

# Microstructural Characterization of Low carbon steel (AISI 1008) used in oil and gas pipeline

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**Abstract**—Result associated with microstructural and strength analyses of the material collected from the oil field are presented. The effects of varying microstructure and hardness by heat treatment process were presented. The microstructural characterization and surface analysis were carried out by scanning electron microscope (SEM), Energy dispersive spectroscopy (EDS), X-ray Diffraction (XRD), and Rockwell for hardness analysis. The energy dispersive spectroscopy EDS indicated that the samples were in the range of low carbon steel (AISI 1008) i.e. highly ductile and soft. Scanning electron microscopy SEM of the surface examination denotes that the material was greatly affected by erosion leading to crack initiation and propagation.

Keywords- Low Carbon Steel, Corrosion, microstructure, Hardness, Microscope

## 1. INTRODUCTION

THE Low carbon steel is very acceptable material due to its huge applications in the industries. It provides good deformability due to its low yield strength, minimum cost, simplicity in fabrication and manufacturing, good strength, toughness and good weldability. Different strengthening mechanism can be applied to enhance the grain refinement in order to increase the strength and toughness both simultaneously. Mild steel or low carbon steel consist a small fraction of carbon that gives better plastic deformation behavior in sealing and leakage prevention. Mild or Low carbon steel are universally used for structural applications, food production industries, automotive and aircraft industries; however, its poor corrosion resistance at ambient atmosphere condition is a matter of serious concern[1, 2]. All ferrous materials consist mainly of iron with small proportions of other alloying elements. These alloying elements have added to obtain specific characteristics such as corrosion resistance, strength and wear resistance to enhance the mechanical behavior of the resultant alloy. Most common steel alloying elements are aluminum,

vanadium, nickel, manganese, molybdenum, chromium, tungsten, boron and phosphorous.

Based on the carbon contents steel is divided into three major categories, low carbon or mild steel (wt. % 0.04-0.30), medium carbon steel (wt. % 0.31-0.6) and high carbon steel (wt. % 0.61-2.40). Increasing the carbon contents results in increasing the hardness of the materials and decrease in the ductility as well as corrosion resistance. High carbon steel can be used where there is hardness and strength is the first priority as in transporting, construction and relative motion between the parts, whereas mild steel can be appreciated in low cost and high ductile application environment. Despite the fact the low carbon or mild steel has high susceptibility to corrosion, is used in several application in which corrosion phenomena is a key factor[3, 4]. Many researches have been conducted to analyze the corrosion phenomenon of mild steel used in various industries. It has been investigated[5] that the corrosion first initiate at grain boundaries and spread to the bulk of the grains. Statically evaluation of the material indicates that the inhibition of the materials has greatly affected the corrosion process[6].

It is also indicated that the corrosion process has a great dependency on geotechnical characteristics of site, the presence of electrochemical corrodant and time period of the materials[7]. The effect of

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inhibition of the materials to increase the corrosion resistance can be enhancing by applying suitable coating parameter time and temperature[8]. Moreover; Hassane Lgaz et. al. has explained[9] the effect of different mixed inhabitant on the corrosion process of the mild steel, which leads to high increase polarization resistance. It is also concluded that the materials hardened by heat treated and recrystallization leads to increase the corrosion resistance[10]. The effect of zinc coating were studied[11] and it is concluded that zinc coating is a promising method of silane coating which is better inhabitant among the several inhabitant. It is investigated that the presence of several ferrous ions in the material can change the corrosion acceleration by one or more aspects[12, 13].

## 2. MATERIALS AND METHOD

Soft iron/Low carbon steel specimens were collected from Mari Gas oil field in the form of ring gasket as shown in Figure 1. For SEM analysis 16 samples, 8 uncorroded and, 8 samples of corroded material were prepared by cutting them into specified and same dimensions, polished the samples with 9-micron diamond paste and nylon cloth, and etched in two different Nital solutions. The two nital solutions were based on the concentration of nitric acid (HNO<sub>3</sub>) in ethanol (C<sub>2</sub>H<sub>5</sub>O). the two etchants containing 8% Nitric acid in ethanol and second 3% Nitric acid in ethanol. The polished samples were etched in two ways in order to get good and visible grains i.e. some samples were etched for 15, 30, 45, 60 and 90s in Nital containing 3% nitric acid while others were etched for a fixed interval of 60s in Nital containing 8% Nitric acid in Ethanol. A SEM analyzer was used to investigate the microstructure with a JEM-5910 (JEOL) SEM equipped with energy dispersive X-Ray electron spectrometer (EDS) was used for recording secondary electron images and chemical analysis of the samples. XRD analysis was used to indicate the crystal structure of both the corroded and uncorroded materials and the compared. Hardness of the material was determined by using Rockwell tester. EDS analysis was carried out to analyze the

constituents present in the material in the Material Research Laboratory (MRL), University of Peshawar, Pakistan.

## 3. RESULT AND DISCUSSION

The SEM analysis of the sample in 8% HNO<sub>3</sub> in ethanol is shown in Figure 2a at 200X. Samples etched for 30 sec revealed the granular structure; however the best result could be obtained for samples etched in 3% HNO<sub>3</sub> in ethanol in Figure 2b. The samples overetched for longer than 30 sec in 8% nital solution. But as the sample etched in the accurate nital solution and time i.e. 8% and 30 sec or 3% and 60 sec, a more visible structure obtained.

According to the lever rule low carbon steel (AISI 1008) contains 97.4% Ferrite ( $\alpha$ ) and 2.6% cementite (Fe<sub>3</sub>C). In SEM images Figure 3, only Ferrite could be observed due to its relatively higher concentration in the examined sample which had two phase BCC crystal structure. Moreover; it is clearly observed from Figure 4, that the pearlitic area is increases strongly after corrosion, this is due to the inclusion of carbon exist in crude oil under high temperature and pressure. As the carbon is absorbed under high temperature and pressure a chemical segregation effect is initiated in which a specific phases precipitated on the grain boundaries. The phase containing high percentage of iron sulfide FeS as observed in Figure 5.

The attack is usually related to the segregation of specific phase formed on grain boundaries. Corrosion then occurred by preferential attack on the grain boundaries, makes the grain boundaries weaker. As the grain boundaries is affected by corrosion and make it weaker so there is high temperature and pressure and crack is initiated and propagated towards the bulk of the grain as indicated in Figure 6.

The X-Ray test was used to find out the corrosion product. The maximum corrosion product were contain iron sulfide shown in the Figure 7. The corrosion product contains  $\alpha$ -FeOOH,  $\gamma$ -FeOOH and amorphous-like phase and the amorphous like phase contains more than 50% of the rust. The

thickness of the rust is uneven showing wavy and undulating near the surface of the steel and rust the amorphous phase take place.

EDS analysis of both the materials i.e. corroded and uncorroded is shown in Figure 8 and Figure 9 respectively. The chemical composition is C 1.96%, Mn 0.68%, P .18%, S 0.01%, O 26.91%, N 8.37%, and same before and after corrosion of the material indicates no chemical change was observed in the material.

The hardness test result of uncorroded specimen obtained was 41 HRB (Rockwell hardness under scale B) as shown in Figure 10. From the above result it is concluded that the specimen was in the range of low carbon steel (AISI 1008), which is ductile, malleable and soft. The corroded specimen was also tested and has 52 HRB, higher than that of uncorroded sample. This is because of the high temperature involved in the production line.



Figure1. Original sample collected from Mari gas oil field

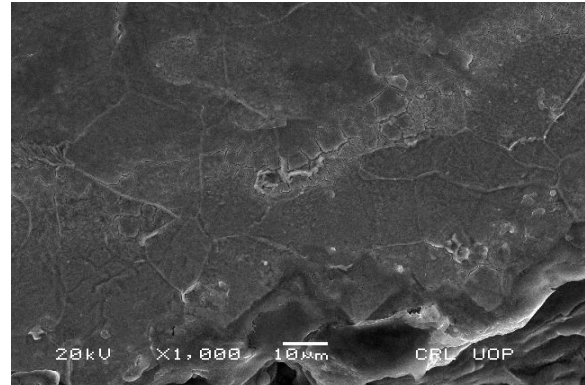


Figure 2a. Microstructure of low carbon steel in 8% nital solution

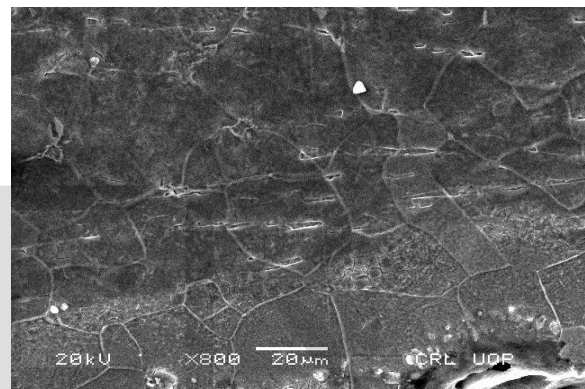


Figure 2b. Microstructure of low carbon steel in 3% nital solution

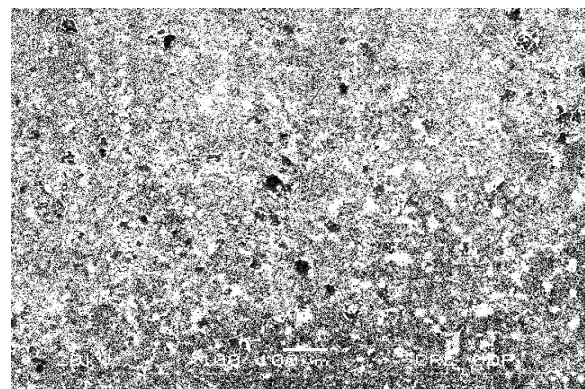


Figure 3. SEM analysis of samples before corrosion



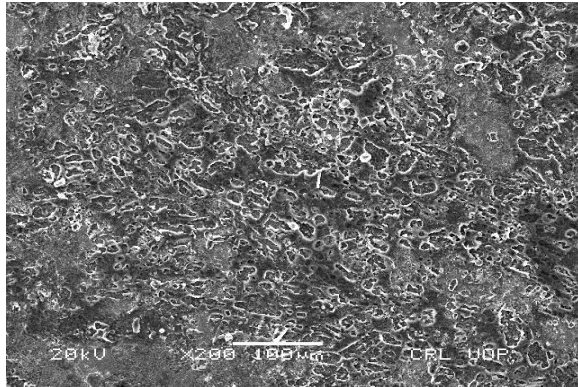


Figure 4. SEM analysis of samples after corrosion

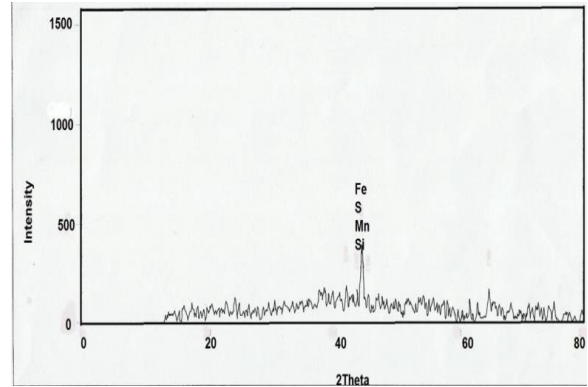


Figure 7. X-Ray analysis of the corrosion product

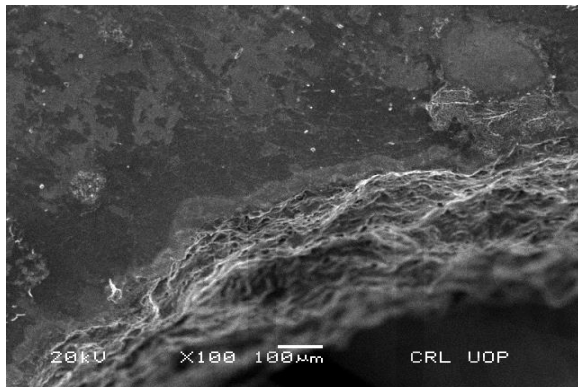


Figure 5. SEM analysis of FeS sites in of the materials

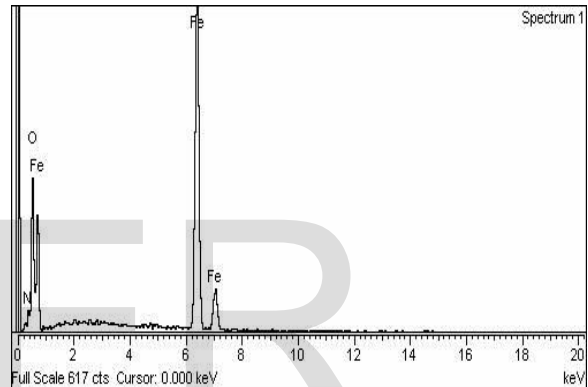


Figure 8. EDS analysis of the samples before corrosion

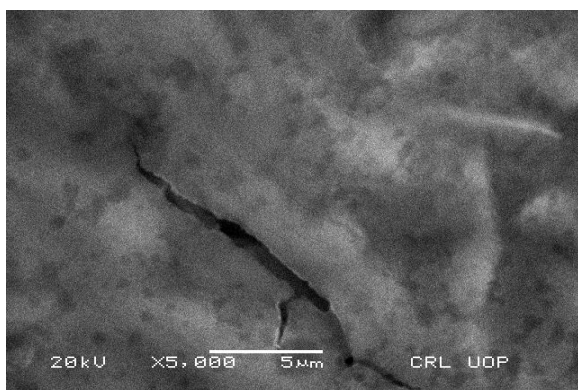


Figure 6. SEM analysis indicates the crack on grain boundaries

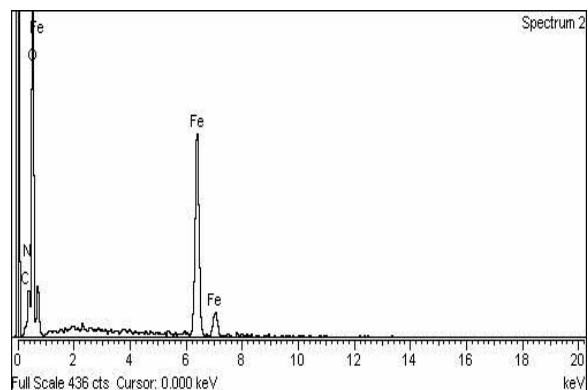


Figure 9. EDS analysis of the samples after corrosion

#### 4. CONCLUSION

- The aim of this project is to analyze the AISI-1008 low carbon steel and to study the effect of corrosion on the microstructure.
  - The results of microstructures, chemical composition, XRD and mechanical properties such as hardness were studied and it was concluded that all these were affected due to Erosion-corrosion.
  - The results obtained for both gasket specimens were observed and compared. There is a clear difference in the microstructure.
  - Care should be taken that the samples be well polished i.e. free from scratches before doing SEM analysis and should be preserved in a place free of moisture and contaminants.
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