

## EFFECT OF HEAT TREATMENT ON THE DAMAGE RATIO OF CORRODED ST 60 MN STEEL

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### ABSTRACT

In this work, the effect of various heat treatments on ST 60Mn steel on its fatigue strength prior to use and after use was studied. It was observed that by normalising for service conditions not exceeding  $10^6$  stress cycles, the fatigue strength was raised by a factor of 1.2.

**Keywords:** crude oil, saline environment, fatigue strength, St 60 Mn steel, heat treatment.

### INTRODUCTION

Manganese steels are used in offshore oil pipelines and fittings where they are susceptible to corrosion fatigue-cracking of a metal under cyclic stress in the presence of corrosive environment. This is the case with ST 60 Mn, a medium carbon Nigerian standard specification, which is used for making oil pipelines. Oil spillage in Niger Delta area of Nigeria resulting from oil pipeline burstings is become an issue.

Corrosion fatigue of offshore structures resulting from interaction with crude oil is as a result of the presence of brine (Gough and Sopinith, 1946). According to Parkin and Kolotykn (1983), corrosion fatigue are induced in a transgranular pathway. Fontana (1986) in his investigation showed that fatigue are observed in corrosion fatigue which corresponds to the crack front being alternately stopped and started by stress cycling.

Some work has been done on corrosion fatigue behaviour of ST 60 Mn steel in many media (Odebisi 1991; and Olubusi; 1995) but no reported work on the combined effect of crude oil and saline environment on ST 60 Mn has been sighted. Thus the object of this work is to ascertain the form of heat treatment-quenching, annealing and normalizing that will enhance the fatigue strength of ST 60 Mn steel in a combined crude oil and saline environment.

### 2.0 EXPERIMENTAL PROCEDURE

#### 2.1 MATERIALS

St 60 Mn steel sample was obtained from Delta Steel Company, Aladja. Crude oil from shell oil company, warri; and sea water from bar beach, Victoria Island, Lagos (all in Nigeria). The chemical composition of the steel and the crude oil are summarized in Tables 3.1 & 3.2 respectively.

#### 2.2 METHOD

The steel sample was cut and machined to standard test pieces conforming to ASM standard. Then the specimens were categorized as follows for corrosion fatigue strength determination and heat treatment.

#### Fatigue Test

- (i) As received: No heat treatment was given. The specimens were clamped into a completely reversed bending fatigue machine with the aid of grips using an AVERT 7505 fatigue testing machine at the Department of Metallurgical & Materials Engineering Laboratory of the Obafemi Awolowo University, Ile-Ife, Nigeria. Cycle loading of varying stress level was applied until  $10^6$  cycles was reached. Bending moments of 90, 70, 50, 30, 10 and 5 Ncm which correspond to fatigue stresses of 54.34, 41, 48, 29.63, 17.78, 5.93 and 2.96 N/mm<sup>2</sup> respectively were chosen. The result is summarized in Table 3.3.

#### Heat Treatment and Corrosion Fatigue Test

- (ii) Specimens were heated to a temperature of 950°C soaked for 45 minutes and water-quenched at room temperature.
- (iii) Specimens were heated to a temperature of 950°C and allowed to cool in the furnace. (annealing).
- (iv) Normalising: Specimens were heated to a temperature of 950°C soaked for 45 minutes and allowed to cool in air.

The as-received in specimens (i) and the heat-treated specimens in (ii) – (iv) were placed in ocean water (20.3% salt content) for seventy two (72) hours after which they were transferred to crude oil for another seventy two (72) hours. The fatigue stresses were carried out on the set of specimens in (ii)-(iv) as in set (i). The results obtained are summarized in Tables 3.3, 3.4, 3.6, 3.7, 3.8 respectively. The corresponding damages are contained in Table 3.9. Figure 1 summarises the results of experiments (i)-(iv).

etching and microscopy. The etchant used were hydrochloric and picric acids for tempered samples and 2% nital for as-received, as received but corroded, quenched, annealed and normalized specimens. The resulting photomicrographs are contained in plates 1-5.

**Metallography**

Metallography was carried out on all the sets of specimens by cutting, mounting, grinding, polishing,

**3. RESULTS AND DISCUSSION**

**3.1 RESULTS**

The chemical composition of ST 60 Mn steel (As-received) is shown in Table 3.1.

**Table 3.1: Chemical composition of ST 60 Mn steel (As-received)**

Element	C	Si	S	P	Mn	Cr	Mo	W	V	As	Sn	Co	Fe
Wt.%	0.38	0.22	0.43	0.07	0.96	0.00	0.19	0.15	0.15	0.01	0.02	0.01	Balance

The manganese content is as high as 0.96% by weight.

**Table 3.2: Crude oil composition**

Element	Hydrocarbons Paraffins & aromatics	Oxygen Phenol and carbocyclic acid	Nitrogen compounds Phenols, indoles & carbonides	Sulphur	Trace metals
Wt.%	50-90%	2%	0.05-0.8%	5%	0.01-0.05%

**Table 3.3: Fatigue Test Results on Dry specimens (As-received)**

Fatigue Stress N/mm <sup>2</sup>	No. of cycles (N)	Log N
53.34	8.1 X 10 <sup>3</sup>	3.91
41.48	10 X 10 <sup>3</sup>	4.04
29.63	3.1 X 10 <sup>4</sup>	4.49
17.78	5.5 X 10 <sup>4</sup>	4.74
5.93	2.9 X 10 <sup>5</sup>	5.47
2.96	10 <sup>6</sup>	6



**Table 3.4: Fatigue Test Result in As-received specimen in combined crude and saline environment.**

Fatigue	No of cycles (N)	Log N
53.34	$1 \times 10^3$	3.00
41.48	$3.1 \times 10^3$	3.49
29.63	$1.2 \times 10^3$	4.08
17.78	$1.3 \times 10^3$	4.11
5.93	$2.97 \times 10^5$	5.48
2.96	$10^6$	6

**Table 3.5: Fatigue Test Data for water-quenched specimens in combined crude oil and saline environment,**

Fatigue	No of cycles (N)	Log N
53.34	$0.05 \times 10^3$	2.70
41.48	$1.5 \times 10^3$	3.17
29.63	$3.4 \times 10^3$	3.53
17.78	$2.4 \times 10^3$	4.38
5.93	$4.8 \times 10^5$	4.68
2.96	$1.55 \times 10^5$	5.19

**Table 3.6: Fatigue Test on Annealed specimen in combined crude oil and saline environment.**

Fatigue stress N/mm <sup>2</sup>	No. of cycles (N)	Log N
53.54	$0.5 \times 10^3$	2.70
41.48	$5.5 \times 10^3$	3.74
29.63	$2.6 \times 10^3$	3.41
17.78	$8.0 \times 10^4$	3.90
5.93	$1.4 \times 10^5$	9.97
2.96	$10^6$	6.00

**Table 3.7: Fatigue Test on Normalised specimen in combined crude oil and saline environment.**

Fatigue stress N/mm <sup>2</sup>	No. of cycles (N)	Log N
53.54	0.5 X 10 <sup>3</sup>	2.70
41.48	5.5 X 10 <sup>3</sup>	3.74
29.63	2.6 X 10 <sup>3</sup>	3.41
17.78	8.0 X 10 <sup>4</sup>	3.90
5.93	1.4 X 10 <sup>5</sup>	5.13
2.96	10 <sup>6</sup>	6

**3.8: Summary of Corrosion Fatigue strength and damage ratio for ST 60 Mn.**

Samples in combined crude oil and saline Environment	Corrosion Fatigue strength N/mm <sup>2</sup>	* Damage ratio
As-received	7.0	0.5
Water quenched	2.9	0.2
Normalised	8.5	0.6
Annealed	5.9	0.4

Damage ratio for ST 60 Mn at 10<sup>6</sup> stress cycle

$$\text{Damage ratio} = \frac{\text{corrosion fatigue strength}}{\text{Air fatigue strength}}$$

$$\text{Air fatigue strength} = 14.5 \text{ Nmm}^{-2}.$$

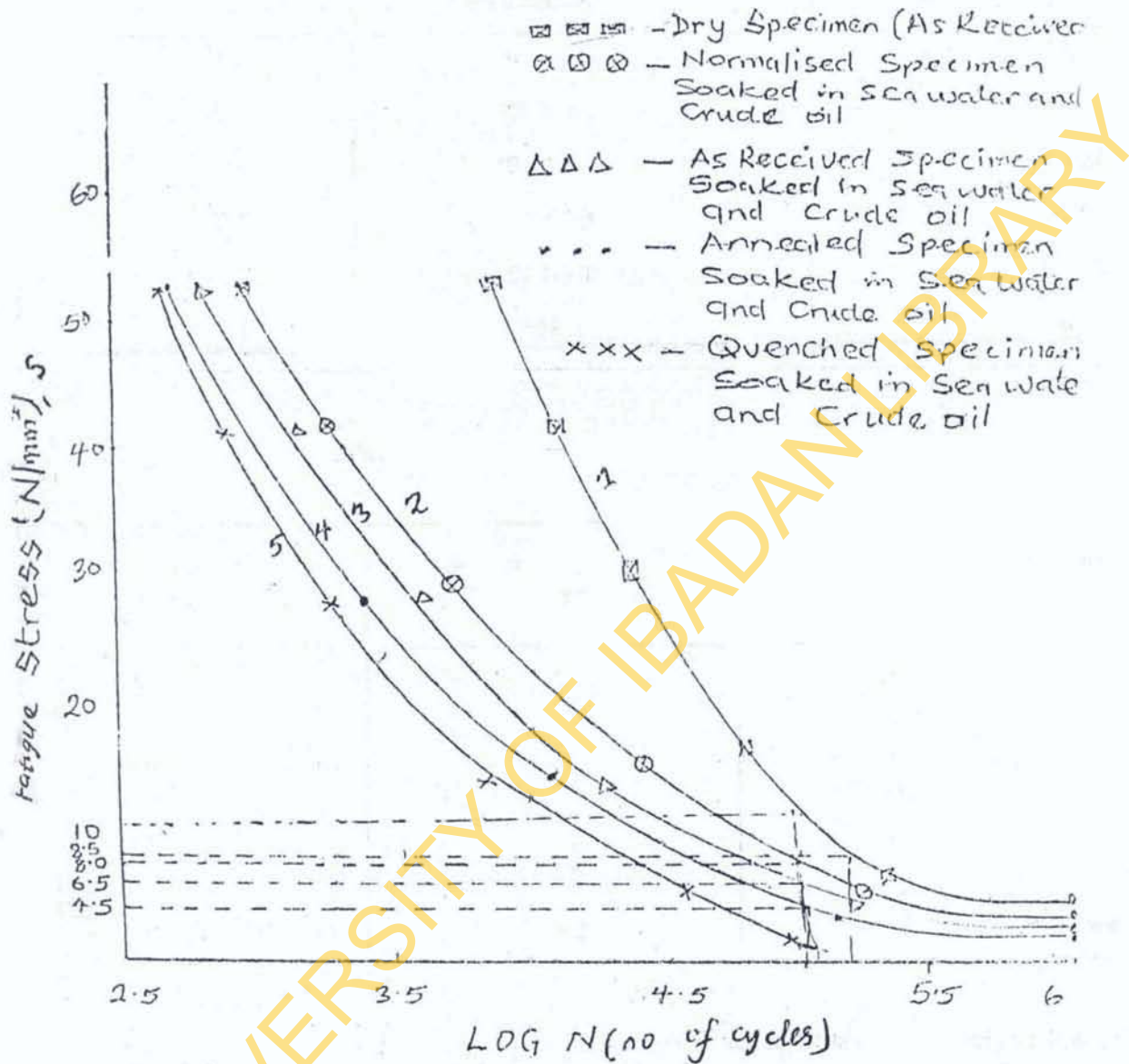
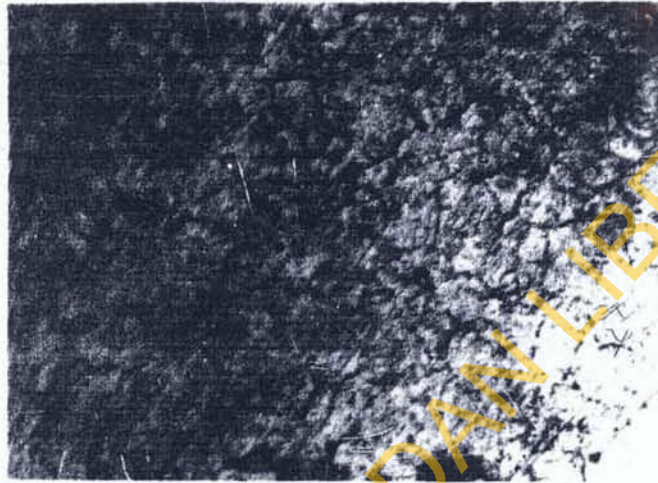


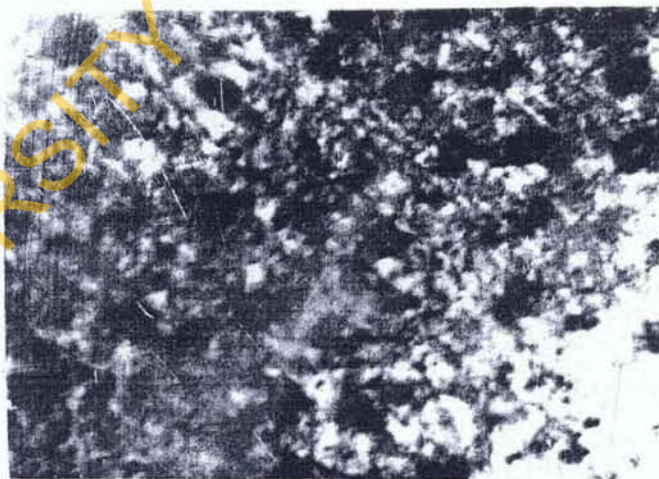
Fig. 1 Fatigue Strength Curves showing the Relationship between Dry Specimen and those (Normalised, As-Received, Annealed and quenched) Soaked in Salty Water (Sea Water) and Crude Oil.



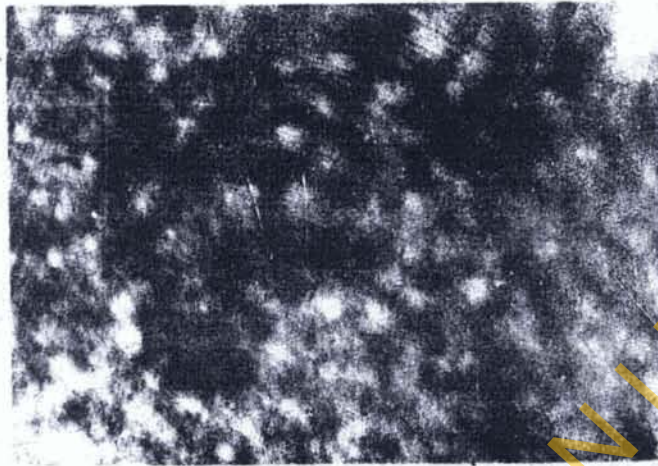
**MICROSTRUCTURE OF ST 60 Mn STEEL SPECIMENS**



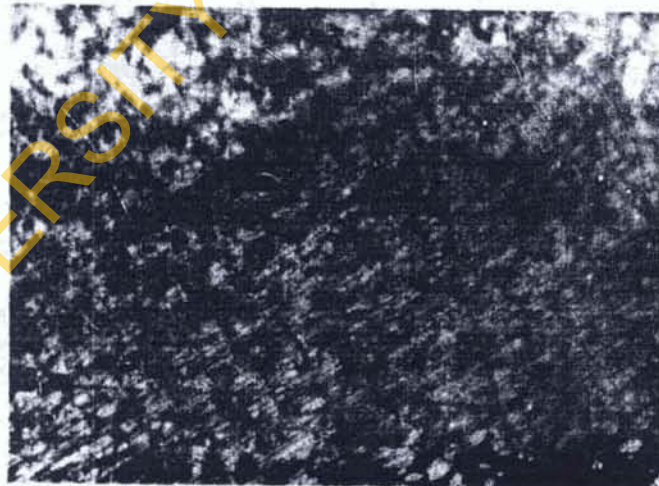
**PLATE 1**  
As-Received condition showing structure of ferrite (light) and pearlite (dark).



**PLATE 2**  
As-Received but corroded condition showing structure of ferrite and pearlite with dark patches indicating corrosion effect (pitting).



**PLATE 3**  
Water-quenched from 850°C showing martenitic structure.



**PLATE 4**  
Annealed at 850°C showing a coarse ferrite grains.

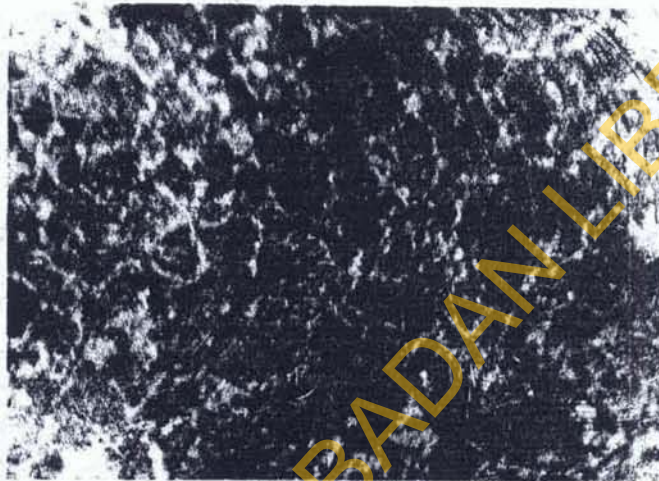


PLATE 5  
Normalised at 850°C showing a finer ferrite-pearlite structure.

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## DISCUSSION

The as-received ST 60 Mn steel is observed from Figure 1 (curve 1) to have the highest fatigue strength. The microstructure shown in plate 1 reveals pearlite as the major phase (dark) in the matrix of ferrite (light). The as-received but corroded sample as depicted in Figure 1 (curve 3) has a low damage ratio, of 0.5 (Table 3.8). This is not surprising as the microstructure shown in plate 2 reveals pearlite phase in ferrite matrix in almost equal proportion with dark patches showing corrosion effect (pitting).

Now when the medium carbon steel is water-quenched, its behaviour is described in Figure 1 (curve 5). The specimen has the lowest fatigue strength and a corresponding damage ratio of 0.2. The microstructure (plate 3) reveals that there has been phase transformation to martensite which is hard and brittle. The low fatigue stress could be attributed to the deleterious effect of sulphide and chloride ions and the internally stressed and hard martensite structure which lead to formation of hardened cracks.

From figure 1 (curve 4) the fatigue strength of the annealed ST 60 Mn steel prior to usage is higher than water-quenched specimen but lower than as-received (as-rolled) sample.

This is discernible from the microstructure shown in plate 4 wherein the coarse-grained pearlite structure is well-distributed thereby allowing easy penetration of sulphide from oil and chloride from saline environment which leads to formation of pits on the materials and with time the pits may be propagated by fatigue stress.

From figure 1 (curve 2) it is observed that normalizing heat-treatment can be used to increase the corrosion fatigue strength of ST 60 Mn steel. This could be attributed to fairly homogenized finer pearlite grains within the ferrite matrix (see plate 5) thus making it difficult for sulphide ions from crude oil and chloride ions (from saline environment) to penetrate the matrix. This corroborates the result of the investigation carried out by Olaosebikan (1991) where the normalized ST 60 Mn steel has enhanced corrosion fatigue strength in a cyanide environment.

## CONCLUSION

From this investigation it can be recommended that manganese steels like ST 60Mn used for oil pipelines should be heat-treated by normalizing prior to use in order to enhance the fatigue strength of this grade of steel and thus prevent oil spillage.

## ACKNOWLEDGEMENT

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