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Research Paper

Evaluation of Fatigue Limit and Mechanism of Fatigue Crack Initiation of Epoxy Coatings used as Lining for Crude Oil Storage Tanks

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ABSTRACT

In this paper, the fatigue performance of three types of epoxy coatings (pure, Novolac and reinforced by glass-flake) were studied in crude oils and air environments. A group of equipment is constructed in a laboratory and used in rotating bending fatigue tests of single and double layers of coats applied to ASTM A537-C1 steel. The results showed that the fatigue limit of A537 steel improved after coatings and that the fatigue limits of the coated and uncoated steel conducted in the crude oil solution was significantly lower than those obtained in the air environment. The results of the microscopic inspection indicate that there are three types of fatigue cracks initiation mechanism in steel coatings and environment affects the shape of fracture surface. In addition, the observation of the broken section of the single-layer test sample showed that it has more coat failure compared to the double layers coats.

1 Introduction

Fatigue and corrosion, consider the primary causative of structural failures of crude oil storage tanks and shipment tankers [1]. Practically the imperative techniques that used to protect these tanks from internal corrosion by utilizing epoxy coats. Epoxy coatings have been widely used because of its excellent chemical resistance, outstanding processability, high electrical insulating properties and strong adhesion affinity to steel materials. Also the epoxy coatings are easy to apply at a temperature range from 5 to 150^{o}C depending on the type of curing hardener used to form a highly cross linked adduct in three dimensional network. Various types of epoxy resin and hardeners can be found and the commonly used include polyamides, polyamines, phenolic resins and anhydrides [2].

The tanks at the ports in southern Iraq are persistently exposed to crude oils full and empty for exporting. The filling and discharging operations of tanks by crude oils display the internal epoxy coats to stresses have a maximum value when fully

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filled and are less valuable when completely discharged. These oscillate in stresses makeup the interior covering epoxy coats are exposed to fatigue, deformation and promote the emergence of cracks in steel coats [3].

Experiment fatigue results generally are described by drawn S-N curve, a plot of cyclic stress amplitude (S) against the fatigue life (N) of the tested specimens. The S-N curve estimates the service lifetime of metals and epoxy coats for the most part included three types of the fatigue failure: high cycle fatigue (HCF) happens at $N > 10^5$ cycles, low cycle fatigue (LCF) happens at $N < 10^5$ cycle and thermal mechanical fatigue (TMF) [4]. A fatigue limit is described as the restricting estimation value of stress where fatigue failure happens in an S-N curve. For epoxy coating and non-ferrous materials a number of cycles typically 10^7 cycles are selected to estimate the fatigue limit value. Underneath this esteem, the tested material can bear an endless number of cycles before fatigue failure happens [5].

There are relatively few limited studies have been published on the fatigue behavior of steel coated by the epoxy coatings. Vosikovsky et al. [6] used experimental laboratory methods to study and investigations into the effect of crude oil containing hydrogen sulphide on fatigue crack growth rates for various crack orientations and configurations. The investigation result demonstrates that the fatigue cracks growth rate of steel is dependent on hydrogen sulphide contents by a crude oil. Tzou et al. [7] conducted an experimental investigation of the impact of dehumidified silicone and paraffin oils on fatigue crack propagation of lower strength bainitic steel. Their results demonstrate that there is a little, but definite trend of higher crack growth rates in the highest viscosity of crude oils. Bae et al. [8] explored the effect of oil viscosity in the range of 10–101500 cst on fatigue crack growth rate (FCGR) of 316 stainless steel tested in silicone oil. The results demonstrate that the viscosity has four orders magnitude did not effect on FCGR. Wang et al. [9] investigated the influence of loading waveform at different applied potential on the crack propagation of A537 steel tested in the 3.5 % NaC1 neutral solution. They are determined and discussing the mechanism of crack propagation and the effect of loading waveform on fatigue crack propagation. Alessandro et al. [10] inspect the influence of glass fiber frequency and load conditions on the fatigue behaviour of injection moulded neat and fiber strengthened polypropylene. They demonstrate that the resistance to fatigue crack propagation increases as the fiber volume fraction increases and the fatigue crack is tremendously dependent on the directions of crack propagation. Ebara [11] briefly summarized based on experimental results the corrosion fatigue behaviour of KA36 and KAS steel used for building of crude oil ship hull structures used to transfer the sour crude oil solution. His results demonstrate that for ship hull structures, to obtain a reasonable fracture control and fatigue life design much more information influence on corrosion fatigue behaviour is needed. This information's effect on the mechanical and metallurgical of strength steels. Tang et al. [12] utilized numerical technique to study the dependence of the applied energy release rate on the crack length in the new fracture specimen consists of invar plate coated by epoxy coatings under thermal fatigue test. Their results demonstrate that increasingly thermal cycles produce bigger fatigue crack growth rates. Likewise, the results show that the isothermal fatigue tests will presumably be inadequate to foresee thermal fatigue crack growth rate in epoxies. Blackman et al. [13] studied the fatigue behavior of epoxy coating polymer reinforcement by nano-silica particles. They demonstrated that the append of nano-silica particles into the epoxy polymer has enhanced the durability, toughness and fatigue behaviour of the epoxy polymer. Wu et al. [14] study corrosion fatigue behaviour of two types of epoxy coating used for watering tanks. They show that coating failure behavior due to fatigue is of great importance and cyclic thermal strain is considered dominant in range (0.2% - 0.3% of strain). Wei et al. [15] study the corrosion fatigue behavior of coated and uncoated AZ31 magnesium alloy by epoxy coatings in 3.5 wt. % Na_2SO_4 solution. Results demonstrate that the fatigue limits of the coated specimens were observed to be improved. Tongue [16] study the performance of epoxy based water coating utilized for ballasting tank under static and fatigue loading. His results show the static and fatigue life of the coats strongly depends on the toughness and coating thickness.

The aim of this paper presents a comparative study of fatigue limits of three classes of epoxy coating. Rotating bending fatigue test was carried out on uncoated and coated specimens in case of single and double layers to obtain the S–N diagrams. The fatigue test achieves in air and in the different types of crude oils collected from different fields of Basrah city southern Iraq. Microscopic observation is used to illustrate the effect of environment on fracture surface after fatigue test.

2 Epoxy coating and crude oil samples

2.1 Types of Epoxy Coatings

The epoxy coating is two-part of liquid state: the base containing epoxy resin and the hardener as reactant. These two parts are mixed shortly prior to application. After blended procedure, the base and hardener start to react and forms a three dimensional cross-linked solidifying structure. In this study four types of commercially epoxy coatings produced by Hempel Paints Company of Emirates LLC, Sharjah, U.A.E. were used for studying (one type used as primer undercoats). These coatings are:

- Hampel primer undercoats 13201, consisting of urethane-modified alkyd primers with zinc phosphate pigments produced by PT-Hampel of Indonesia ltd.
- HEMPADUR 15600 is a two-component, amine adduct cured pure epoxy paint. The coat’s thickness ranges from 160 to 200 µm.
- HEMPADUR 85671, this is a two component solvent free amine cured phenol Novolac epoxy coating. The thickness of the coat is in the range of 250 to 600 µm.
- HEMPADUR multi-strength GF 35870 is an amine-adduct cured epoxy coating. This type is reinforced with glass-flakes. The maximum thickness of this coating is 350 µm.

Refer to the type of coats as: HEMPADUR 15600 is model A, HEMPADUR 85671 is model B and HEMPADUR multi-strength GF 35870 is model C.

2.2 Crude Oil Samples

The test crude oil samples were collected from four different fields located in the different regions of Basrah province southern Iraq named: north Rumaila, south Rumaila, Zubair and west Qurna. Table 1 shows the physical properties of these types of crude oils. These properties are obtained from the laboratory of the oil fields.

Table 1 Physical properties of crude oils.

Properties	Fields			
	North Rumaila	South Rumaila	Zubair	West Qurna
API	33.8	30.2	34.1	32.31
Carbon residue %	7.3	6.33	3.95	8.4
Sulfur %	3.9	2.92	1.1	4.3
H_2O %	1	0.75	0.2	0.9
N_2 (g) %	0.13	0.98	0.09	0.079
TAN mgKOH/gm oil	0.37	0.112	0.06	0.333
NaCl ppm	14	13	3.8	10.81
Asphalt %	2.2	1.45	0.5	0.94
H_2S (g) ppm	1.1	1	07	1.5
CO_2 (g) ppm	3	3	23	2.5
Viscosity (cst)	12.1	17.6	5.8	28.3
Vanadium ppm	43	55.7	31	62

3 Fatigue Test

The rotating bending fatigue test has been conducted on samples of ASTM A537 C1 carbon steel which is widely employed in constructing of petroleum storage tanks in southern Iraq. The ASTM A537 C1 steel have the following chemical compositions (wt. %): Fe: 97.58, C: 0.24, Mn: 1.6, P: 0.035, S: 0.035, Cr: 0.025, Si: 0.5, Mo: 0.8, Ni: 0.25, Cu: 0.35 [17]. The identical form fatigue experiment specimens within the form of smooth cylindrical specimen are used for testing [18, 19]. A fatigue test sample has three areas: the test section has a radius of 4 mm and the two grip ends have radius of 9 mm. The grip ends are designed to transfer applied load from the rotating bending machine grips to the test specimen section. The dimensions and configuration of test specimen are shown in Fig.1 A and B respectively.

Figs.1 C to H shows the samples after coats by the three types of Hampel coats. The primer layer is initially applied directly to the dry-cleaned steel surface and left to dry for 2 days. The three classes of Hampel coatings were applied by airless spray to primer layer on the steel-substrates at nominally standard dry film thicknesses as given by produced organization. The main function of the primer was to wet the steel surface and enables the subsequently applied coats to adhere to the steel surface much better. The thickness of the coatings applied was measured using a paint thickness tester (Gain Express Holdings Ltd, China) to guarantee the coat's thickness in range of applications. The thickness as average value of the single and double layers of coatings are about 180 µm and 250 µm of the three typical coats used. The epoxy coated specimens were left to cure for 14 days at 25ºC and atmosphere pressure. A total of 28 specimens have been used for testing in air and crude oil solutions.

The fatigue tests were performed in air and crude oil solutions at (25°C) and atmospheric pressure using a group of equipment constructed in a laboratory, including rotating bending fatigue machine type (SM 1090, Hi-Tech Ltd., UK). The crude oil solutions have been pouring on the gauge part of the specimen during the test. The fatigue tests in crude oil environments have been performed in a (35 cm × 35 cm × 25 cm) closed chamber by spraying the crude oil with a volume flow rate of 1 m^3/h at ambient condition.

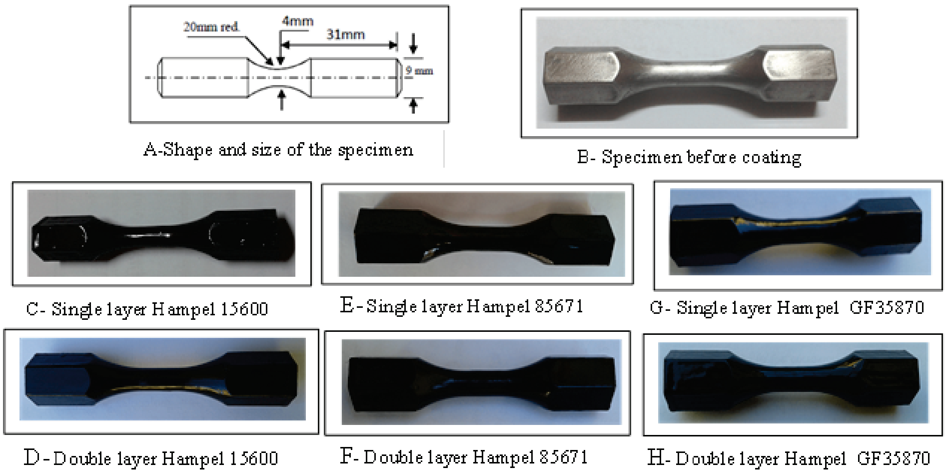

Fig. 1- Shape and size of the specimen before and after coatings

Fig.2 illustrates the group of equipment collected in a laboratory and used in the fatigue test in the crude oil solution.

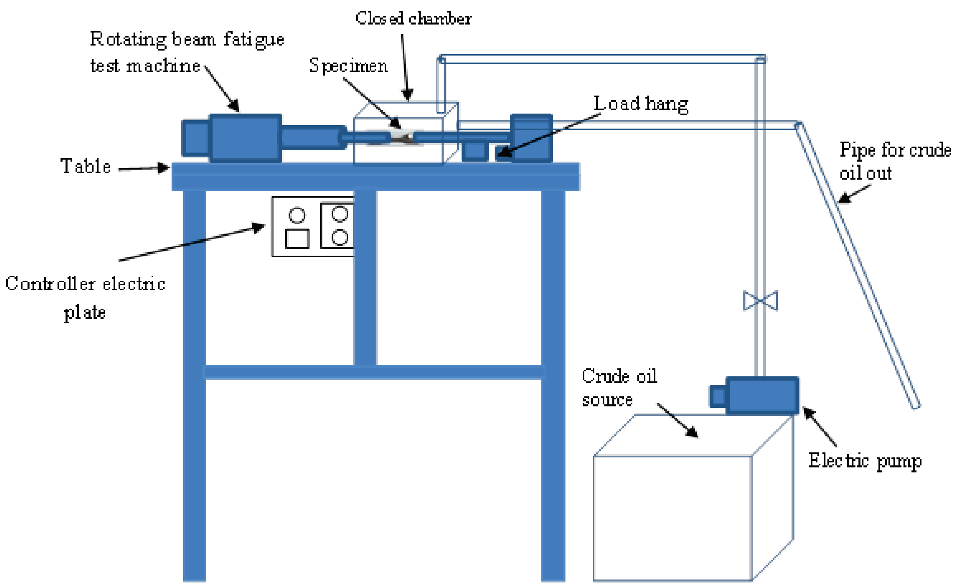

Fig. 2- Schematic diagram of the equipment group used in the fatigue testing in various environments.

All fatigue tests were achieved at loading frequency 60 Hz and at cycle range (10^1 to 10^7) cycles. The increase or reduction rate of fatigue limits as percentage value for epoxy coatings in different environments was calculated qualitatively using the formula [20]:

$$\%\ FL = \frac{\sigma_c - \sigma_{uc}}{\sigma_{uc}} \quad (1)$$

Where σ_c and σ_{uc} are the fatigue limits of the coated and uncoated specimens respectively.

4 Results and Discussion

In order to obtain the S-N diagram of uncoated and coated samples by single and double layers of epoxy coatings in the air environment, rotating bending fatigue tests were carried out in the air environment at ambient atmospheric conditions. The fatigue test results are shown in Fig.3.

The S-N curves in Fig.3 showed that the fatigue properties of uncoated steel A537 alone are lower than the fatigue properties of coated steel by the three types of epoxy coatings. The applied stresses as a function of the number of cycles to failure for the steel A537 alone and for that coated by single and double layers are similar to the changed tendency. It can be seen from Fig.3, that at the high stress level, there is a small drop in fatigue life up to 2* 10^3 cycles, but after this cycle the slope begins to increase to large values. That is due to the low fatigue performance which was caused by the crack initiated and spread through the tested specimen.

The final point in each curve of Fig.3 indicated that the specimen was not fractured at 10^7 cycles, but after this cycle. As shown in Fig. 3, fatigue tests for high stress were initiated and this high stress was gradually reduced during tests of the specimens until the fatigue limit of coated and uncoated steel was obtained. The fatigue limit was defined as the maximum applied stress a material can withstand and obtained at fatigue lifetime of 10^7 cycles. A fatigue limit is a characteristic of coated steel material and its geometry. Table 2 listing the fatigue limits values of all coated and uncoated specimens tested in air environment at 10^7 cycles obtained through the parallel projection method in the S-N curves of Fig.3.

The improvement in fatigue life and in the fatigue limit of the steel A537 may be seen from the S-N curves after the epoxy coatings have been applied. These improvements in the fatigue limit values and fatigue life were attributed to the epoxy coating adding strength to the A537 steel and due to adhesion force between epoxy coating layer and steel surface, the epoxy layer strengthen the test specimen during tests. Likewise, the epoxy coats applied contribute to reduce the ability of initiation and growing of cracks on surface of the steel sample that can cause the fatigue failure during rotating bending test. Therefore, both the fatigue life and the fatigue limit of the coated samples may be increased.

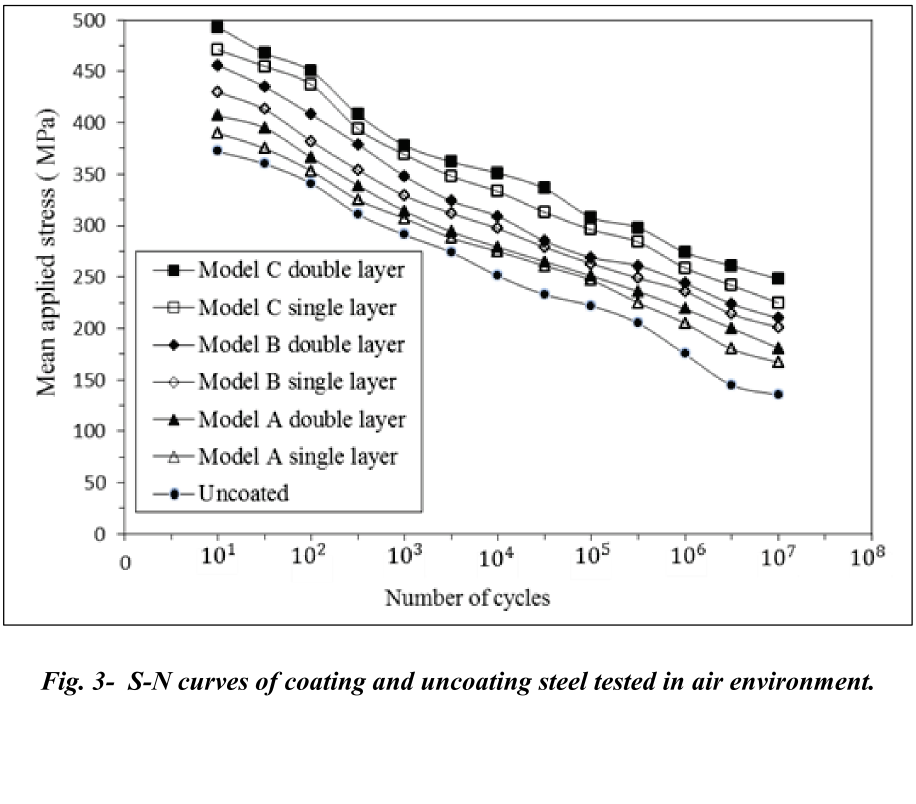

Fig. 3- S-N curves of coating and uncoating steel tested in air environment.

Table 2 Fatigue limit values at air environment.

Environments	Fatigue limit values (MPa)						
	Uncoated	Model A		Model B		Model C	
		Single layer	Double layer	Single layer	Double layer	Single layer	Double layer
Air	136.2	167.8	181.6	201.2	210.4	225.1	248.3

As it is clear from Fig. 3, the applied coating in case of two layers improves fatigue resistance and gives a high fatigue limit compared to the single coat layer. The difference of fatigue strength between the single and double layers of coating is large at low cycles, but it converges at 10^4 cycles and at the end of the test becomes a very small difference especially in the case of model B coats. This has been attributed to the fact that the bond strength between epoxy coating compositions and between coating and steel substratum becomes law in large cycles which reduces the strength and causes initiation of cracking in coating layer and spreads to the steel substrate and then failures. Consequently, any crack that develops acts as a stress concentration level riser and contributes to an increase in the fatigue failure.

Glass-flakes reinforcement in model C coating is a significant influence factor of fatigue performance since it has improved internal coats strength and enhances the physical and mechanical properties of coats. The glass-flakes can be increasing restriction of the segment movement of the epoxy coating chains, thus increasing the cross-linking density of the coating network. The glass-flake improve the coating fatigue behavior and resistance to cracks compared to other types of coats. As shown in Table 2, the fatigue limit obtained when using model C coats is twice value of that uncoated sample.

Figs. 4, 5, 6, and 7 shows S-N curve obtained from the test of coating specimens in the typical four different types of crude oils solutions. Fig.8 compares of the S-N curves of uncoated steel A537 tested in air and various crude oils. Comparing the fatigue test results of Figs. 4 to 7 with those of Fig.8, it has been observed that for all coated specimen, there was an improvement in the fatigue life performance and an increase in the mean applied stress compared to uncoated case. In addition, some dispersed data in the results of fatigue tests can be clearly seen. Similarly, in all samples, higher increases in fatigue strength can be observed in the entire fatigue range, but the changed increments of uncoated specimens are higher than those of coated. These results reflect that the fatigue performances of the coated specimens were better than those of the uncoated specimens when tested in the crude oils.

Table 3 shows compare of fatigue limits of the three models of coatings with those of the uncoated in the four types of crude oil solutions obtained by method of parallel projection of Figs. 4 to 7. It is clear that the fatigue limits obtained from fatigue testing crude oil of west Qurna have the largest values compared to other types of the crude oil solutions. This was due to the composition of each type of oil and properties, in particular viscosity, which has higher values in the crude oil from west Qurna (28.3 cst) as given in Table 1. The fatigue limit of the coated and uncoated specimen tested in crude oil is lower than the fatigue limit obtained from fatigue tested in air, as is clear from the comparison of the values of Table 2 and Table 3, but the fatigue limit is higher than that of the uncoated specimen. This was mainly due to the differences in service environments (air and crude oil) and the physical and chemical properties of different crude oil.

The percentage reduction rate of fatigue limits of various types of coatings tested in different crude oil and air environments were calculated by using Eq. (1) and the results indicated in Table 4. From Table 4, the greatest increase of the fatigue limit was obtained of model C coatings, with values of 90.5 and 97.98 for the single and double layer tested in the crude oil from Zubair field, while an increase of 66.6% and 83.56 % was observed during a test in air. Model A has the lowest valuable of percentage reduction rate of fatigue limits which have values of 17.76% and 31.39 % for both single and double layers tested in the crude oil from Zubair field and 23.43% and 33.77% for both single and double layers of epoxy coatings tested in air.

The highest values of fatigue limits obtained from fatigue testing in the crude oil from west Quran, while the lowering values obtained from tests in crude oil from Zubair fields. These behaviors of applied coats test is attributed to the crude oil physical properties and chemical adsorptions. The chemical adsorbs at, just like the chewing pasting on the specimen surface leads to make the specimen surface rough. Stress concentration easily occurred at the root of the chemical adsorption surface under fatigue loading, thus providing necessary conditions for fatigue crack initiation and the specimen fracture in crude oils.

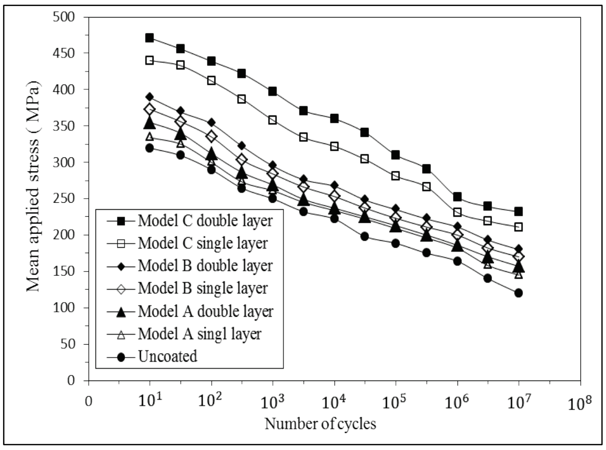

Fig.4- S-N curves obtained from testing of coating and uncoating steel in west Qurna crude oil

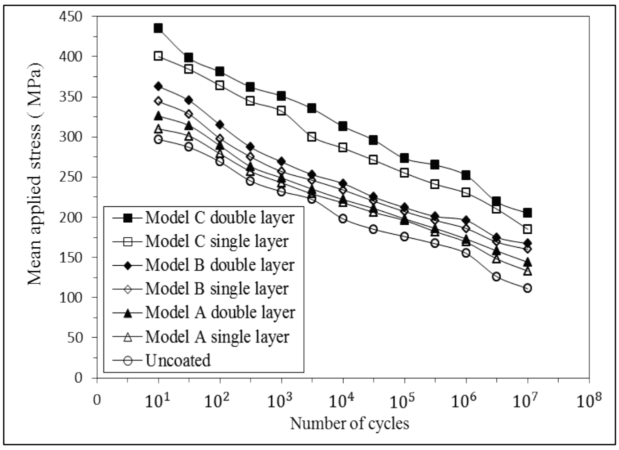

Fig.5- S-N curves obtained from testing of coating and uncoating steel in south Rumaila crude oil

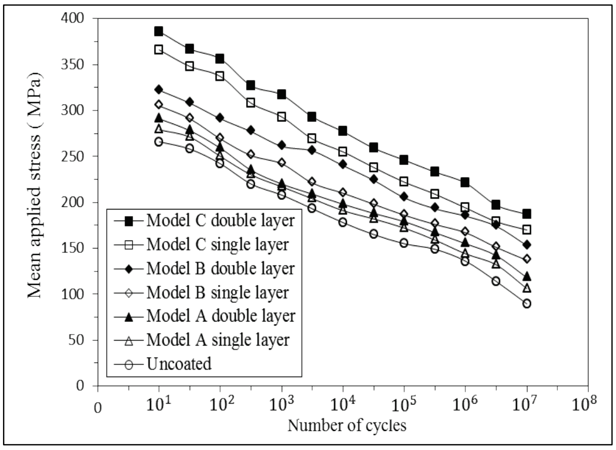

Fig.6- S-N curves obtained from testing of coating and uncoating steel in Zubair crude oil

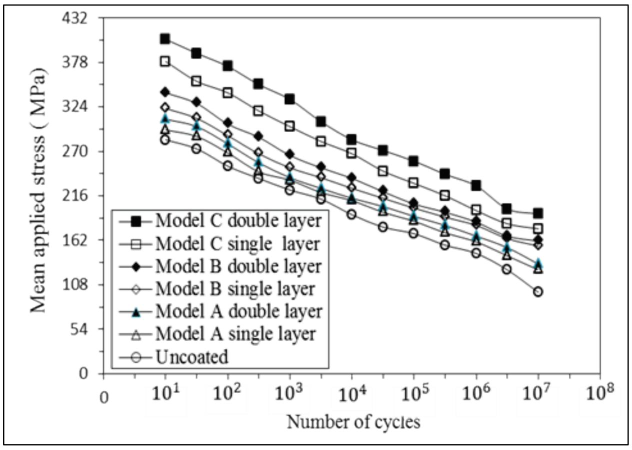

Fig.7- S-N curves obtained from testing of coating and uncoating steel in north Rumaila crude oil

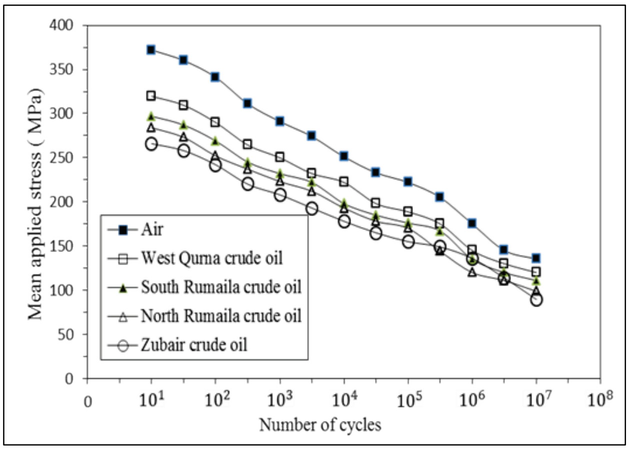

Fig. 8- S-N curves obtained from testing of uncoated A537 steel in different environments

Table 3 Fatigue limits of uncoated and coated steel in different crude oils.

Specimen	Fields			
	South Rumaila	North Rumaila	Zubair	West Qurna
Uncoated	111.4	98.7	89.5	120.3
Model A Single layer	133.3	126.2	106.3	145.2
Model A Double layer	144.1	133.1	118.6	157.5
Model B Single layer	160.5	155.4	137.2	170.6
Model B Double-layer	167.6	161.6	153.4	180.1
Model C Single layer	185.3	176.3	170.5	211.4
Model C Double layer	205.2	194.1	177.2	232.3

Table 4 Percentage increase and reduction in fatigue limits under different environment

Environments	Decrease rate of fatigue limit %	Increase rate of fatigue limit %					
		Model B		Model A		Model C	
	Uncoated	Single	Double	Single	Double	Single	Double
Air	----	48.56	55.21	23.43	33.77	66.6	83.56
South Rumail	17.73	43.94	50.40	19.95	29.11	66.33	84.2
North Rumail	27.05	57.33	63.32	27.65	33.33	78.6	96.5
Zubair	33.85	53.74	71.28	17.76	31.39	90.5	97.98
West Qurna	11.23	41.79	45.87	21.14	31.22	75.75	93.1

The crude oil is a complex mixture of more substances in particular water, materials, asphalt, dissolved gases and salts ($NaCl$, $MgCl_2$, $CaCl_2$ …). These salts significantly reduce the steel material fatigue strength. This was attributed to that the salts of attack upon the extrusions and intrusions at slip bands created in the surface, preventing work hardening and strengthening of steel metal test. In addition, the deposition of asphalt as a film on the surface of the specimen during testing, and due to this layer is thin and weak; it can become sites and points for initiations and propagation of cracks to surface of steel specimens. For this reason, the fatigue limit of uncoated and coated steel is low when testing in crude oil environments compared to air.

The fatigue performance can be affected by the compositions of each type of epoxy coating tested. Amine cured Novolac epoxy resins generally contain multiple epoxide groups (vinyl groups, alkyl group, aryl group ... etc.), the multiple epoxide groups allow this resin to achieve high cross-link's density, resulting high crack resistance compared for amine curing pure epoxy coats. The glass-flake in Hampel GF 35870 epoxy coating makes the components very cohesive, provides additional strength and increases the resistance to propagation of crack compared to other types of coatings. The flake-glass becomes as bridge that are perpendiculars to the crack propagation direction through the applied coatings.

Figs.9 to 13 illustrates the overall fatigue fracture surfaces (one side of the fracture specimen) of the uncoated and coated steel after the fatigue failure has occurred. As is clear from all these figures, the shape of the fracture surface of the specimens can be significantly different from Fig.9 to Fig.13 due to exposure to different environmental conditions and due to specific differences in the composition of the coatings.

From Fig.9, the A537 steel surface of the sample conducted in dry air illustrated sharp edges, several cracks and defects. The cracks are initiated from several sites on the surface. The double layer coating of model B has exhibited more cavities covered the surface, in contrasts the single layer indicates fewer cavities and some concaves. On the other hand, both single and double layer of model A show inclined fracture surface, edges and pits appeared in the surface. The single layers of model C shows the large coat's failure on surface compared to double layers of coats which indicate extra cracks propagation in direction alongside of the fracture specimen surface.

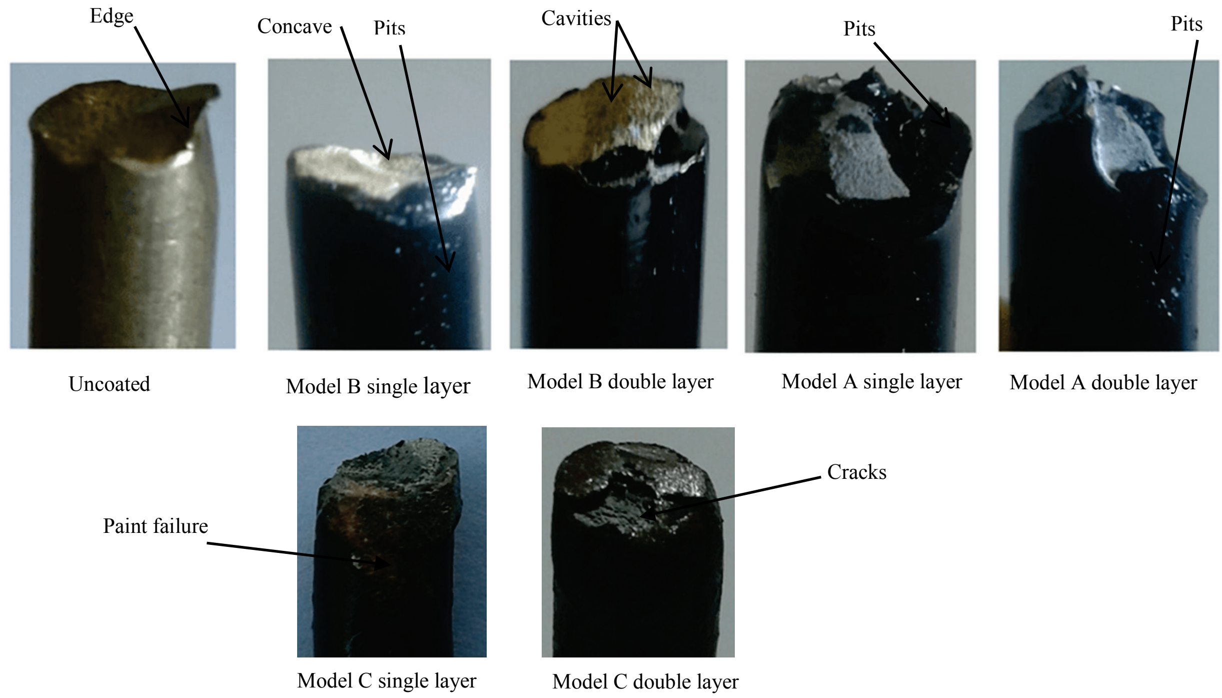

Fig. 9- Fracture surface of specimens after fatigue test in the air.

Its illustrated from Fig.10, the fatigue fracture surface of the A537 steel examined in the west Qurna crude oil have the concave-convex regions. It clears that the spread of cracks from the marginal regions to the center. Also, it can be seen that there are different radial cracks along the fracture surface extension from edges to the center of specimen. The model B double layer coats shows V-shape fracture surface and have knife edges compared to the single layer. The model A of single layer coat shows flat surface edges, coating failures and pits at specimen surface. Model C, both single and double layers, the fracture surface shows circular fatigue cracks, pits and coats failure. The circular crack appears to have been nucleated at the edge section of the steel coating and propagates toward the center of specimen.

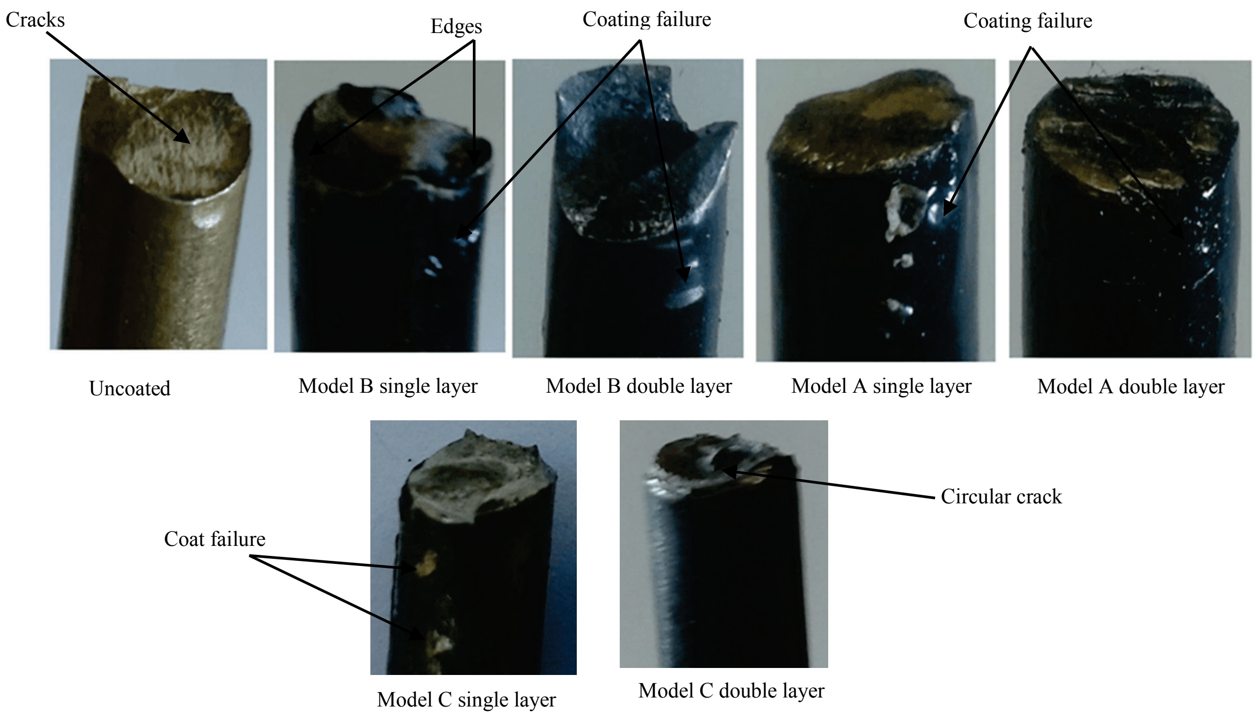

Fig. 10- Fracture surface of specimens after fatigue test in west Qurna crude oil.

The uncoated samples in Figs.11, 12 and 13 showed the sharp edges on the side of the broken sample surface and some concaves on the surfaces. There is a large difference in the way of the fraction surface of coated specimen tested in air and crude oils. It can be seen the epoxy coats deteriorates due to the influenced of fatigue stress. The single layers of model A coat has more deteriorate near fracture surface compared to double layers as indicated in Figs.10, 11, 12 and 13.

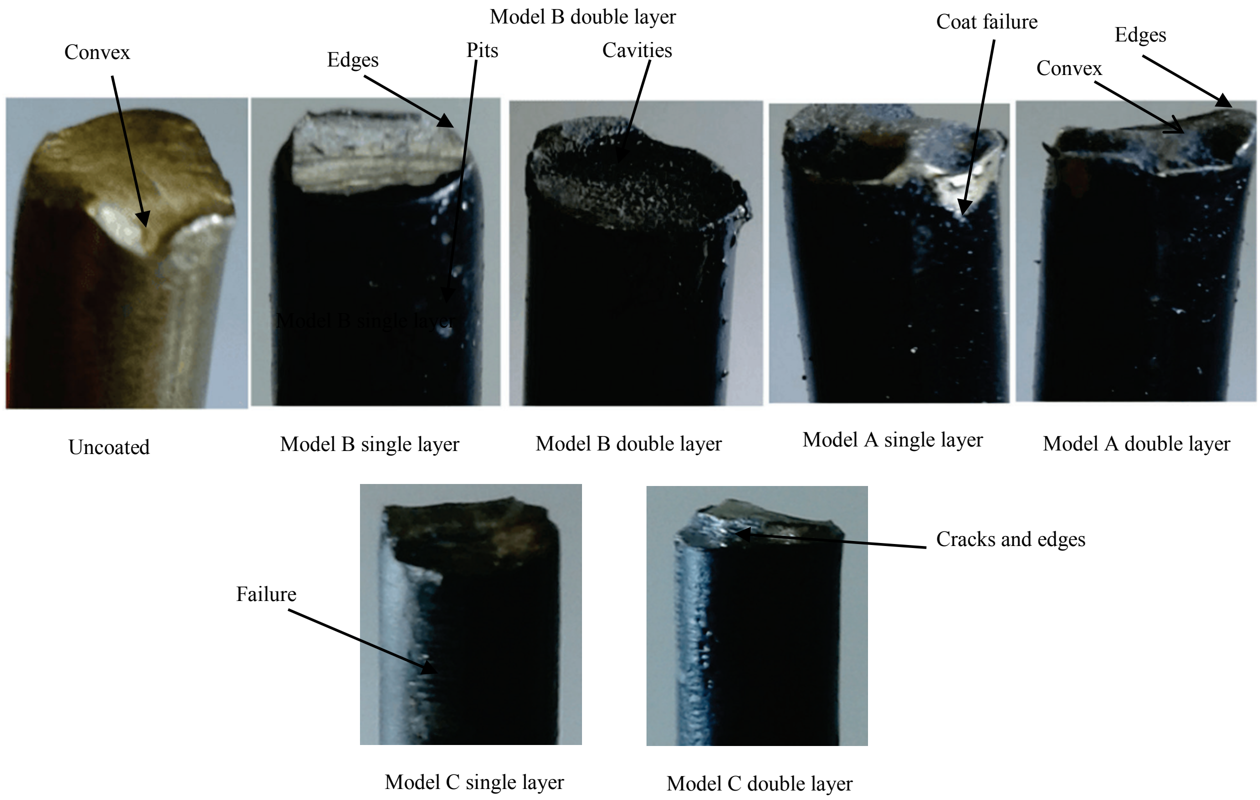

Fig. 11- Fracture surface of specimens after test in south Rumaila crude oil.

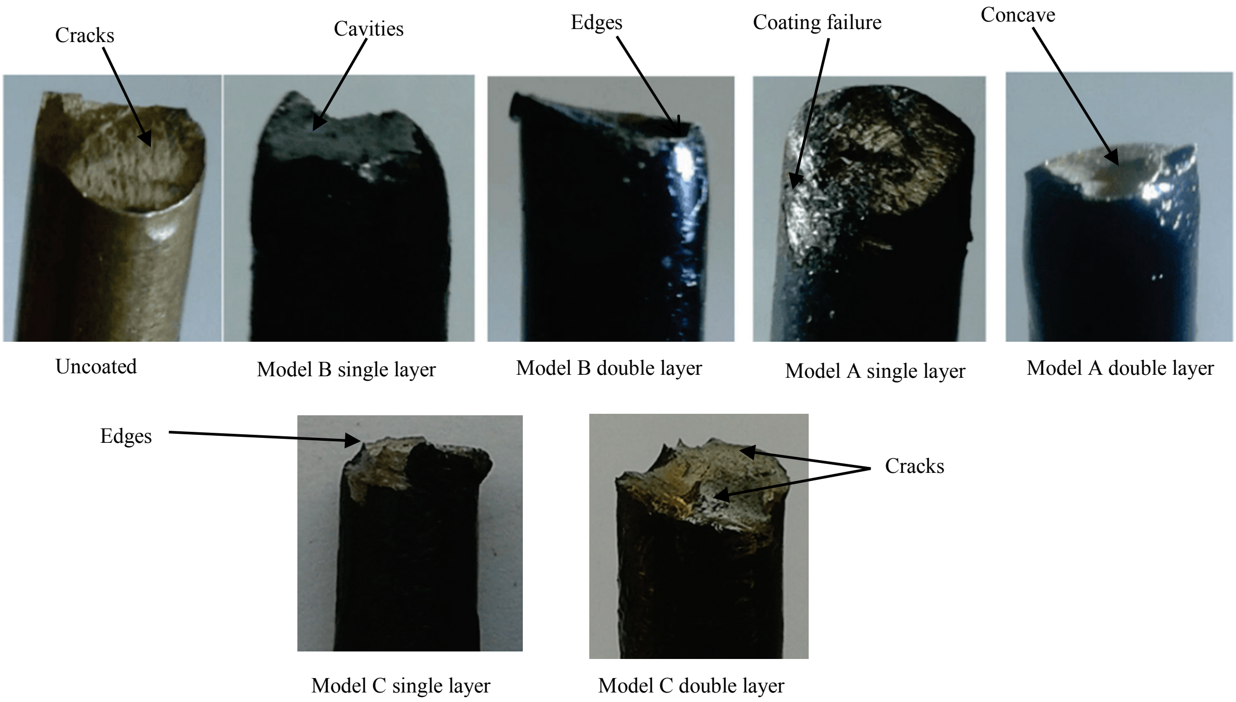

Fig. 12- Fracture surface of specimens after test in north Rumaila crude oil.

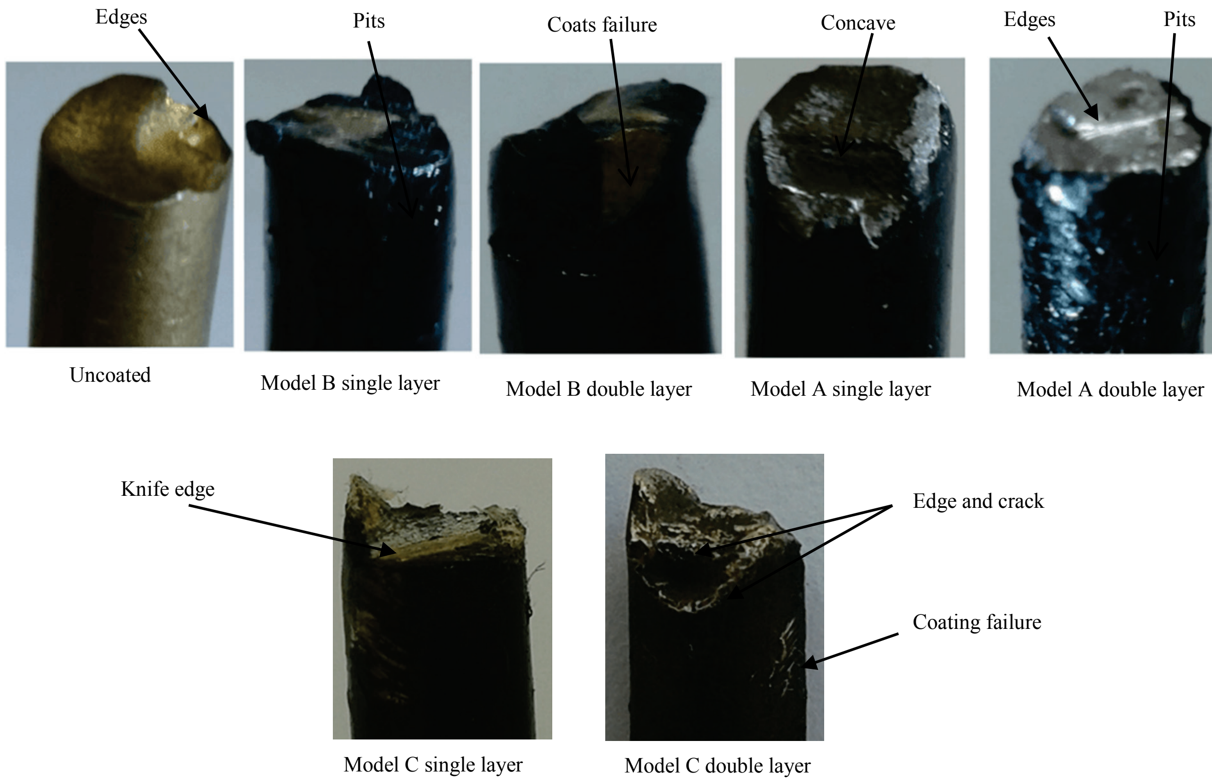

Fig. 13- Fracture surface of specimens after test in the Zubair crude oil.

This can be seen from the shapes of specimens fracture surface that there are different crack emergence mechanisms that lead to the coated steel's fatigue failure. The first mechanism is the emergence of a circular crack in the specimen and spread in the perimeter of the specimen to the middle and results in sharp edges initiated. This type starts and prevalence at specimen peripheral and continuous during a test until the complete failure of the test sample is occurring. As mention earlier, the crude oil has different compositions, in particular the asphalt, which adheres to the surface of the fatigue specimen and initiates a thin membrane on the surface, which can be a catalyst for the emergence of this kind of fatigue cracks. The second type is the emergence and spread of crack as incision through the specimen material. The incision generates mostly close to the edge of the specimen and spreads longitudinally to the center of the specimen until the complete fatigue failure occurs. Material defects such as bubbles and pores can help initiate and spread this type of crack. The third type is the emergence of crack in the center of the specimen in the form of a slit and spreading it inclined diagonally to the edge of the sample surface. The inclusion found in steel may be generated and pusher this type of crack.

5 Conclusion

The fatigue performance of A537-C1 steel in cases of uncoated and coated by the three types of epoxy coatings used for applications of crude oil storage tanks was investigated using rotating bending fatigue tests. From the above discussion, the following conclusions can be recorded:

1. The fatigue limits of ASTM A537 C1 carbon steel conducted in air and crude oils have been improved after the application the epoxy coatings.
2. The fatigue limit of the coated and uncoated specimen conducted in the crude oil solution is lower than that obtained in the air environment.
3. The application of double layer epoxy coats improves the fatigue limit and increases the fatigue life of steel by small values compared to single coat layer.
4. The initiation of fatigue cracks in tested coated steel depending on the composition of epoxy coatings and environment.
5. Microscopic inspection shows that the environment affects the shape of the specimens fracture surface. It has also been found that after fatigue testing, the single layer of coats has some pits and coats failure on the surface of the coated specimens compared to doable layer coats.

6. There are three mechanisms of cracks initiation during coat fatigue failure: the first is the emergence of a circular crack in specimen tested, the second is the emergence and spread of crack as incision through the specimen material and the third is the emergence of cracks in the centred of specimen and spreading through the sample thickness.

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