

كلية التسراث الجامعة

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Effect of Atmospheric Temperature on SCC of low Carbon steel in 0.1M of sulfuric acid solution using U-Bending test Dr. Kamal Hameed Gati Al-Turath University - College of Sciences – Iraq - Baghdad

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Abstract

The effect of atmospheric temperature on stress corrosion cracking susceptibility of low carbon steel which immersed in 0.1M of sulfuric acid solution during 14 days was studied using U-bend test. Two testing were carried out, the first in January and the second in July in the weather conditions of Iraq. The variance of atmospheric temperature between them was equal to 40°C. Corrosion rate was calculated using weight loss method. The increasing in temperature will increase the chemical reaction average, so the corrosion rate and weight loss of the used specimens in the second test was greater than the first. An invisible deep cracks was generated in the second specimen at the eleventh day and it's propagate gradually until fractured into two pieces at the last day as a result of high corrosion reaction besides the propagation of microcracks which accelerates the specimen's failure, but the first specimen was not fractured. The corrosion process was affected the convex regions of the U-shape specimen because the stress which affected it was concentrated in it much more than the others. The temperature increasing decrease stress corrosion life of the second specimen.

Keywords: low carbon steel, corrosion rate, U-bend test, stress corrosion cracking, atmospheric temperature.

Introduction

In the nature, most of the metals are found as an **ore** such a state combined chemically condition. All these metals exist in the form of their oxides, carbonates, etc, except gold, platinum and silver [1]. The corrosion process of metals in aqueous solutions in general is an electrochemical process [2]. "The driving force for metallic corrosion is the Gibbs free energy change (ΔG)" [3], which indicates to the change in free energy of the metal and the combination of surrounded environment which brought about by the corrosion process. Corrosion rate can be defined as "the speed at which any metal in a specific environment deteriorates" [4].

Stress corrosion cracking phenomenon can be defined as the process which generate cracks propagation as a result of combination between a metal or alloy affected by stress which may be external or residual and corrosive environment media which may be atmospheric air or a solution such as acidic or salt etc. The three items which can affect the SCC process are: (susceptible material, corrosive environment, and tensile stress) [5]. The material variables are the main important factors used to determine the SCC susceptibility process. For stress corrosion cracking process, the passive layer should be broken. The stresses concentrated at the crack tip which cause intensive plastic deformation in the plastic zone are high [6]. There are many different proposed mechanisms used to discuss the SCC phenomenon depending on the metallurgy, environment, and stresses which may cause or affect this process, and no consensus has been reached [7].



Many testing methods are available in the form of international and national standards and procedures, comprising a wide range of these methods for estimation of SCC susceptibility and also a variety of specimen configurations [8]. The rectangular strip is the general shape of this test specimen which is U-bend specimen obtained after bending this strip 180° around a predetermined radius and used in this stress corrosion cracking test with constant strain state during the testing time. The elastic and plastic strain types are present in U-bending specimens as usual. It's possible to form the shape of this testing specimen (U-shape) in case of thin sheet or wire with small diameter and only elastic strain was produced [9]. The corrosion rate calculation's relation using weight loss method (Eq. 1) depends on the exposure area (A) of the specimen to the environment solution and the time of immersion (T) and weight loss value (Δ W) for the same metal used in the test [10, 11].

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

Where: K - 8.76x10⁴, Δ W - weight loss in (g), D - metal density in (g /cm³).

A - exposure area to corrosion in (cm^2) , T - time of exposure in (hours).

Many researchers have studying the effect of many parameters like temperature on SCC susceptibility of metals and alloys, some of them are: Echlas, Gaida, [12], study the effect of heat treatment on corrosion resistance of medium carbon steel immersed in Sulfuric spring. They found that the annealing steel have great resistance to the corrosion process, but the hardened metal has lower corrosion resistance and the normalized steel is in between. Calcium carbonate was formed on the metal surface which acts as an isolating layer which decrease corrosion rate with time. G.A. Rassoul, D.R. Rzaige, [13], study the corrosion behavior of carbon steel specimens in different temperatures with many concentrations of sodium chloride in water under the effects of pressure equal to 3 bar. They found that specimens which immersed in water containing (100,400,700,1000PPM) of NACL solution and under temperature were increased from (90-120°C) under pressures of 3 bars, and the corrosion rate was increased with NACL concentrations and Temperature. S.B. Farina, G.S. Duffo, J.R. Galvele, [14], they found this alloy was susceptible to SCC in three kinds of environment solutions (1M NACL, 1M KBr, and 1M KI), and the crack propagation was initially intergranular and then it changing to the transgranular, and this propagation was increased when the temperature increased in all these solutions. Yameng Qi, Hongyun Luo, and others, [15], they study the corrosion behavior of carbon steel exposed to hydrogen sulphide environment at different temperatures, and they found that the corrosion rate was increased at the first time and decreased significantly with increasing of temperatures, and with this increasing more fine and compact corrosion film was formed which indicated better protection to the steel metal.

Experimental Procedure

The commercial low carbon steel which chemical composition was illustrated in Table 1 using **XRF** test is the material used for manufacturing and preparing the rectangular cheats according to the standard dimensions for this test, ASTM G30-97 (2003), and the method using to bend these cheats is **male and female former** as shown in Figure 1.

A grinding process with Silicon Carbide (SiC) emery paper with grit size of **320**, **400**, **800**, **1000** and **1200** successively was using, and polished it to obtain a mirror finish surface, free from scratches. The corrosive environment used for test is 0.1M of Sulfuric Acid. The Natal



solution with concentration 2% was prepared for etching process which prepared by adding 2ml of 98% Nitric Acid to 100ml of 98% Ethanol using burette to drop the Nitric Acid gradually to Ethanol in glass container. A solution of 10% HCL Acid was used for cleaning specimens after test and before determination their weights besides of distilled water by soft brush.

Table (1) Chemical composition of specimen's materials (wt %) using (XRF) test.

Materials*	Fe	Cr	Mn	Cu	Si	C	Р	Mo	Ni
L.C.S.	Bal.	0.14	0.49	0.19	0.48	0.14	0.48	-	-



Figure (1) Rectangular plate and U-Bend specimens machining [9]

Before starting test, the U-shape specimen was tested using Instron 1122 in order to know the relation between the applied load and the distance between the two legs of used specimen. After compression test, another specimen with the same dimensions was prepared and used in U-bend test for studying the effect of temperature on SCC susceptibility. The data of this test was illustrated in Table 2, and the plot of this test was shown in Figure 2.

After preparing specimen, its initial weight (W_1) was determined using sensitive balance with accuracy equal to 0.0001g, then using it under constant applied stress by making the distance between the specimen's legs equal to 2cm using the bolt passed through their holes and plastic insulator by screwing it with suitable nut, and according to compression test, the external applied load equal to 845N and then it immersed in 0.1M of Sulfuric Acid solution using plastic container as shown in Figure 1 immediately for 14 days of testing time.

This test was carried out in January where the atmospheric temperature approximately equal to $5^{\circ}C\pm 3$ during the testing time. Another similar test was carried out in July which atmospheric temperature equal $45^{\circ}C\pm 3$. For each test, the specimens were removed every 24 hours from the environment solution and the final weight (W₂) was determined after cleaning and drying it and weight loss (ΔW) was determined too to use it in corrosion rate calculation. The etching process was carried out and suitable optical photos were taken, and then immersed them again in the acidic solution. Someday, the solution was added to the plastic container in case of its evaporation as a result of exposure to atmospheric temperature. The data of these two tests are illustrates in Table 3.

Table (2) U-Bending L.C.S specimen compression and recycling test using Instron 1122



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		Compressio	Recycling Process						
Deflection	Load	Deflection	Load	Deflection	Load	Deflection	Load		
(mm)	(N)	(mm)	(N)	(mm)	(N)	(mm)	Load (N)		
0	0	5	692	15.6	753	23	845		
0.3	45	5.3	700	15.8	755	22.8	770		
0.8	170	5.8	709	16.3	760	22.3	600		
1.3	300	6.3	713	16.8	765	22	500		
1.8	440	6.8	715	17.3	770	21.8	435		
2.3	580	7.3	720	17.8	780	21.3	285		
2.5	630	7.8	720	18.1	786	20.8	150		
2.7	667	8.3	725	18.3	795	20.4	52		
2.8	665	8.8	728	18.5	805	20.2	0		
2.9	655	9.3	730	18.8	817		-		
3.03	655	9.8	732	19	830	Contract Contractorer	A state of the second state of		
11	671	10.3	733	10.3	845				
3.3	679	10.5	733	19.5	945		Lineture 1172		
3.4	690	11.0	735	20.2	945		Instron 1122		
3.5	674	11.5	733	20.5	945		-		
3.33	660	11.0	737	20.0	040		Entrance		
3.0	657	12.3	740	20.95	040				
3.05	05/	12.8	740	21.5	845				
3./	052	13.3	742	22	845	ALC: THE			
3.8	000	13.8	745	22.5	845		in game		
4	675	14.3	747	23	845				
4.3	686	14.8	749			9/1/00			
4.8	698	15.3	750						
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Figure (2) Instron 1122 test plot for U-shape low carbon steel specimen **Results and Conclusion :**

From Table 3, a relation between immersion time and weight loss for these tests was shown in Figure 3 and between immersion time and corrosion rate was shown in Figure 4, and the optical photos of the convex region every 24 hours for these two tests were shown in Figures 5, 6, 7, and 8. In the bending test, a flat strap of metal is bent into a U-shape. In the outer surface of this rectangular plate, the material will stretch while compressing the material on the inside surface. So the surface on one side of the beam will be in compression, while the other side is under tension. In the outer side surface, and as a result of tension stress effect, there is a plastic deformation occurs on this surface and the microcracks will initiate. So with immersion this specimen in the acidic solution, these cracks will be growth with the greater velocity in the second test more than the first and this will decrease the stress corrosion life and will accelerate the failure and make this specimen fractured.

Figure 3 shows that the weight loss in January increased from the first day of test time to the 10th day more than the interval 10-14 day, its change approaches to constant value at this interval, but in June, this relation seem to be a linear behavior except the last day, and the weight loss as a general view is greater in June than the test in January because of the high



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temperature beside of the effect of corrosive environment and applied stress will increase the corrosion reactions and so the corroded areas will increased too. Also the corrosion rate in the second test is greater than the first because of the weight loss increasing as shown in Figure 4. The corrosion rate in these two tests was decreased with the increasing of immersion time. When we compare the corroded area and the topography of this area in these tests, an evidence of the temperature effect was obtained. The increasing of temperature caused increasing in chemical reaction average and increasing in diffusion average.

In the Figure 5, a small pitting appears from the second day, and the corrosion process was increased in small average and from Figure 6, there are some cracks appear and it growths gradually from the fourth day, but when we comparing this case with the second test in July, and from Figure 7, it's clear that the corrosion process velocity was greater than the first and the specimen was began to fracture at eleventh day and it propagates gradually until separation into two pieces as a result of high corrosion reaction in the last day.

This test indicates that the low carbon steel material is susceptible to corrosion process under the effect of applied stress in the sulfuric acid solution with concentration equal to 0.1M and this solution is a good corrosive media for this metal because of the high percentage of Iron (Fe) in the specimen metal which approximately equal to 98% and low percentage of (Cr) which is less than 1.

immorgion	L. C. S. in	n January	7	L. C. S. in July			
time	W1=70.93	364 g		W ₁ =73.3819 g			
(day)	W_2	$\Delta \mathbf{W}$	C.R.	W_2	$\Delta \mathbf{W}$	C.R.	
(uay)	(g)	(g)	mm/y	(g)	(g)	mm/y	
1	69.8262	1.1102	13.4251	71.7664	1.6155	19.5354	
2	68.8170	2.1194	12.8144	70.5856	2.7963	16.9071	
3	67.7991	3.1373	12.6459	69.4016	3.9803	16.0439	
4	67.0122	3.9242	11.8633	68.3137	5.0682	15.3218	
5	66.2184	4.7180	11.4105	67.4392	5.9427	14.3724	
6	65.7802	5.1562	10.3919	66.4101	6.9718	14.0511	
7	65.2303	5.7061	9.8573	65.5105	7.8714	13.5978	
8	64.6901	6.2463	9.4417	64.4008	8.9811	13.5755	
9	64.2814	6.6550	8.9417	63.5191	9.8628	13.2518	
10	64.0427	6.8937	8.3362	62.4954	10.8865	13.1645	
11	63.8194	7.1170	7.8238	61.5291	11.8528	13.0300	
12	63.7571	7.1793	7.2346	60.6016	12.7803	12.8788	
13	63.6821	7.2543	6.7479	59.9901	13.3918	12.4569	
14	63.5101	7.4263	6.4145	59.8554	13.7265	11.9699	

Table (3) Weights loss and corrosion rate data for U-Shape bending test in January and July during 14 days



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Figure (3) Weight loss vs. immersion time in January and July months Figure (4) Corrosion rate vs. immersion time in January and July months



Figure (5) Corroded regions of U-shape specimens each day in January



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Figure (6) Corrosion effect on convex area of the specimen at the last day in January



Figure (7) Corrosion effects on the convex area during 14 days in July



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Figure (8) Corroded surface and fracture line of specimen at the last day

المستخلص تطرق البحث الى دراسة تأثير درجة حرارة بيئة الغلاف الجوي على قابلية التشقق الناتج من عملية التآكل تحت تأثير الاجهاد للفولاذ منخفض الكربون والمغمور في تركيز 0.1 مولاري من محلول حامض الكبريتيك خلال 14 يوماً باستخدام اختبار الانحناء U-bending test. وبعد اجراء فحص المرونة للعينة والتي تم تحضير ها على شكل حرف (U) باستخدام جهاز الانسترون 1122 تم إجراء اختبارين ، الأول في شهر كانون الثاني والثاني في شهر تموز في الظروف الجوية للعراق. وكان التباين في درجة حرارة الغلاف الجوي بينهما بحدود 40 درجة سيليزية تقريباً ، وتم حساب معدل التآكل باستخدام طريقة فقدان الوزن الناتجة من عملية التأكل . إن الزيادة في درجة الحرارة للبيئة المحيطة بالعينات ستؤدي إلى زيادة معدل التفاعل الكيميائي ، وبالتالي فإن معدل التأكل وفقدان الوزن للعينات المستخدمة في الاختبار الثاني كان أكبر من الأول .

في اليوم الحادي عشر شو هدت شقوق عميقة غير مرئية في العينة الثانية وانتشرت تدريجياً حتى انكسرت إلى قطعتين في اليوم الأخير نتيجة تفاعل التآكل العالي بالإضافة إلى انتشار الشقوق الصغيرة مما عجل من فشل العينة ، ولكن العينة الأولى لم تنكسر حيث أثرت عملية التآكل على المناطق المحدبة للعينة لأن الإجهاد الذي أثر عليها كان يتركز فيها أكثر بكثير من غيرها. ولهذا تم التحقق من ان زيادة درجة الحرارة للبيئة المحيطة بالعينة عند اجراء الاختبار يؤدي إلى تقليل عمر التآكل

الكلمات الدالة: الفولاد منخفض الكاربون، معدل التآكل، فحص الانحناء، التآكل الاجهادي، درجة حرارة الغلاف الجوي.



References

- **1.** E.E. Stansbury, R.A. Buchananm, "Fundamentals of Electrochemical Corrosion", ASM international, p 40, 2000.
- **2.** E.E. Stansbury, R.A. Buchanan, ASM International, "Fundamental of Electrochemical Corrosion", USA, 2000.
- **3.** Linn W. Hobbs, "Electrochemical Corrosion, Gibbs Free Energy, Anodic Corrosion & EMF Series", Massachusetts Institute of Technology, p 2, 2006.
- 4. Einar Bardal, "Corrosion and Protection", Springer, p 8, 2004.
- **5.** Kevin C. Stewart, "Intermediate Attack in Crevice Corrosion by Cathodic Focusing", PH.D thesis, University of Virginia, 1999.
- 6. Prashant Kumar, "Elements of Fracture Mechanics", New Delhi, p 203, 2009.
- 7. Russell H. Jones, "Stress Corrosion Cracking Materials Performance and Evaluation", ASM International, 1992.
- **8.** Victoria Burt, "Corrosion in Petrochemical Industry", 2nd Edition, ASM International, p 128, 2015.
- 9. V. S. Raja and Tetsuo Shoji, "Stress Corrosion Cracking-Theory and Practice", WP Publishing Ltd, UK, p135, 2011.
- **10.** ASTM G30-97, "Standard practice for making and using u-bend stress-corrosion test specimens", West Conshohocken: ASTM International, 2003.
- 11. ASTM, G1, "Standard Practice for Preparing, Cleaning, and Evaluating corrosion Test Specimens".
- 12. Ekhlas A., Gaida I. "Influence of Heat Treatments on the Corrosion Resistance of Medium Carbon Steel using Sulfuric Spring Water", Tekreet magazine, vol. 19, No. 3, Iraq, p 14, 2012.
- **13.** G.A. Rassoul, D.R. Rzaige, "Effect of temperature on corrosion of carbon steel boiler tubes in dilute sodium chloride solution", Iraqi Journal of Chemical and Petroleum Engineering, Vol.9 No.3, p 37, 2007.
- 14. S.B. Farina, G.S. Duffo, J.R. Galvele, "Stress Corrosion Cracking of Zircaloy-4 in Halide Solutions, effect of temperature", Material research, Vol. 5 No.2, Argentina, 2002.
- **15.** Yameng Qi, Hongyun Luo, and others, "Effect of Temperature on the Corrosion Behavior of Carbon Steel in Hydrogen Sulphide Environments", Int. J. Electrochem. Sci., p 2101, 2014.