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Macro-microstructuctural studies of stress corrosion cracking (SCC) of mild steel in simulated environments

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Abstract
Macro-microstructuctural analysis of stress corrosion cracking *(SCC)* of mild steel in simulated environments has been carried out. Mild steel samples used were all machined and suspended in 1M concentration of H_2SO_4, *HCl, NaOH* and distilled H_2O. Except for the control samples, each specimen was suspended and tensed to stress values of *126.8, 169.1, 211.4, and* $253.6N/m^2$ for a period of 360 hours. Macrostructural analysis show that corrosion attack on the samples increased with increase in stress values. Microstructurally, crack initiation, development and propagation were observed to increase with increase in stress values for specimens suspended in H_2SO_4, and *HCl* environments. Comparatively, specimens suspended in H_2SO_4 environment are more susceptible to stress corrosion attack than those in *HCl.* In *NaOH* and H_2O environments, the specimens exhibited little or no stress corrosion attack.

Keywords: Stress, Corrosion, Crack, Macrostructure, Microstructure, Propagation, Environment

1. Introduction

Stress corrosion cracking can be defined as crack formation due to simultaneous effect of static tensile stresses and corrosion *(Ede, S.E. 2012)* [3]. It is a localized form of corrosion which normally occurs with simultaneous existence of other forms of corrosion. Its damage is not obvious to casual inspection and is characterized by production of marked loss of mechanical strength with little metal loss. Several major disasters have involved stress corrosion cracking, including the rupture of high-pressure gas transmission pipes, the explosion of boiler, and the destruction of power stations and oil refineries *(Parkins, R.N. 2002)* [11].

Though there are no general rules which apply to one particular material or one specific environment since the same material may show quite different modes of fracture in different environment. However, most metals will exhibit SCC when stressed in specific environment while some do not. The important variables that determine length of time before failure are level of stress, environment, temperature, metal composition and metal structure *(Lees, D. J. 1982)* [9].

There is general agreement that the initiation of stress corrosion cracking takes place at a surface pit or notch. A similar defect may occur if there is a break in a surface film permitting local corrosion to take place. However, “theories of Trans granular cracking” and “grain boundary cracking explain that stress corrosion cracking in many alloys suggest that plastic deformations take place at the crack tip. A combination of an applied tensile stress and corrosive environment on the small area at the crack tip results in the yield stress being exceeded in this local region. Thus, material at the crack tip deforms plastically as the tip is being opened slightly. This process would fracture any protective film present. As the tip yawns, fresh solution is drawn in to dissolve the highly anodic metal in the tip at favourable sites such as emergent dislocation. Therefore, concentration polarization which would reduce the corrosion rates at the crack tip is prevented to a large extent due to the constant dilution of the solution by the yawning action. Note, the sizes of the crack take little part in the process and are weakly anodic. The current density values indicate lower corrosion rates prevalent at the sides of the crack *(Moore, J. J. 1981)* [4].

The aim of this work is to reveal the mechanisms of stress corrosion cracking on mild steel in some environments where it is normally exposed while in service. It is also geared towards to estimate the time it takes before failure can occur in these environments that are aggressive on mild steels.

2. Experimental Materials and Procedures

Experimental procedures undertaken involved the study of macrostructures and microstructures analysis of stress corrosion cracking on mild steel. The mild steel used for this study was obtained from Delta steel company Aladja, Delta State, Southern part of Nigeria. Its chemical compositions and other mechanical properties are as shown in table 1. The chemicals used (*H_2SO_4, HCl, NaOH* and distilled *H_2O)* were obtained from conventional laboratories with the percentage purity clearly indicated.

Table1: Chemical compositions of the specimen

620577	Heat No
0.26	Carbon
0.77	Manganese
0.26	Silicon
0.013	Sulphur
0.021	Phosphorus
410	Yield Strength
602	Ultimate Tensile
24	Elongation (%)

The steel specimens were machined and holes drilled at both ends for suspension in the facility for SCC test. Constant stress or constant displacement test where specimens and a loading method that stresses the specimens while exposed in the solution was used. The susceptibility to SCC was assessed by the time taken for development and propagation of cracks on the surface of specimen. Here, the specimens were tested simultaneously in four different environments of *(1MH_2SO_4, 1MHCl, 1MNaOH and H_2O)*. A set of four steel specimens was each fixed in four stainless cups through holes drilled at the lower ends of the specimens. At the upper part of the specimens, steel springs were each passed and hooked through the hanging pulleys and finally tensed to stress. In each of the four stainless cups, 1M solution of *(H_2SO_4, HCl, and NaOH)* and water were used and each set of experiment was allowed for 72 hours for five consecutive times. At the end of every experiment, the set was dismantled and the specimens removed, washed, dried and examined macro structurally by visual inspection. Microstructural examinations were also done by metallographic procedures such as cutting, filling, grinding, polishing, etching and microscopic examination of grain to reveal crack initiation, development and propagation within the microstructure of the specimens. Stress values of *126*.8, *169.1*, *211.4* and *233.6 N/m^2* were used on the specimens during the experiment; while control specimens were not tensed to any stress, but were immersed in the same environments with stressed specimens.

3. Experimental Results and Discussion

Plates 1-4 show the macrographs of the steel specimens after subjecting them to various levels of stress

Plate 1: Macrograph of specimens tensed to various levels of stress in 1MH_2SO_4 environment.

Plate 2: Macrograph of specimens tensed to various levels of stress in 1MHCl environment.

Plate 3: Macrograph of specimens tensed to various levels of stress in 1MNaOH environment.

Plate 4: Macrograph of specimens tensed to various levels of stress in H_2O environment.

Plate 5a: Micrograph of specimen tensed to 253.6N/m^2 for a period of 360 hours in 1MH_2SO_4 environment. (x500), temp (25 ^{0}C).

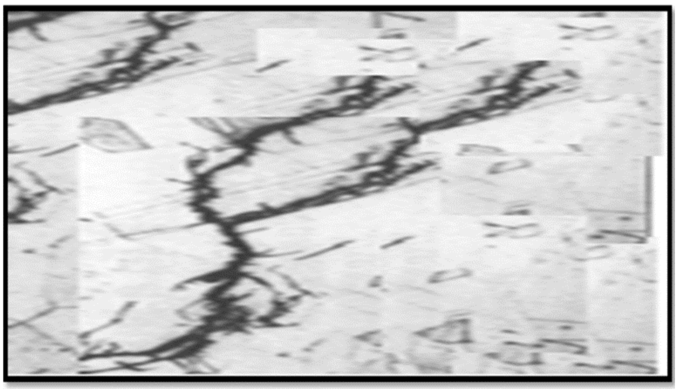

Plate 5b: Micrograph of specimen tensed to 211.4N/m^2 for a period of 360 hours in 1MH_2SO_4 environment. (x500), temp (25 ^{0}C).

Plate 6a: Micrograph of specimen tensed to 253.6N/m^2 for a period of 360 hours in 1MHCl environment. (x500), temp (25 ^{0}C).

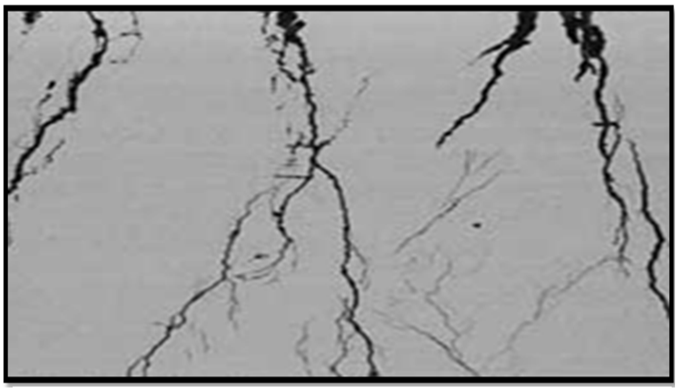

Plate 5c: Micrograph of specimen tensed to 169.1N/m^2 for a period of 360 hours in 1MH_2SO_4 environment. (x500), temp (25 ^{0}C).

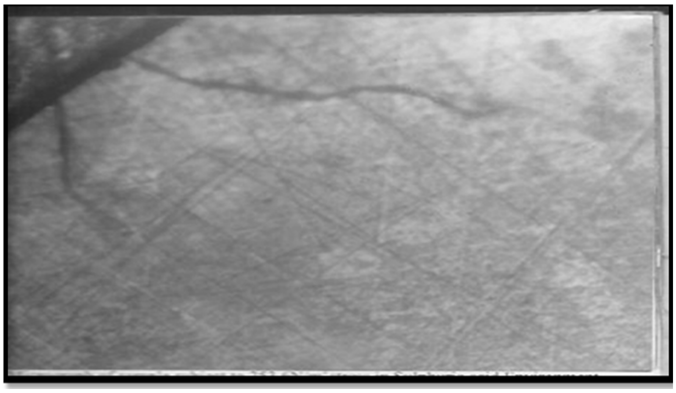

Plate 6b: Micrograph of specimen tensed to 211.4N/m^2 for a period of 360 hours in 1MHCl environment. (x500), temp (25 ^{0}C).

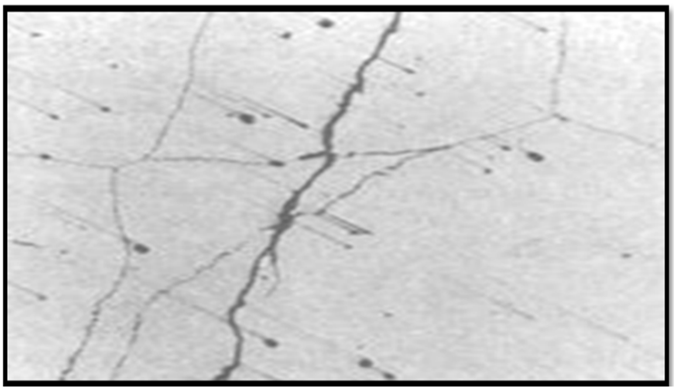

Plate 5d: Micrograph of specimen tensed to 126.8N/m^2 for a period of 360 hours in 1MH_2SO_4 environment. (x500), temp (25 ^{0}C).

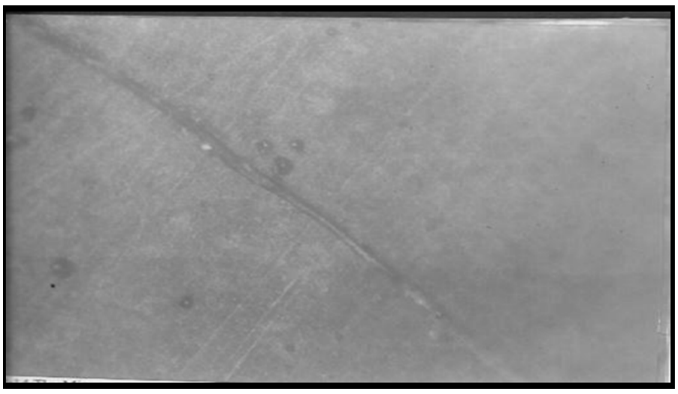

Plate 6c: Micrograph of specimen tensed to 169.1N/m^2 for a period of 360 hours in 1MHCl environment. (x500), temp (25 ^{0}C).

Plate 5e: Micrograph of control specimen immersed in 1MH_2SO_4 environment for a period of 360 hours. (x500), temp (25 ^{0}C).

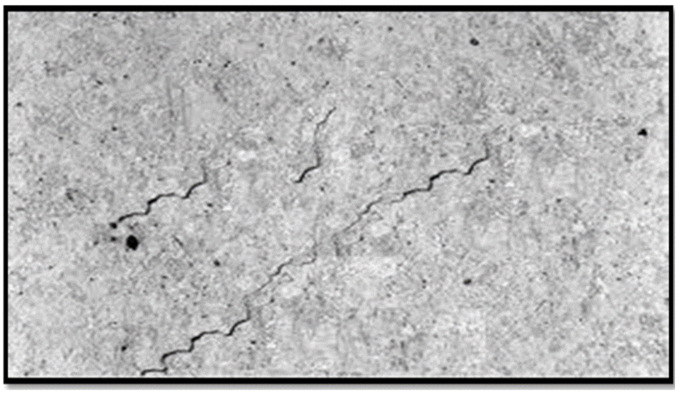

Plate 6d: Micrograph of specimen tensed to 126.8N/m^2 for a period of 360 hours in 1MHCl environment. (x500), temp (25 ^{0}C).

Plate 6e: Micrograph of control specimen immersed in 1MHCl environment for a period of 360 hours. (x500), temp (25 ^{0}C).

Plate 7: Micrograph of specimen tensed to 253.6N/m^2 for a period of 360 hours in 1MNaOHenvironment. (x500), temp (25 ^{0}C).

Plate 8: Micrograph of specimen tensed to 253.6N/m^2 for a period of 360 hours in H_2O environment. (x500), temp (25 ^{0}C)

Visual inspection of the specimen shows that those stressed in H_2SO_4 *(plate 1)* were seriously attacked by the environment. Presence of stresses on the specimen surfaces made them to neck with obvious appearance of notches and cracks on these surfaces. These cracks were not just surface cracks, but were believed to have propagated into the specimen. It was also observed that those specimen stressed in *1MHCl (plate 2)* were moderately attacked by the environment as cracks and notches were sparsely seen on their surfaces. Therefore, sulphuric and hydrochloric acid environments propagate SCC and other forms of corrosion on mild steel. Moreso, plates 3 and 4 which represent those specimens tensed in *NaOH* and H_2O respectively were not attacked. It clearly shows that mild steel is not susceptible to SCC in these environments. Further macroscopic investigation revealed that those specimens stressed in H_2O environment suffered from uniform corrosion to relatively low rate while NaOH was discovered to be an inhibitor on mild steel as those specimens stressed in its solution were seen with their lustre colour thorough out the experimental period. Consequent upon this, *NaOH* and H_2O environments do not propagate SCC on mild steel.

The microstructures of specimens used in the four environments studied are presented in plates 5 - 8. Plates 5(a-e) represent those specimens stressed in $1MH_2SO_4$ at various levels of stress. Microstructural examinations revealed levels of initiation and propagation of cracks in each of the micrographs examined. It was also observed that the higher the level of stress the more pronounced were the development of cracks in each specimen, with plate 5a seen to experience sevier cracks while plate 5d had less crack. It was also discovered that plate 5e (control micrograph) had no crack.

These developments are in conformity with what was observed in plate 1 earlier discussed. Furthermore, plates 6(a–e) represent microstructures of those specimen stressed in *1MHCl* acid environment under various levels of stress. Crack initiation and propagation were observed to increase as the levels of stresses increased except for the control plate (6e) which has no crack. However, developments of these cracks were less when compared with those specimens that were stressed in sulphuric acid environment. Generally SCC and other forms of corrosion propagate in these two environments. An explanation to these could be based on three conditions necessary for SCC to occur which had earlier been discussed in the literature of the work. They are (1) Hydrogen embrittlement (2) Film-induced cleavage and (3) Active path dissolutions. However, theories of transgranular and grain boundary cracks also gave a clearer explanation to these behaviors. Finally, fractures of mild steel in these environments are very much possible *(Parkins R. N. 2002)* [11].

Plates 7 and 8 represent microstructures of those specimens stressed in *1MNaOH* and water environments respectively. At the highest level of stress used in this experiment, specimens in these environments never suffered any form of SCC. The result is in conformity with the plates 3 and 4 earlier discussed. It shows that these environments do not propagate SCC. Explanation to this could be attributed to the behavior of neutral solution like water on steel. It states that when steel is exposed to near-neutral solution like water, the solubility of the protective oxide will be much lower than in acid solution and the extent of dissolution will tend to be smaller *(Moore, J.J. 1981)* [4]. It also further states that if the near neutral solution contains inhibiting anions like sodium hydroxide, the dissolution of the oxide film may be suppressed and the oxide film stabilized to form a passivating oxide film which can effectively prevent the corrosion of the metal which is then in the passive state. More so, one other factor that is known about SCC is that for many metals, if cathodic polarization is increased by methods of deoxygenation, inhibitors or cathodic protection, SCC may be delayed or eliminated *(Moore, J.J. 1981)* [4]. This may be an example of what happened in sodium hydroxide *(NaOH)* and *(H_2O)* environments since these environments are easily polarized cathodically, deoxygenated and not aggressive on mild steel.

4. Conclusion

The effect of applied tensile stress in some selected environments had been investigated on the macrostructure and microstructure of mild steel. The following deductions can be drawn

1) Corrosion attack, crack initiation, development and propagation thrive most in $1MH_2SO_4$ acid environment.

2) Susceptibility to stress corrosion cracking increases with stress values for all samples in *H_2SO_4* and *HCl* environments.
3) 3) Mild steel samples immersed and tensed in *1MNaOH* and water environments did not suffer corrosion attack, crack initiation, development and propagation during the period of the experiment at low and high stress levels.
4) Mild steel is not recommended to be used in *H_2SO_4* and *HCl* environments at both low and high stress levels.
5) NaOH was discovered to be an inhibitor on mild steel. Therefore, mild steel should be used in such environments during service.

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