

Study on the Combined Effect of Corrosive Environment and Impact Loading on the Behaviour of Low Carbon Steel

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Abstract: Investigations were carried out to study the corrosion behaviour, stress corrosion cracking (SCC) and hydrogen embrittlement (HE) of low carbon steels in acidic media by weight loss measurement and Impact test to fracture respectively. The effect on corrosion behavior of metal with different concentration of acidic media was monitored for a specified immersion period. The mechanism of stress-corrosion cracking and hydrogen embrittlement in low carbon steels was investigated using notched specimens, under high velocity loading conditions leading sudden fracture. It was observed that nitric acid environment was most corrosive to steels because of its oxidizing nature on the other hand the ammonium thiocyanate environment showed hydrogen embrittlement of the material. From the experimental results, it can be observed that material experiences the ductile-to-brittle transition with increasing in soaking period. On analysis, at room temperature, HNO₃ was found to be more aggressive towards mild steel compare to ammonium thiocyanate. Few studies have been carried out to determine the influence of hydrogen on structural steel and its mechanical characteristics, in turn mechanism of hydrogen embrittlement.

The result of impact test shows a decreasing trend indicating of reduction of material toughness. The material exhibit brittle failure in NH₄SCN treated specimen than the HNO₃ treated specimen. The surface analysis also carried out with the help of Scanning electron microscope (SEM). Overall the result suggests that corrosion was observed significantly HNO₃ medium. These deductions are due to higher carbon content in medium carbon steel coupled with various aggressive corrosion constituents contained in these media. Hydrogen embrittlement, as well as carbon cracking, is responsible for SCC of these materials in the acidic media.

Keywords: Corrosion, Stress corrosion cracking, Hydrogen Embrittlement, Low carbon steel, Nitric acid, Ammonium Thiocyanate.

I. INTRODUCTION

Corrosion is a natural impact of atmospheric environments like marine, industrial, urban and rural and affects the structural stability. Unfortunately, metals are subject to corrosion. Corrosion can take many forms; the form in which the interaction of corrosion and mechanical stress to produce a failure by cracking is known as stress corrosion cracking. Stress corrosion cracking (SCC) is a type of subcritical cracking of materials that occurs as a result of the combined and synergistic interactions among tensile stress, corrosive environment, and a susceptible material [1]. The annual loss due to corrosion can be compared with that of other natural calamities like earthquakes and cyclones, only its impact is indirect. "Loss due to corrosion will be up to 6 % if indirect cost of corrosion, in the form of loss of functioning of the plants, accidents, material discarded prematurely, and the various human liabilities are taken in to account" says Dr Baldev raj[2]. A series of mechanical and chemical operations in which acids play a very important role are acid pickling, industrial acid cleaning, cleaning of oil refinery equipment. In recent years it was found that at the ambient temperature and in

corrosive environment the carbon steels and low alloy steels suffered from SCC as well as HE, although the mechanical stress was lower than the yield strength [2,3]. This work examines the corrosion behaviour of mild steel when exposed to various concentrations of nitric acid and ammonium thiocyanate to investigate the stress corrosion behaviour and hydrogen embrittlement of low carbon steels. The purpose of this study is to study the influence on corrosion and after corrosion effect using impact test, SEM (Scanning Electron Microscope) fractography on specimens of the low carbon steel.

2. TEST MATERIAL AND EXPERIMENTAL METHOD

2.1. Test Material

Steel is an alloy of iron, carbon and several other elements. The industrial and operating environment leads to deterioration of metals under the action of air and moisture, steam and other gases, mineral acids and bases used in industries. The hydrogen in steel causes loss of ductility in a material, thus making it brittle and can be a cause for severe failures. Test specimens were prepared as per the

ASTM standards from a commercial material. Their chemical compositions as supplied by the manufacturer are shown in Table 1.

Table1. Chemical composition of the test specimen

	% Composition				
	C	Si	Mn	P	S
Composition %	0.448	0.291	0.88	0.026	0.020

2.2. Experimental method

The experiments were carried out to evaluate the corrosion behaviour of mild steel in various concentrations of nitric acid (HNO₃) and ammonium thiocyanate (NH₄SCN). The test specimens were exposed to acidic media for different immersion period. Impact testing techniques are used to evaluate the fracture behavior of materials under the influence of the two acidic media. After preparing the specimens, hydrogen charging was achieved by submerge the specimens in NH₄SCN solution 35% concentration at room temperature for different time durations[4,5]. The charpy impact test was performed by using the treated and untreated specimens. The samples were chemical treated at Chemistry laboratory, using lab equipment's.

2.2.1 Immersion test

The corrosion rate of low carbon steel was evaluated from immersion test. The equipment for the immersion test and its procedure are almost the same as those reported elsewhere [6, 7], and so they are mentioned briefly. Test specimens were prepared as per ASTM standards from commercial materials. The chemical composition is listed in Table 1. The specimens were ground and polished to a 320-grit finish on each surface and degreased with trichloroethylene.

The nitric acid solution for the immersion test was prepared by diluted with distilled water generated at the laboratory facility using distillation unit. The specimens weighed before and after immersion to the accuracy of +/- 0.5mg. The corrosion rate was calculated based on weight loss method. A typical experiment setup was shown in the figure1.The tests were conducted at room temperature.

Similarly the ammonium thiocyanate solution for the immersion test was prepared by diluting with distilled water to concentration of 35%w/vol at room temperature. A set of two specimens were exposed to chemical solution for 24hrs, 48hrs, 72 hrs and 96hrs duration and for every 48hrs cycle. After the specimen immersion in each batch, the specimens were weighed to evaluate corrosion rate.

The specific ratio of the solution volume to the specimen surface area was about 5ml per 100mm[8,9].

2.2.1. Impact test

Notched-bar impact test of metals provides information on failure mode under high velocity loading conditions leading sudden fracture where a sharp stress raiser (notch) is present. Charpy impact test is practical for the assessment of brittle fracture of metals. The aim of the present work was to access and characterizing of the level of degradation caused by corrosion. As per standard two specimens each were tested for charpy impact test. Impact test specimens were treated for 1 hr duration in 3M of HNO₃.



Fig 1. Typical experimental setup for corrosion rate measurement

For designing a system it is necessary to know the effect of hydrogen embrittlement on the mechanical properties especially fracture toughness at service conditions. The purpose of this study is to evaluate the susceptibility of structural steel to hydrogen embrittlement in charpy impact test. Two structural steel impact test specimens were prepared. After preparing the specimens, hydrogen charging was achieved by submerge the specimens in NH₄SCN solution 35% concentration at room temperature. The charpy impact test was performed by using the treated and untreated specimens.

3. RESULTS AND DISCUSSION

3.1. Corrosion Rate Measurement

The Low carbon steel reacts with nitric acid rigorously in nitric acid environment. The specimen immersed in 1M concentration of HNO₃ for 72hrs and cleaned after 48hrs cycle. The specimens were taken out after 72 hrs. and

cleaned as per standard procedure [5]. The specimens weighed before and after immersion to the accuracy of $\pm 0.5\text{mg}$. The corrosion rate was calculated based on weight loss method and it is found that 2.17 inches per year for 1M concentration of HNO_3 , which is in line with the earlier study with reference to low carbon steel [7]. A typical data of corrosion rate for 3M concentration HNO_3 is shown in fig.3.1. It is observed that the corrosion was rigorous at early hours of immersion and reduces as the time duration was increased and same may be witnessed from fig.3.1. This shows a resistance to corrosion and this corrosion resistance is due to the passivating action of nitric acid and the decrease in the strength of the acid as corrosion complexes get formed in the media [8,9]. On the other hand, the corrosion rate was increasing in trend and was constant after 48 hour of immersion. This was depicted in the fig.3.2. The corrosion rate of NH_4SCN was very less or negligible compare the HNO_3 .

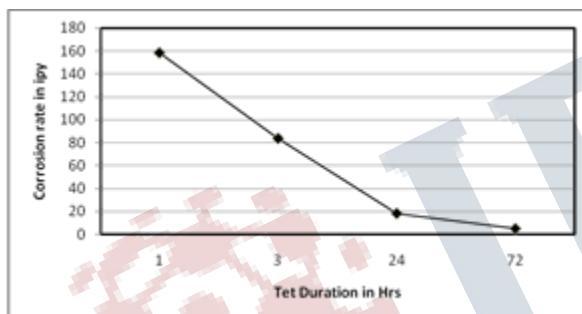


Fig 3.1 Corrosion rate in HNO_3

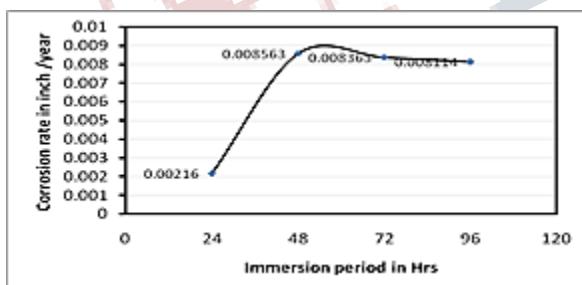


Fig 3.2 Corrosion rate in NH_4SCN

3.2. Charpy Impact behaviour of stress corroded and Hydrogen Embrittled Steel

Based on these results from the immersion test, the test duration was refined to 1hr for impact test specimens. The test specimens were masked except the notch area. Impact test specimens were treated for 1 hr duration in 3M of HNO_3 . The effect of corrosion can be observed as the energy absorbed for the treated specimens were decreasing i.e. impact resistance was reduced with the corrosion. As

expected, all of the other investigated steels show a loss of ductility and a decrease of impact resistance with increasing hydrogen content[9,10].

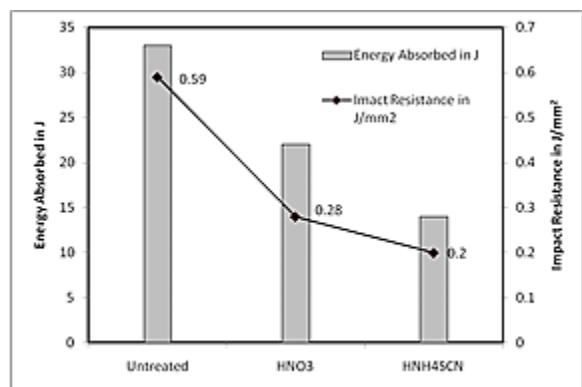


Fig 3.3 Variation of impact toughness of corroded specimens

From the broken steel specimen a white elliptical spot seen on the fracture as shown in fig.3.6. These are called as white spots and are generally thought caused by excessive hydrogen in the steel [11,12] which make a peculiarity from the fractured surface of HNO_3 treated specimens. The fracture surface showed two different regions of fracture behavior i.e. a brittle type of fracture had a dark, dull appearance proceeded by ductile fracture across the remaining cross section[13,14]. The dark grey shaded area at the extreme surface of the specimen indicates the shear lips.

Scanning electron microscope (SEM) fractographs of fractured surface of corroded impact samples are shown in Fig.3.7a-c and 3.8 a-b. The fracture surfaces specimens showed regions of ductile rupture, although frequently a heterogeneous distribution of micro void sizes was present, as shown in Figs 3.7 b-c. The corroded specimens showed a tendency towards larger and shallower micro voids.

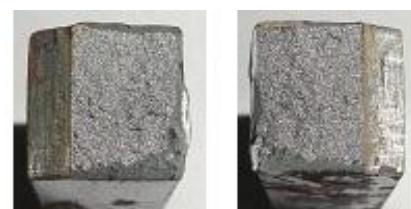


Fig. 3.4 Fractured surfaces of the impact specimen (Untreated)

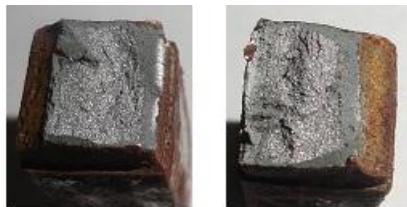


Fig. 3.5. Fracture surfaces of the impact specimen (HNO₃ treated)



Fig.3.6 Fractured surfaces of impact specimen (NH₄SCN treated)

A brittle fracture can show characteristics of transgranular or intergranular cracking when analysed through scanning electron microscope (SEM)[15,16,17]. From Fig 3.8b the fracture surfaces of impact specimens, micro void coalescence can be observed during ductile failure at the specimen, indicating more energy absorption. Fracture surfaces of samples revealed distinctive mixed mode of fracture indicating a ductile–brittle fracture transition. There are indication of cleavage facets and ductile tearing on the fracture surface. Based on careful analysis of fracture surfaces images of Fig.3.8 a and b, the distinctive fracture features, microvoid coalescence during ductile failure and transgranular cracking characterized by cleavage and river pattern were observed at the latter cases.

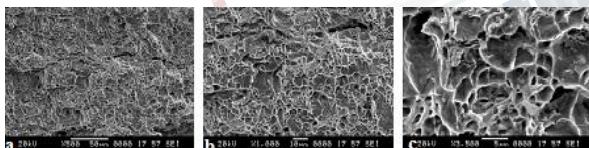


Figure 3.7. SEM images of fractured surface of impact specimen (HNO₃ treated)

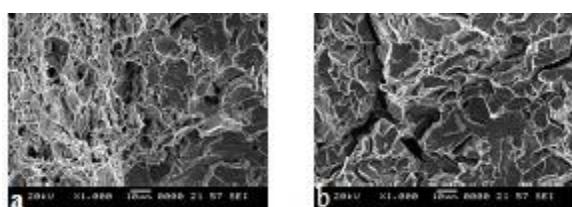


Fig3.8 SEM images of fractured surface of impact specimen (NH₄SCN treated)

From the broken steel specimen and fractographs of the distinctive fracture features revealed mixed mode of fracture indicating a ductile–brittle fracture transition in the thiocyanate treated specimen and transgranular cracking characterized by cleavage and river pattern were observed.

4. CONCLUSION

Based on the tests, the following conclusions can be drawn:

- The mild steel reacts rigorously in acidic media compare to weak acid.
- Impact strength of the material showed a decreasing trend and it is least for the NH4SCN immersion test which directs towards the loss of ductility.
- The fracture surface demonstrated a mixed mode of failure in the HNO₃ immersion test. The corroded specimens showed a tendency towards larger and shallower micro voids which exhibits the ductile mode of failure. Trans granular cracking can be witnessed which leads to the understanding of stress corrosion cracking.
- By dipping in a 35 percent NH4SCN solution, the test specimen loses its ductility. The experiments indicate that the specimens were embrittled by the hydrogen generated as a result of corrosion reaction with a solution. The hydrogen charging decreased the energy absorbed of the test material. Study of intergranular cracking related failures has shown that intergranular fractures can occur when the material is exposed to corrosive environment.

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