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## RESEARCH ARTICLE

### Corrosion Fatigue Inhibition of SS Type 304 in Acidic Media in Presence of Some Pyrimidine Derivatives

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#### Abstract

The corrosion fatigue (CF) susceptibility of SS type 304 in 0.1 M HCl solution and the inhibiting effect of pyrimidine derivatives using a corrosion cell on a cantilever rotary-bending fatigue test machine were investigated. Scanning electron microscopy (SEM) was conducted on surface fracture of some specimens. The results indicated that the inhibitive action of pyrimidine derivatives can be attributed to its adsorption and inhibiting the anodic dissolution of the metal by the effect of blocking the active sites on the surface then improve the fatigue life of the specimens. Polarization measurements revealed that these pyrimidine derivatives behave as mixed-type inhibitors.

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#### Introduction

It is well known that corrosion fatigue involves the interaction of the corrosive environment and the applied cyclic stresses. The life of structures and components subjected to fatigue conditions is the summation of the period spent at each stage of the fatigue process from the formation and growth of macroscopically detectable cracks up to the failure stage [1]. The corrosion fatigue behaviour of 316L alloy in chloride solutions is analysed and compared to the stress corrosion cracking behaviour of the same alloy at the same electrochemical conditions. The kind of corrosion fatigue test (cyclic tensile stress, tension – compression strain) is shown to have a very sensitive effect on the resulting damage [2]. They emphasize the relation and the differences between SCC and true CF mechanism, which can occur in a 316L austenitic stainless steel subjected to a cyclic load in chloride solutions. Corrosion fatigue tests in 3.5% NaCl aqueous solution have been conducted on low carbon steel, S10, a stainless steel SS 304, and an aluminium alloy, A3034-T4, in order to clarify their crack initiation behaviour from corrosion pits [3]. It was found that the size of corrosion pits increased with decreasing stress level.

Corrosion fatigue under the load of low frequency and high mean stress has been generally defined as stress corrosion fatigue (SCF) [4]. It is a specific failure process due to the interaction between stress corrosion cracking (SCC) and corrosion fatigue (CF). The authors studied the environment sensitive fracture behaviour of Cr18Ni9Ti austenitic stainless steel under the load of different frequencies and high mean stress in boiling MgCl<sub>2</sub> solution and emphasized on the interaction between SCC and CF and also on the effects of applied potential on the interaction [5]. Corrosion fatigue of 12Cr steel in air at room temperature and 33% NaCl solution at 80 °C, and the influence of corrosion pits on crack nucleation has been investigated [6]. The authors showed that the stress intensity ( $\Delta K$ ) for crack nucleation from a pit in rotating bending fatigue tests is very low in air.

Corrosion fatigue crack initiation was examined on 12 Cr Mo martensitic stainless steel in dilute NaCl solution at 80°C under either galvanostatic or potentiostatic control. The effects of mean stress, NaCl concentration and corrosion current on crack initiation were investigated [7]. They showed that the results confirmed that each corrosion fatigue crack had initiated at a corrosion pit. The initiation and growth of fatigue micro-cracks in a BS 4386-500 structural steel has been studied in laboratory in air and aerated 3.5% NaCl solution. Experiments were performed using constant load fully reversed bending at a frequency of 25 HZ [8]. The fatigue strength of the material reduced in the aqueous environment to 130 Mpa compared with 290 Mpa in air. The corrosion fatigue behaviour of alloy 600 was studied as a function of applied potential and heat treatment in highly salinity brine (4M NaCl) in the absence and presence of low concentrations of sodium tetrathionate and sodium thiosulphate [9]. The addition of tetrathionate to the chloride solution did not change the fatigue life of the solution annealed material with respect to that in pure chloride solution, while the addition of  $S_2O_3^{2-}$  improved the corrosion fatigue resistance.

Fatigue tests have been performed on a dual- phase stainless steel, 339 J4L, and an austenitic stainless steel, 304, in order to clarify the crack initiation and early crack growth behaviour in 3% NaCl [10]. Crack initiation in room air occurred due to ductile trans-granular slip in both stainless steels. In contrast, cracks were generated at the austenite- ferrite phase boundary of SS 339 J4L and at the corrosion pit of SS 304 in 3% NaCl. McEvily [11] examined the dislocation substructures generated at the plastic zone of a fatigue crack in SS type 304. In vacuum, the dislocation arrays were found to be denser and more homogeneous than in air. It was concluded that absorbed – gas-induced embrittlement was not involved in the fatigue crack growth mechanisms of 304 SS fatigued in air. Low – cycle fatigue tests were performed on 304 LN austenitic steel by Raman et al [12] to observe effect of prior cold work. Various prior cold works (10, 20 and 30%) increased the low-cycle fatigue resistance by only 0.50%.

Fukuyama et al. [13] reported that solution-treated SS type 304 showed the same fatigue crack growth behaviour below 10MPa in both high pressure hydrogen and argon atmospheres. The threshold fatigue crack growth (FCG) behaviour of AISI 304 austenitic stainless steel was investigated by Kelestemur et al [14] under three different atmospheres: dry argon, hydrogen and moist air. The results of test done under the three different atmospheres showed that the material is sensitive to the hydrogen atmosphere. The nature of the fracture was only trans-granular under both hydrogen and moist air. Examination of various failures in SS type 304 unit's uncovered evidence of fatigue, corrosion fatigue, and stress corrosion cracking. The cyclic load that was causing these failures had a frequency of 0.0625 HZ. It was therefore considered possible that the propagation of the cracks was accelerated by the wet environment in which the units were operating [15].

Masaki et al [16] investigated the fatigue strength in the long life regime of two types of austenitic SS types 316 and 304 specimens with stress concentration regions, rotating bending fatigue tests were performed with pit specimens that had a few artificial corrosion pits at their centers. Inhibitor addition is one of the most accessible and effective methods of lowering the CFC growth rate. It has been established that a corrosion inhibitor is able to decrease sufficiently the CFC growth rate, in some cases even below that in air [17].

In this paper, the effect of pyrimidine derivatives on the corrosion fatigue (CF), with cyclic frequency 10.5 Hz of 304 SS in 0.1 M HCl solution was presented and the % inhibition efficiency of these pyrimidine derivatives was calculate

## 2. Experimental Methods

Chemical compositions of AISI 304 austenitic stainless steel specimens were shown in Table (1). Cylindrical tensile specimens were used in the fatigue test in air and in the corrosive environment. The cylinder specimen with diameter 10 mm, the gage length of the middle part 20 mm with diameter 4 mm was used as test section and the overall length 140 mm as shown in Fig.(1). The gage length of all specimens were prepared by treatment with emery papers up to 1000, then degreased in acetone in an ultrasonic bath, washed with bi-distilled water and then dried. Fatigue tests were performed at 10.5 Hz on a cantilever rotary-bending fatigue test machine as shown in Fig. (2), with different cyclic stresses (181 MPa – 288 MPa). The specimens were coated with epoxy resin except for the gage section which was immersed in the corrosion cell as shown in Fig. (3) during the fatigue. The corrosive environment was used, 0.1 M HCl which was prepared with analytical grade reagents and distilled water and was used as the blank solution. The inhibited solutions containing ( $3 \times 10^{-6}$  –  $1.2 \times 10^{-5}$  M) pyrimidine derivatives were used as inhibitors as shown in Table (2).

Galvanostatic polarization studies were carried out on SS type AISI 304 in 0.1 M HCl solution in the presence and absence of the inhibitors at various concentrations and at 30 °C in three neck cell. The working electrode has dimensions 1 cm x 1 cm and was weld from one side to a copper wire used for electric connection. The sample was embedded in a glass tube using epoxy resin [20]. Saturated calomel electrode (SCE) and platinum coil were used as reference and counter electrodes, respectively. All experiments were carried out with 100 ml and at 30°C. The structure, name and molecular weights of investigated inhibitors [21] are presented below:

### 3. Results and Discussion

#### 3.1. Polarization Curves

The corrosion behavior of SS type 304 is studied in 0.1 M HCl solutions in the absence and presence of ( $3 \times 10^{-6}$ – $1.2 \times 10^{-5}$  M) inhibitors (I – IV) at 30°C. Figs. (4&5) show the galvanostatic polarization curves of austenitic stainless steel type 304 in 0.1M HCl in the absence and presence of inhibitors I&II at various concentrations (similar curves were obtained for other compounds and are not shown). The order of inhibition efficiency (% IE) is as follows: I > II > III > IV.

#### 3.2. The Corrosion Fatigue Behavior of Stainless Steel Type 304 in HCl.

The results representing the corrosion fatigue behavior of SS type 304 in HCl solution in the absence and presence of inhibitors I & II with different concentrations. The experiments were carried out as follow:

1- Stress amplitude (S) versus number of cycles to failure (N) (fatigue life). 2- Stress amplitude versus percentage protection efficiency (% IE). 3- Inhibitor concentrations versus percentage protection efficiency (% IE). 4- Inhibitor concentrations versus cycles to failure (fatigue life). 5- SEM examinations of the failed specimens.

##### 3.2.1- Effect of Changing Applied Cyclic Stresses and Inhibitor Concentrations on the S/N Curve and Inhibition Efficiency % IE

To characterize the response of the SS type 304 to cyclic bending stresses in 0.1MHCl solution in absence and presence of inhibitors I&II at variance concentrations, the stress amplitude ,S, versus number of cycles to failure ,N, were determined. All tests were conducted at stress values in the range 141 MPa to 500 MPa. The results are shown in the Table (2) and in Fig. (6). It is clear that the fatigue life of stainless steel type 304 in 0.1 M HCl is considerably shorter than that obtained in air. It is clear also that there are fatigue limits in air amount 360 MPa and 141 MPa for 0.1 M HCl solution. The fatigue strength of stainless steel 304 specimens of the two environments air and 0.1 M HCl can be compared at such a number of cycles  $10^7$  as follows:

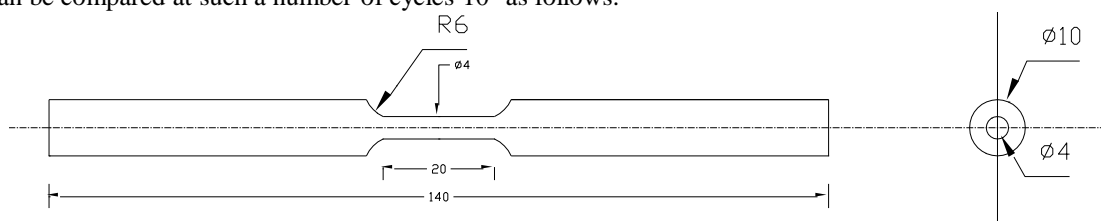


Fig. 1. Specimen of Fatigue Test.

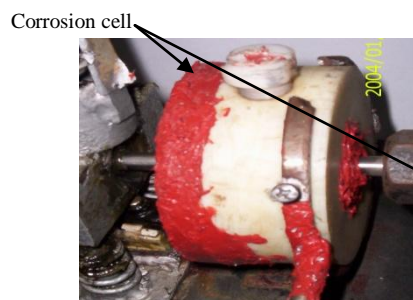


Fig. 3. Photo of Corrosion Cell



Fig. 2. Photo of Machine of Corrosion Fatigue Test

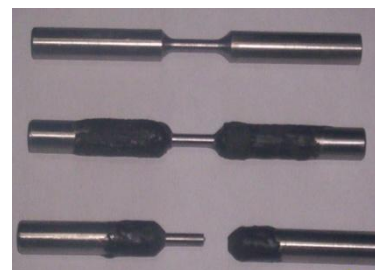
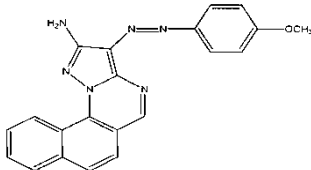
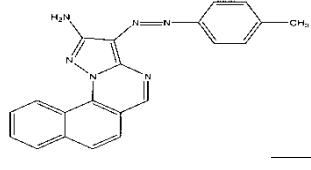
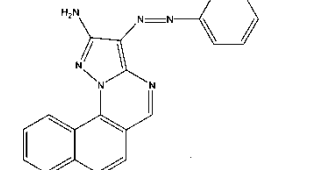
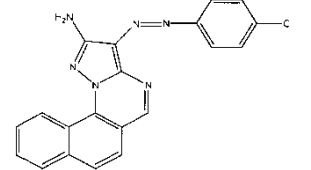


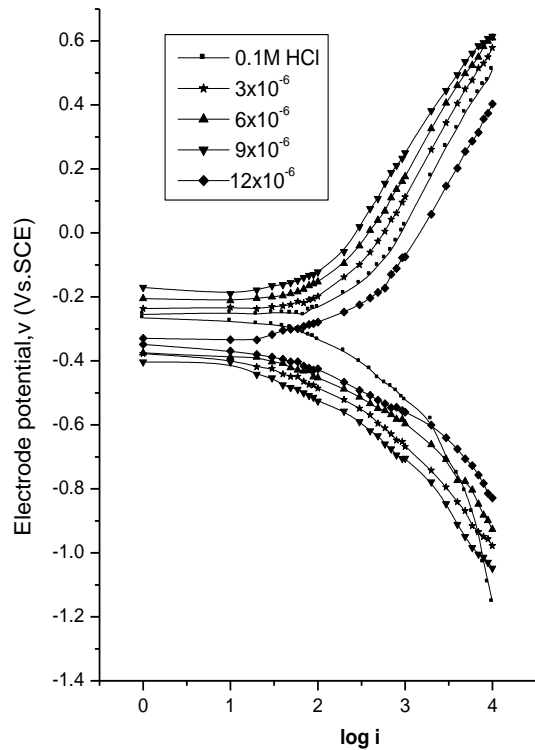
Fig. 1. Photo of Specimen of Corrosion Fatigue Test

**Table (1).** Chemical Compositions of AISI 304 Stainless Steel (Weight %)

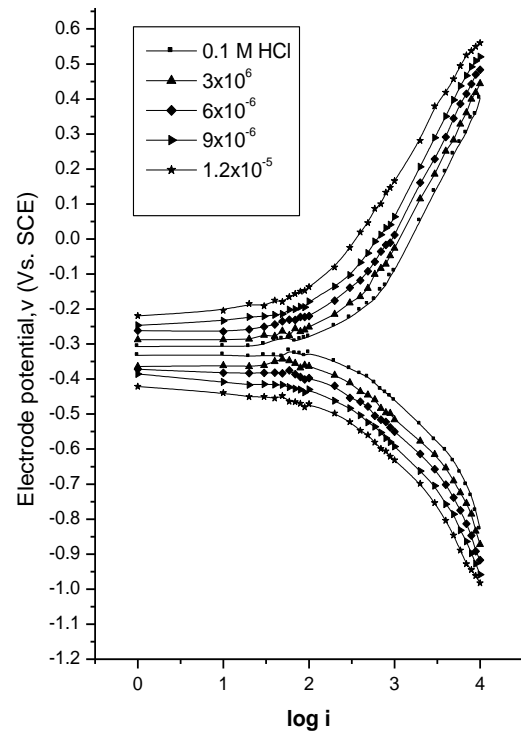
C	Mn	P	S	Si	Ni	Cr	Fe
<b>0.07</b>	<b>2.00</b>	<b>0.045</b>	<b>0.030</b>	<b>0.75</b>	<b>9.0</b>	<b>18.0</b>	<b>Reminder</b>

Inh.	Name	Structure
<b>I</b>	3-(4-Methoxy-phenylazo)-1,4,11C-triaza-Cyclopenta [c] Phenanthren-2-ylamine	
<b>II</b>	3-(4-Methyl-phenylazo)-1,4,11C-triaza-Cyclopenta [c] Phenanthren-2-ylamine	
<b>III</b>	3-(4-Hydrogen-phenylazo)-1,4,11C-triaza-Cyclopenta [c] Phenanthren-2-ylamine	
<b>IV</b>	3-(4-Chlore-phenylazo)-1,4,11C-triaza-Cyclopenta [c] Phenanthren-2-ylamine	

$$\frac{\text{Fatigue Strength in Air}}{\text{Fatigue Strength in 0.1M HCl}} = \frac{360}{141} = 2.55 \quad (1)$$



**Fig.4** Polarization Curves of SS Type 304 in 0.1 M HCl in the Absence and Presence of Various Concentrations of Compound I at 30 °C



**Fig.5** Polarization Curves of SS Type 304 in 0.1 M HCl in the Absence and Presence of Various Concentrations of Compound II at 30 °C

Because of the wide separation of the two curves the fatigue life cannot be determined or compared at any definite stress value. However, the approximate % reduction in the fatigue life can be calculated according to the equation [22].

$$[N_{air} - N_{0.1M HCl} / N_{air}] \times 100 \tag{2}$$

Where:  $N_{air}$  is the number of cycles to failure (fatigue life) in air and  $N_{0.1M HCl}$  is the number of cycles to failure (fatigue life) in 0.1 M HCl of specimens at the same stress.

**Thus at a stress value of 400 MPa:**

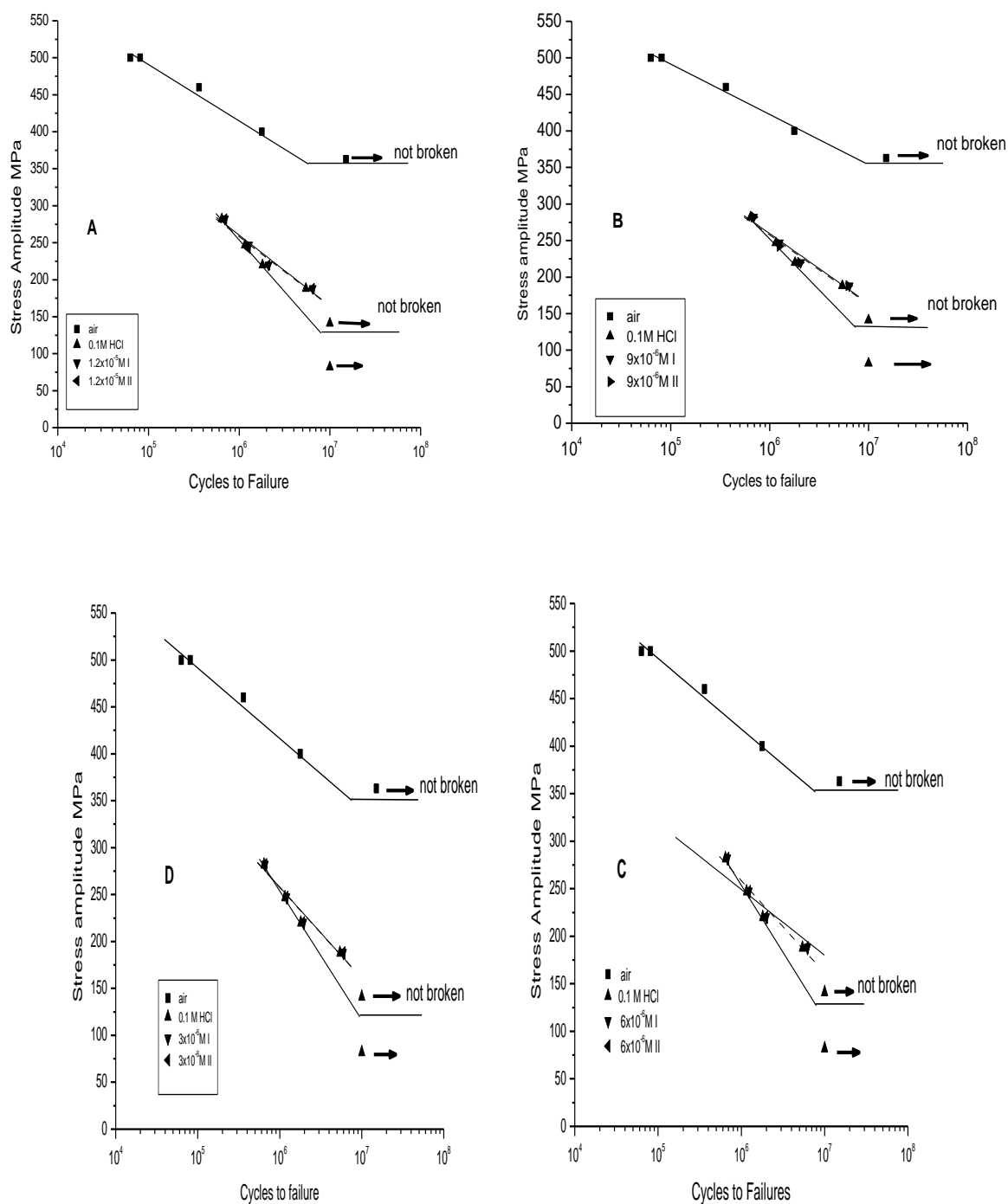
**The relative reduction of fatigue life of the specimen at 0.1 M HCl =  $[1780000 - 7 \times 10^4 / 1780000] \times 100 = 96.1\%$**

However, no single mechanism seems to be valid for all combinations of material, environment and stress state. Corrosion fatigue seems to be most prevalent in mediums that produce pitting attack. These two facts indicate that fatigue resistance is reduced in the presence of a corrosive because corrosion pits act as stress raisers and initiate cracks [20] Pitting corrosion occurs on the surface of stainless steel 304 in the aggressive environment HCl which represents initiation crack and the interaction of the applied cyclic stress, the propagation crack occurs rapidly. This explains the relative reduction of fatigue life of the specimen up to 96.1 %.

The presence of the inhibitors with the aggressive solution 0.1M HCl improve of the relative reduction of fatigue life of the specimen as shown in the results of the Tables (2 & 6) which, the protective efficiency % IE range from 3 % up to 17 % with variation cyclic stresses and inhibitors concentrations. This resulting to the inhibitors reduces the corrosion attack, which improve the passive film formed on a SS-304 surfaces and it prevents pit growth. A reasonable explanation is that the active sites on the metal surface are blocked by the adsorbed inhibitive species, which depends on the inhibitor concentrations.

**Table (2)** the corrosion fatigue behavior at inhibitors concentration  $1.2 \times 10^{-5}$  M.

Specimin Material	Environment Temperature	Stress Ratio	Corrosive Media	Stress Frequency Hz	Cyclic Stress MPa	N <sub>2</sub> of Cycles To Failure	IE %
Austenitic Stainless Steel 304	30°C	-1	Air	10.5	500	63046	-----
					500	81030	-----
					460	360800	-----
					400	1780000	-----
					363	15065000	-----
Austenitic Stainless Steel 304	30°C	-1	0.1M HCl	10.5	282	644490	-----
					247	1155420	-----
					220	1807470	-----
					188	5424303	-----
					141	> 1E7	-----
					82	> 1E7	-----
Austenitic Stainless Steel 304	30°C	-1	0.1M HCl +Inhibitor I	10.5	282	697467.078	7.6
					247	1282400.658	9.9
					220	2109136.743	14.3
					188	6466311.606	16.1
Austenitic Stainless Steel 304	30°C	-1	0.1M HCl +Inhibitor II	10.5	282	693922.383	7.1
					247	1260794.304	8.4
					220	2074433.319	12.9
					188	6405559.413	15.3



**Fig. (6):** Stress Amplitude vs. Number of Cycles to Failure of SS Type 304 in 0.1 M HCl in Absence and Presence of the Two Inhibitors with Concentration (A)  $1.2 \times 10^{-5}$  M , (B)  $9 \times 10^{-6}$  M , (C)  $6 \times 10^{-6}$  M and (D)  $3 \times 10^{-6}$  M.

**3.2. 2- Effect of Cyclic Stress Amplitude on the Inhibition Efficiency:**

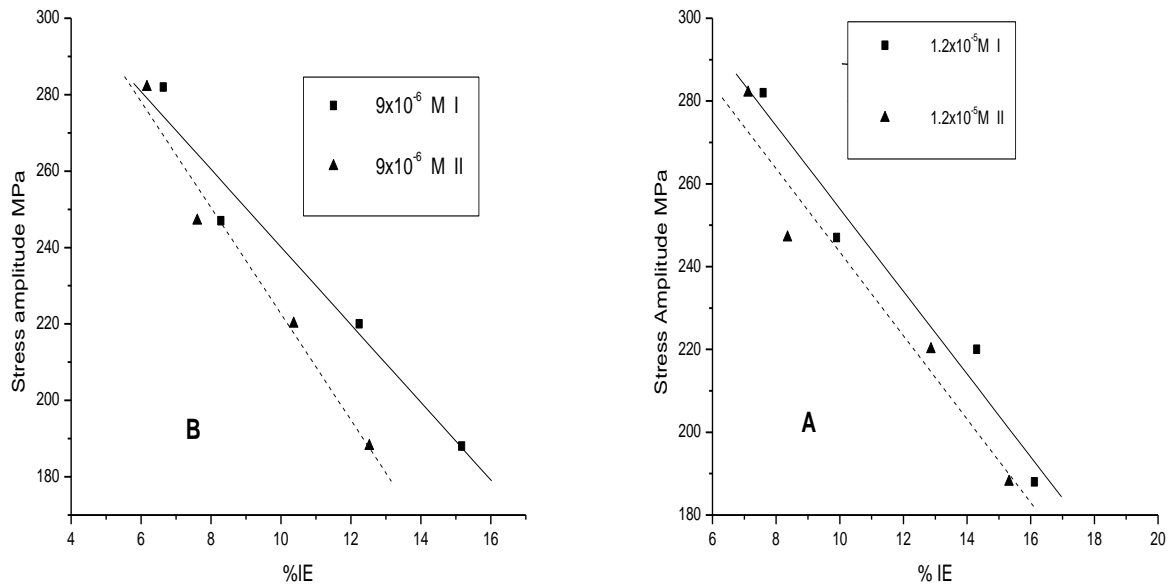
The data obtained from the corrosion fatigue of SS type 304 in 0.1 M HCl in the presence of the two inhibitors with different concentrations ( $3 \times 10^{-6}$  -  $12 \times 10^{-6}$  M) and different cyclic stresses (188 MPa – 282 MPa) at 30°C. The inhibition efficiency of the inhibitor depends on the cyclic stress value and the inhibitor concentration. As

shown in Fig. (7), the inhibition efficiency % IE decreases with the increasing of applied cyclic stress with variation of inhibitor concentrations from ( $3 \times 10^{-6}$  M- $12 \times 10^{-6}$  M), due to the interaction of applied cyclic stress with the film formed on the surface of a SS type 304 specimen is the formation of a slip step which causes rupture of the protective layer film.

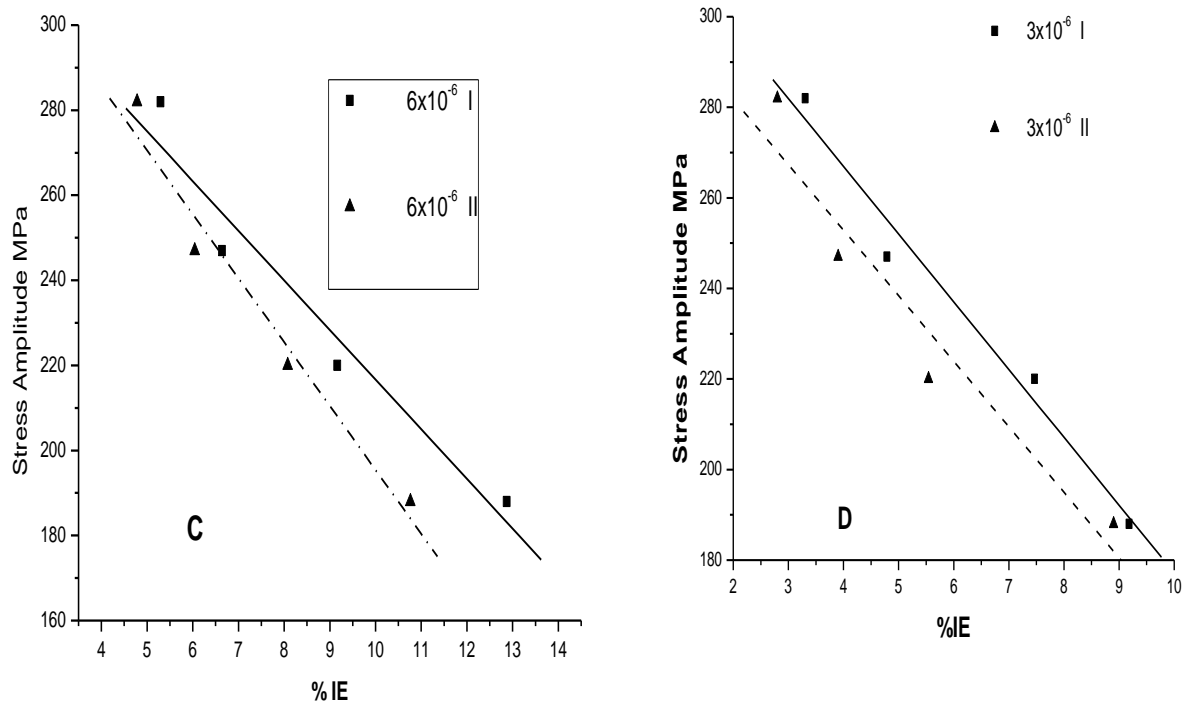
The protective layer film (inhibitor molecules) mainly adsorb on the active sites of surface of SS-304 specimen and inhibition efficiency varieties with the inhibitor concentration, which the percentage protective efficiency %IE at  $1.2 \times 10^{-5}$  M in range (7.1-16.1), at  $9 \times 10^{-6}$  M in range (6.1-15.1), at  $6 \times 10^{-6}$  M in range (4.1-12.9) and at  $3 \times 10^{-6}$  M in range (2.7-9.2) at different cyclic stresses (188 MPa-282 MPa). We observe that inhibition efficiency decreases with decreasing the concentration of inhibitor i.e. the protective layer film (inhibitor molecules) also decreases.

**3.2.3- Effect of Inhibitors Concentrations on the Fatigue Life:**

The data obtained from the corrosion fatigue of stainless steel SS type 304 in 0.1 M HCl in presence of the two inhibitors I&II with different concentrations ( $3 \times 10^{-6}$  –  $1.2 \times 10^{-5}$  M) and different cyclic stresses (188 MPa – 282 MPa) at 30 °C.





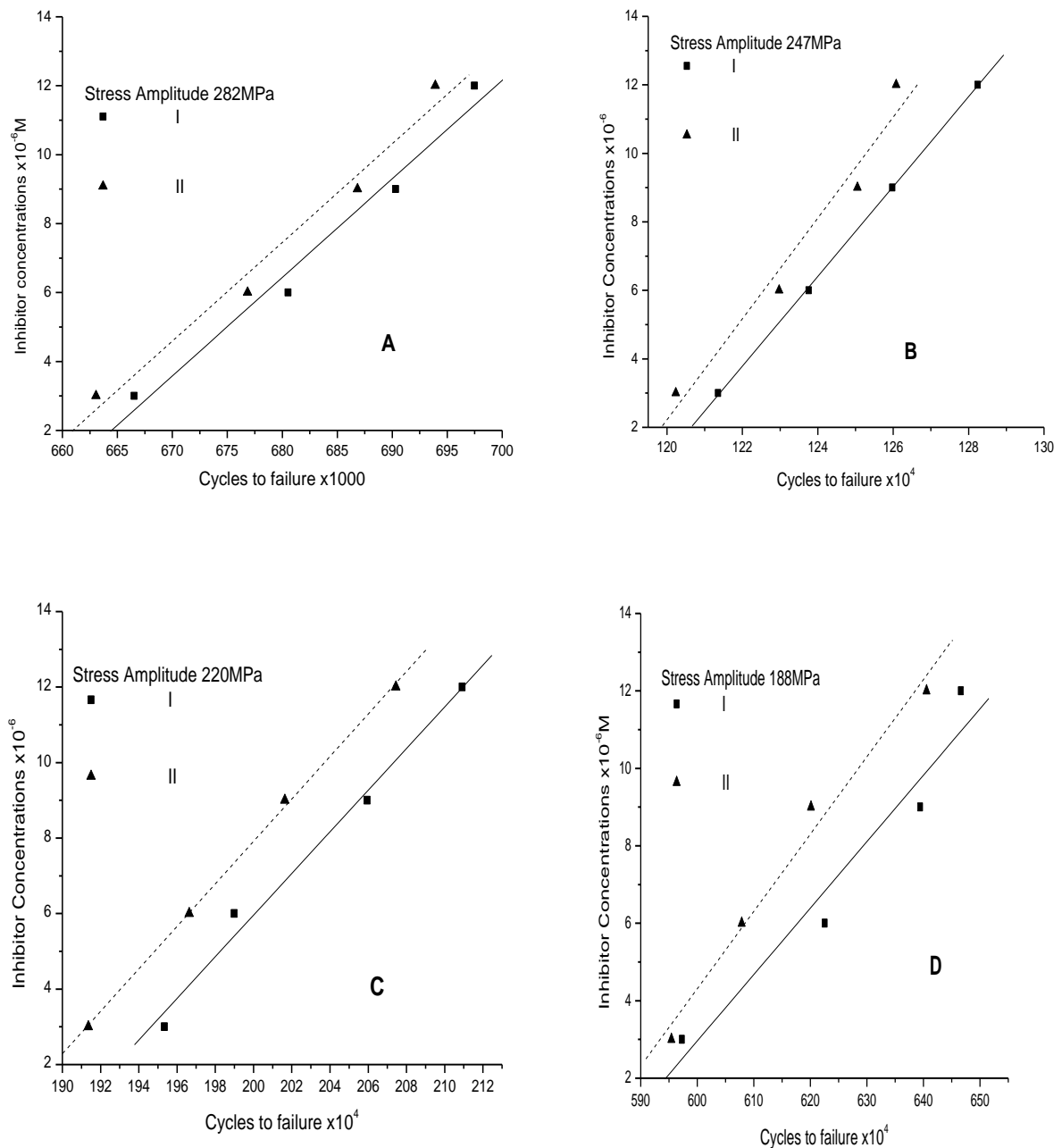


**Fig. (7):** Effect of Cyclic Stress Amplitude on the Inhibition Efficiency % IE in Presence of the Inhibitors I & II with Concentration (A)  $1.2 \times 10^{-5}$  M, (B)  $9 \times 10^{-6}$  M, (C)  $6 \times 10^{-6}$  M, (D)  $3 \times 10^{-6}$  M.

The number of cycles to failure (fatigue life) increases with the increasing of the inhibitors concentrations at different cyclic stresses as shown in Fig. (8), due to reduction of corrosion fatigue crack growth (CFCG) rate. The number of cycles to failure range at cyclic stress 282MPa at different concentrations of the two inhibitors is ( $6.66 \times 10^5$ -  $6.97 \times 10^5$ ), the number of cycles to failure range at cyclic stress 247MPa at different concentrations of the two inhibitors is ( $1.2 \times 10^6$ -  $1.28 \times 10^6$ ), the number of cycles to failure range at cyclic stress 220 MPa at different concentrations of the two inhibitors is ( $1.91 \times 10^6$ -  $2.11 \times 10^6$ ) and the number of cycles to failure range at cyclic stress 188 MPa at different concentrations of the two inhibitors is ( $5.95 \times 10^6$ -  $6.47 \times 10^6$ ). It is clear that the number of cycles to failure range increases with decreasing the cyclic stress, which the dislocations increase with increasing the cyclic stress, in the other side the number of cycles to failure decrease. In other words when the cyclic stress decrease the dislocation also decrease and the number of cycles to failure increase.

### 3.2.4- SEM Fractographic Examinations:

Scanning electron microscope (SEM) (JEOL, JSM-T100) examinations were carried out on the fractured surface specimens in order to identify the mode and severity of failure. Figure (18) represents the fatigue fracture surface grown in air. SEM micrographs showed that the mode of fracture morphology is a predominantly transgranular fracture [11-14]. Fatigue striations and corrosion products are also clear as shown in micrograph of Fig. (18). The corrosion fatigue Fractographic representing the specimens fatigued in 0.1 M HCl solution and at cyclic stress 247MPa is shown in Fig. (19).

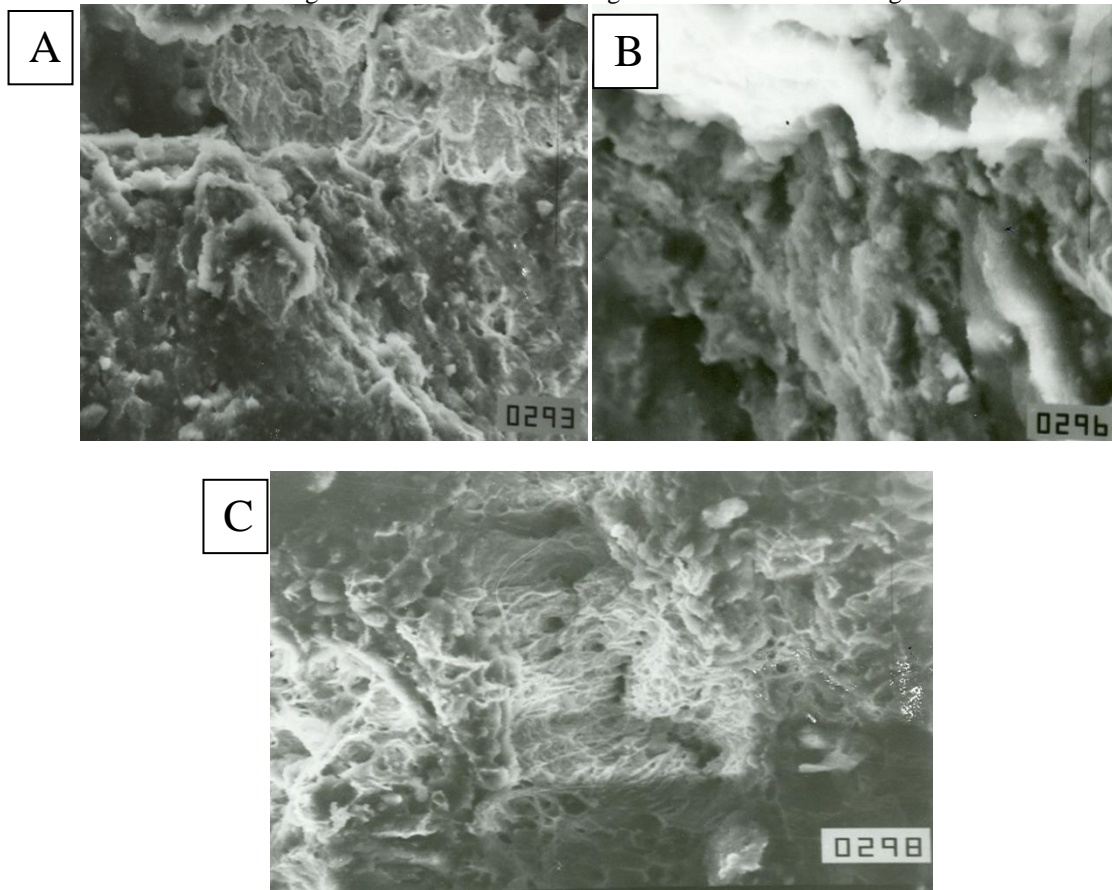


**Fig. (8):** Effect of Inhibitors Concentrations on the Fatigue Life at Cyclic Stress (A) 282 MPa, (B) 247 MPa, (C) 220 MPa, (D) 188 MPa.

Transgranular cracking is characteristic of the failure and shows a secondary crack branching from the major crack. The secondary crack initiates transgranular and then propagates intergranular. Under application of an adding inhibitors, on the other hand, the mode of failure is also mixed intergranular and transgranular but less severe than that under the 0.1 M HCl as shown in the Fig. (20) which represents the corrosion fatigue fracture surface grown in 0.1 M HCl in presence of inhibitor I (methoxy) at cyclic stress 282 MPa and inhibitor concentration  $1.2 \times 10^{-5}$  M.

Mixed intergranular and transgranular cracking are characteristic of the failure. Fatigue striations are also clear in

this micrograph. The transformation of the mode of cracking from transgranular in air to mixed trans- and intergranular in 0.1 M HCl solution in the absence and presence of inhibitors, that enhanced deformation associated with grain boundaries causes enhanced preferential dissolution of the grain boundaries side by side with that at slip bands. The shifting from transgranular in air to intergranular under corrosion fatigue conditions may result from localized dissolutions due to high dislocation densities and geometrical constraints at grain boundaries.



**Fig. (9):** SEM Fractographic of Specimen 304 Type SS in (A) Air at 500 MPa, (B) 0.1 M HCl at 247 MPa, (C) 0.1 M HCl in Presence of Inhibitor (I) at 282 MPa at Concentration  $1.2 \times 10^{-5}$  M.

#### 4. Conclusions

In the present study, rotary bending fatigue tests were performed in laboratory air and in 0.1 M HCl in absence and presence of two inhibitors aqueous solution using material as-received 304SS austenitic stainless steel. The conclusions can be made follows:

- 1- The fatigue strength in 0.1 M HCl solution more lower than the fatigue strength in laboratory air which the fatigue strength ratio in air to in 0.1 M HCl equal 2.55, indicating reduction corrosion resistance of the 0.1 M HCl solution case.
- 2- Using the two inhibitors methoxy-phenylazo and methyl-phenylazo improve the corrosion resistance in 0.1 M HCl solution, which the protection efficiency %IE at different inhibitor concentrations and different cyclic stresses ranges (3.3-16.1 %).
- 3- The inhibition efficiency % IE at different inhibitor concentrations and different cyclic stresses decreases with increasing the cyclic stress amplitude.
- 4- The inhibition efficiency % IE at different inhibitor concentrations and different cyclic stresses increases with increasing the inhibitors concentrations.
- 5- The number of cycles to failure (fatigue life) increase with increasing the inhibitor concentration.
- 6- The mode of fracture is transgranular in air and becomes mixed trans-and Intergranular in 0.1 M HCl in absence and presence of inhibitors under free corrosion condition.

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