite transform to tempered martensite and refine the martansite structure also removed relief stresses resulting from quenching.

3-2 Microstructure Results

Figure 11 shows microstructure of a carbon steel before heat treatment (as received sample) which consists of ferrite (α) and pearlite (P) phase. Ferrite phase is more corrosive than pearlite because it is soft phase and lower hardness, thus this phase is easily affected by corrosive media and erosion corrosion with and without slurry.

Figure 12 shows a metal surface exposed to pure corrosion in 3.5 % NaCl solution, pits are seen on the surface resulting from pitting corrosion with chloride ions. This is due to breaking of oxide film and the oxide film is porous, and non adherent as indicated by shrrier L.L. [13].

Figure 13, 14 and 15 show parts of metal surface are removed leaving dark, deep and large cavities with no definite form.

Furthermore, it removes the surface layer and produces new and naked surface exposed to corrosive medium which contains chloride ions and break carbonic acid

4- Erosion – Corrosion Results

Erosion is a progressive loss of original material from a solid surface due to mechanical interaction between the surface and a fluid or impinging liquid or solid particle [14]. Erosion-corrosion occurs because of relative movement between corrosive fluid and the metal surface. These results are confirmed by the researcher Huijbregts [15]. He showed that the erosion-corrosion depends on water chemistry, water velocity, and chemical composition of the steel. This report focuses on the relationship between the chemical composition of steel and erosion corrosion resistance. This relationship has been neglected in the past because water chemistry and velocity were thought to be the dominant factors.

4-1 Weight Loss Rate Results

In this work erosion – corrosion rate is increased when the silica particles suspension in liquid (slurry) are destructive from the stand point of erosion – corrosion.

Figure 16 indicates the results of erosion- corrosion of a carbon steel in slurry (1wt% SiO_2) in 3.5wt% NaCl solution after different heat treatments. It was seen that weight loss increases continuously with the immersion time for all samples which are heat treated in different conditions.The sample which was hardened and tempered at 600C° has the lowest weight loss after immersion times (0.5-2hr) as comparing with other heat treated samples.

It has been shown that the hardened and tempered sample at 600C° has weight loss rate lower than that of hardened and tempered sample at 300C° because of presence of hard and tough tempered martensite structure which resists the impingement of hard silica particles in 3.5wt%NaCl solution, also induces compressive residual stresses on the surface resulting from high speed of particles which impacts the surface and improves the erosion – corrosion resistance of the sample.

From **Figure 17** it is noticed the erosion – corrosion rate increases with time , the increase is slow in the early stages , when the silica particles bombardment of the surface increase the hardness of metal surface due to deformation and hardening the surface. From above mention figure it is seen that weight loss rate or corrosion rate in unit (gmd) increases continuously with exposed time as result of mechanical effect of pure erosion and electrochemical effect of corrosive medium. The combined effect of sand in the slurry will lead to pitting corrosion

It was shown that weight loss amount by erosion corrosion with slurry is greater than that weight loss amount by other factors. This is to erosion resulted from striking the metal surface by erosion medium (SiO_2 particles) suspended in 3.5 % NaCl solution .This comes in good agreement with Barik R.C. et al [16]. They studied erosion – corrosion of material. Loss resulted from mechanical damage as of result of erosion.

4-2 Microstructure Results

Figures 18 and 19 show parts of soft metal surface are removed leaving dark, deep, small and large cavities with no definite form. This is due to flow velocity of fluid especially in areas facing the nozzle. It can be seen that the picture of as-received sample (Figure 18) has very fine horse – shoe like traces like pitting. While the annealed sample picture (Figure 19) has extreme horse-shoe- like traces in flow stream direction, it means that this sample has very low resistance to erosion-corrosion effects.

From **Figures 20**, **21** and **22** it can be seen the metal surface is exposed to damage due erosion corrosion with slurry (1wt% SiO_2) and high velocity of the erosive corrosive medium which cavitations and impingement by sand particles.

It has been shown that the normalized sample picture (Figure 20) has not clear horse –shoe –like traces for erosion-corrosion. While the picture of tempered sample at 300C°(Figure 21) has light horse-shoe –like traces for erosion – corrosion and those traces are less than that of normalized sample. It can be seen also that the tempered sample at 600C° (Figure 22) has small and low traces of horse- shoe –like, it means that the this sample has the highest resistant to erosion-corrosion effects as compared to other treat treated samples.

Conclusions

1- Weight loss amount by erosion – corrosion with slurry is greater than the weight loss amount by general pure corrosion for all samples.

2- Samples with quenching and tempering heat treatments show better erosion – corrosion resistant than normalized and full annealed samples.

3- Normalized samples shows better general pure corrosion resistance than other heat treatment samples over a range of immersion times (5 – 20 day).

4- Annealed sample shows the lowest corrosion resistance and erosion – corrosion resistance during exposure times as compared to other heat treated samples.

5- The metal surface shows pits in case of general pure corrosion in 3.5wt % NaCl solution, and have deep dark and large cavities with no definite form in case of erosion –corrosion with slurry (1wt% SiO_2) in 3.5wt% NaCl solution.

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Table (1) the chemical composition analysis of a carbon steel type C35

Element	Concentration wt%
Fe	Rem
Cu	0.136
Mo	0.018
Cr	0.051
Ni	0.077
P	0.0087
S	0.037
Si	0.208
Mn	0.573
C	0.299
Sn	0.025

Table (2) General pure corrosion test conditions

Medium Type	Single – Phase :(Corrosive Medium)
Temperature (C °)	25 (Room Temperature).
Medium Pressure (bar)	Zero (Static Medium).
Flow Rate (Q) (l/min)	Zero (Static Medium).

pH	(4.4) By pumping CO_2 gas in sea water and without sand.

Table (3) Erosion – corrosion test conditions

Medium Type	Two-Phase :Sea water (Corrosive Medium)and 1wt% silica sand as slurry(Erosive Medium)
Temperature (C °)	25 (Room Temperature).
Medium Pressure (bar)	1
Flow Rate (Q) (l/min)	36
pH	(4.4) By pumping CO_2 gas in sea water and with sand.

Table (4) Illustrates chemical composition of the used sand in wt%.

SiO_2 %	**CaO %**	**MgO %**	**AL_2O_3 %**	**Fe_2O_3 %**	**SO_3 %**	**CL %**	**Na_2O %**	**K_2O %**
98.37	0.56	0.06	1.06	0.16	Very low	0.04	0.12	0.15

Table (5) Illustrates percentage of weight and size of the used sand.

No.	**wt % of each sieve No.**	**Sieve No. (particle size) mm**
1	15.724	Over 0.355
2	28.259	Over 0.250
3	40.114	Over 0.150
4	12.324	Over 0.106
5	3.072	Over 0.075
6	0.373	Over 0.053
7	0.324	Over Bottom

Table 6

Heat Treatment	**Temperature C°**	**Cooling Medium**	**Time of Heating**
Full annealing	**860**	**Furnace**	**½ hour**
Normalizing	**860**	**Air**	**½ hour**
Hardening	**860**	**Water**	**½ hour**
Tempering	**300**	**Air**	**1 hour**
Tempering	**600**	**Air**	**1 hour**

Table 7 Results of impact and hardness tests

ample No.	Heat treatment type	Impact energy Kg.m	Hardness HV Kg/mm^2
1	As received	7.18 kgm	216
2	Full annealing	7.67 kgm	119
3	Normalizing	7.32 kgm	245
4	Quenching + tempering at 300C°	5.57 kgm	312
5	Quenching + tempering at 600C°	6.05 kgm	266

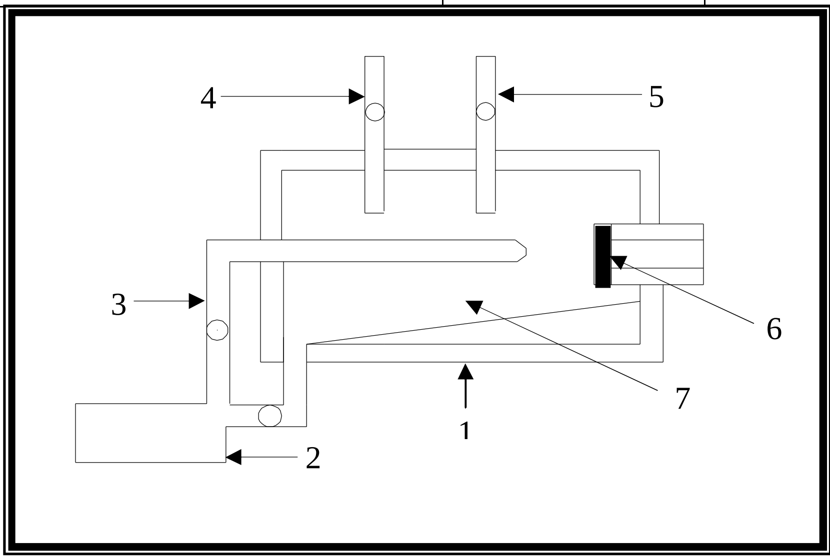


Figure (1) Illustrates corrosion and erosion-corrosion device

1	Tank Perspex (plastic) Tank
2	1 H.P Motor
3	P.V.C PIPE
4	Outlet gas pipe
5	Inlet gas pipe
6	Metal specimen
7	Effective Media

Figure (2) Schematic diagram of corrosion, erosion and erosion-corrosion device

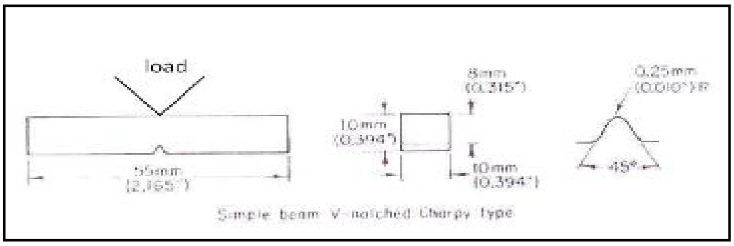


Figure (3) The Charpy V-notch impact specimen Dimensions [9]

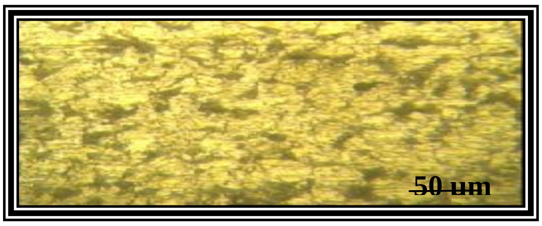


Figure (4) Microstructure of as received sample(without heat treatments), dark areas are pearlite; light areas are ferrite

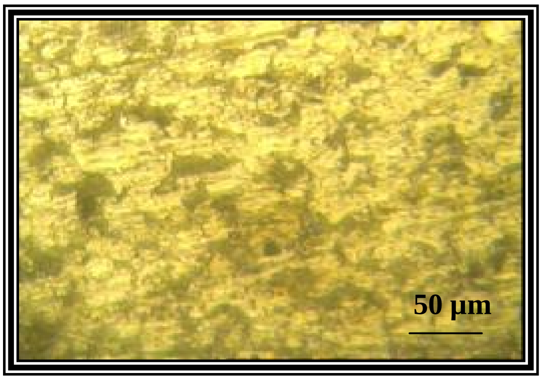


Figure (5) Microstructure of full annealed sample, large grains of ferrite and coarse lamellar pearlite

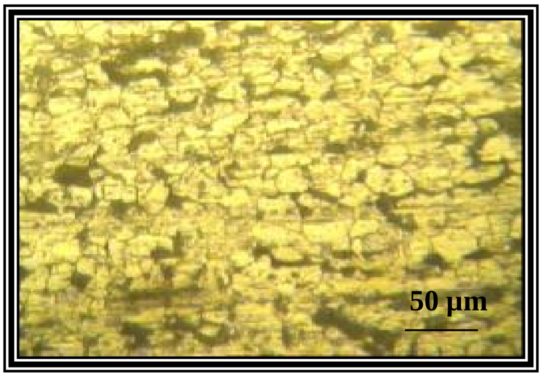
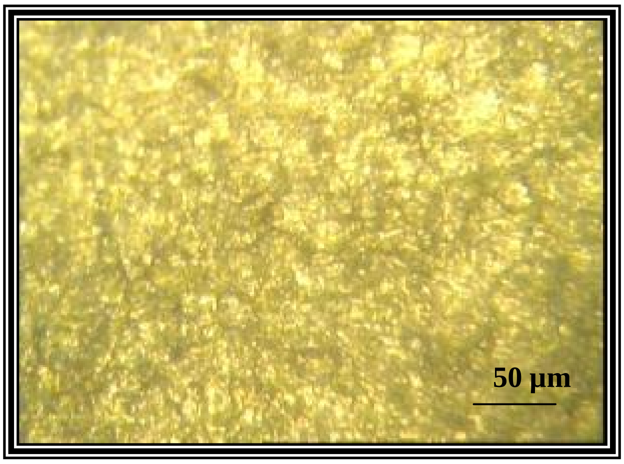


Figure (6) Microstructure of normalized sample; the white Ferrite network surrounding the dark pearlite areas.



Figure(7) Microstructure of quenched sample and tempered at 300C°,the structure is tempered martensite,

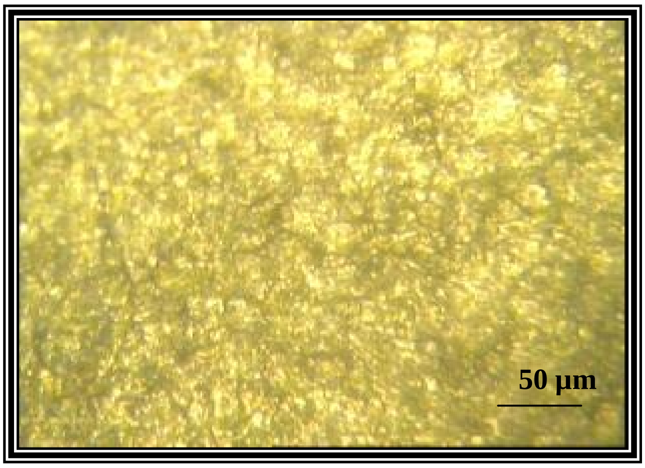


Figure (8) Microstructure of quenched sample and tempered at 600C°,the structure is tempered martensite

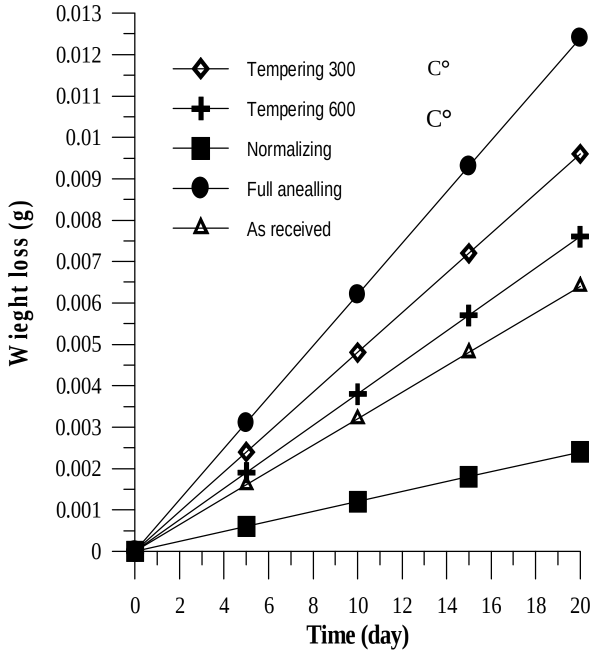


Figure (9) Weight loss - immersion time for general corrosion in 3.5% NaCl solution

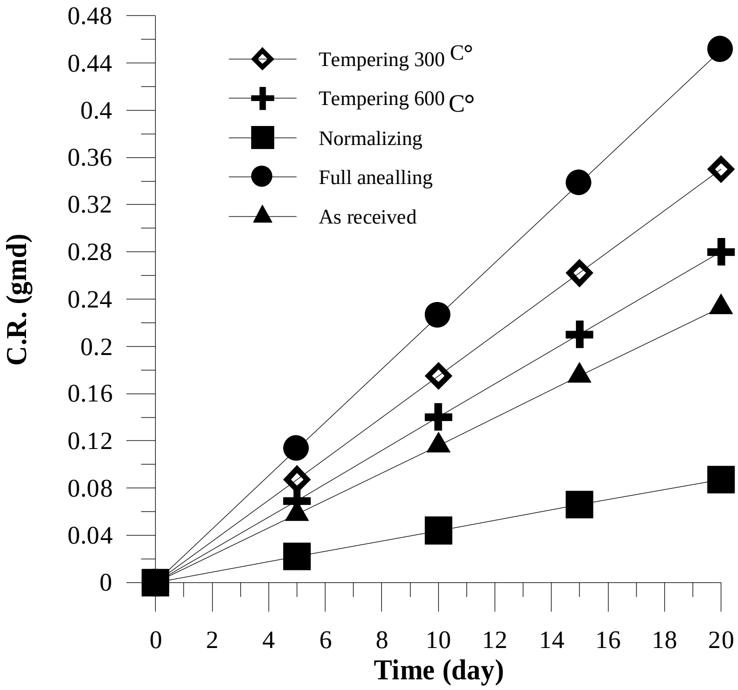


Figure (10) General corrosion rate – immersion time in 3.5% NaCl solution

Microstructures After Generel Pure Corrosion

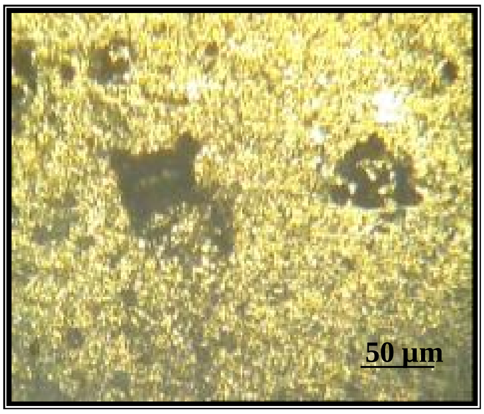


Figure (11) As received sample

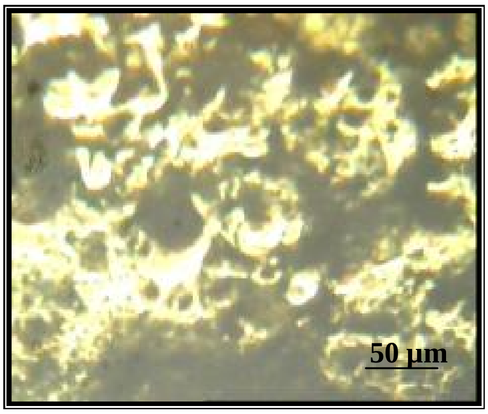


Figure (12) Full annealed sample

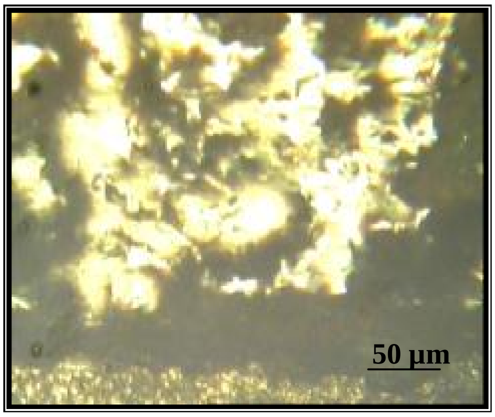


Figure 13 Normalized sample

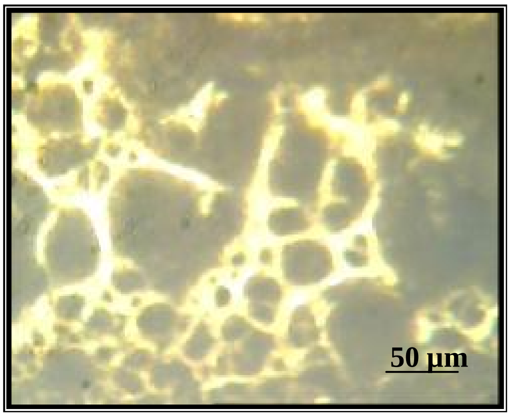


Figure 14 Tempered sample at 300C°

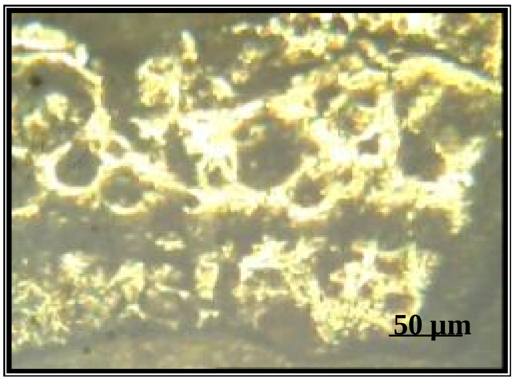


Figure 15 Tempered sample at 600C°

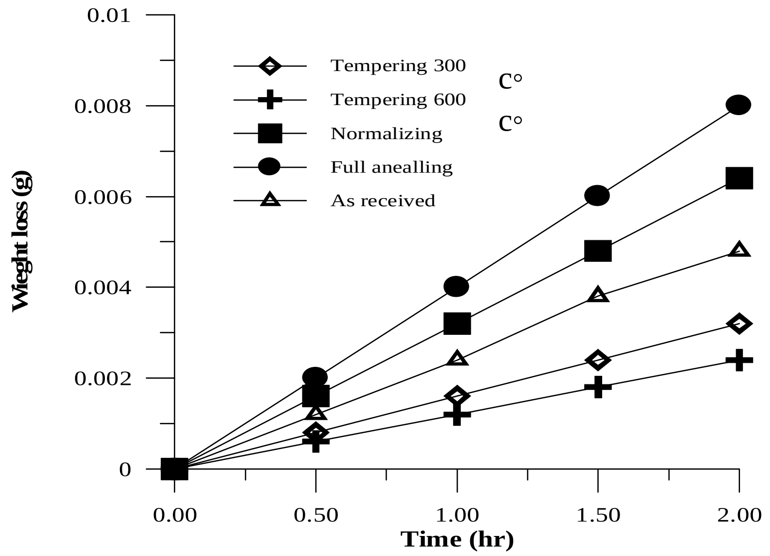


Figure 16 Weight loss - immersion time for erosion –corrosion in slurry of (3.5%NaCl+1%SiO_2)

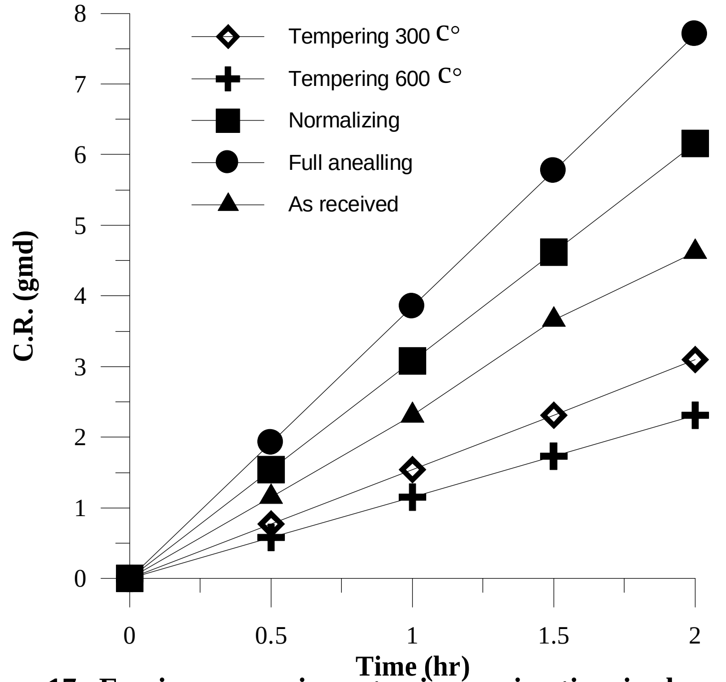


Figure 17 Erosion –corrosion rate - immersion time in slurry of (3.5%NaCl+1%SiO_2)

Microstructures after Erosion- Corrosion

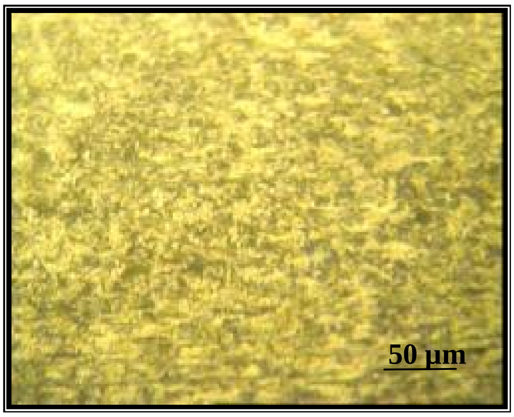


Figure 19 Full annealed sample

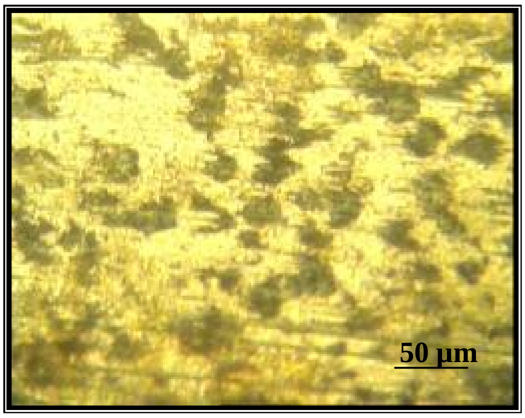


Figure 18 As received sample

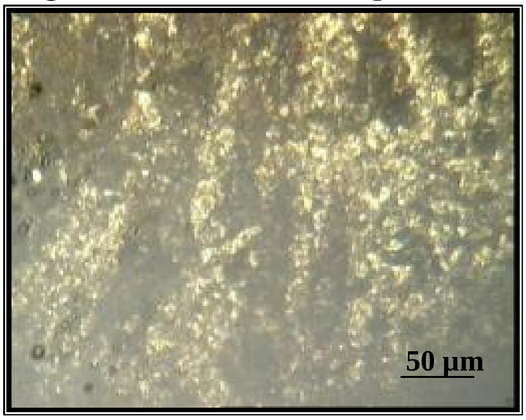


Figure 20 Normalized sample

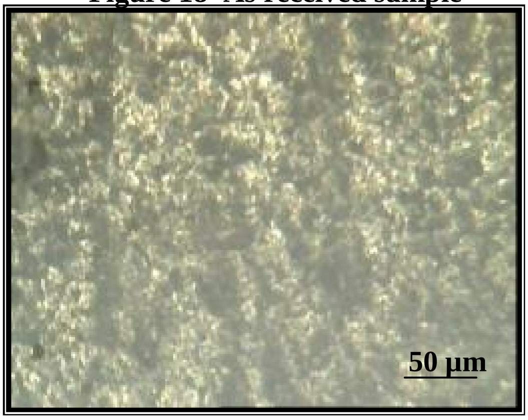


Figure 21 Tempered sample at 300C°

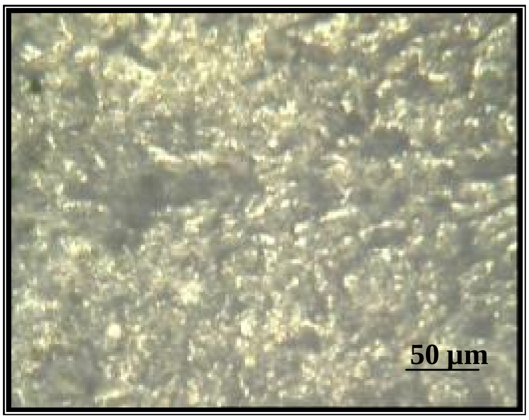


Figure 22 Tempered sample at 600C°