



## Original Research Article

### Effects of Tempering and Normalizing Heat Treatments on Stress Corrosion Cracking Behaviors of Modified AISI 4340 (300M) Steel in Acidic Chloride Media

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#### ABSTRACT

*Stress corrosion cracking (SCC) is an unpredictable calamitous and highly detested type of corrosion. 300M steel is a special but notable failure-prone structural material by SCC. The aim of this study was to find out SCC mitigation extents of 300M steel by tempering and normalizing heat treatments in seven aqueous media that contained different concentrations of hydrochloric acid (HCl) and sodium chloride (NaCl) up to 17.5%. 56 ASTM E8 standard tensile test samples were produced from procured un-heat-treated 300M steel out of which 14 were heat-treated by tempering and 14 by normalizing. The samples were procedurally cleaned to uniformly smooth surface finishes and each loaded to the same maximum tensile stress of 1427.4 MPa. The loaded samples were immersed in pairs of one tempered with one un-heat-treated and one normalized with one un-heat-treated in each of the seven prepared media under separate constant temperatures of 60 °C and 100 °C for one hour, and removed for integrity assessment with respect to internal and surface cracks by micro-examination. Results obtained showed no SCC for all the heat-treated samples but multiple trans-granular crack features alongside craters that increased in intensity with the acid and chloride contents and propagated in the matrix structure to the surfaces for the un-heat-treated samples tested at 100 °C in media that contained from 14% HCl and 14% NaCl.*

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## 1. INTRODUCTION

Corrosion is an unavoidable material degradation process that stems from the material's natural interplays with its environment (Fontana, 1987). Corrosion results in gradual cumulative wastage of dimensional sizes and mass, un-cherished appearance, and reductions in mechanical and physical properties such as strength, hardness, thermal and electrical conductivities of a material. The level to which a material corrodes depends principally on its metallurgy, surface conditions, chemical make and corrosivity extent of its environment

(Guma and Durami, 2019). Problems of corrosion in engineering use of materials directly or indirectly affect everybody to various levels in terms of safety, technological and economic costs depending on the corrosion rates and the control methods used (Guma *et al.*, 2017). Different forms of corrosion are encountered in engineering use of steel but one highly repugnant type is known as stress corrosion cracking (SCC). SCC occurs by formation and growth of cracks on a metallic material in a corrosive environment often at temperatures well above ambient values. It can result in sudden and difficult-to-predict severe degradation mode of structural failure. It has been responsible for several serious structural failures or accidents in our technological history of which in some cases it may not be known and reported as the causative factor (Xiaoyuan, 2010; Guma *et al.*, 2014).

There are countless types of micro-environments which are not equally corrosive to materials. Some environments are far more corrosive than others. Also, not all materials have the same level of corrosion resistance in the same environment as some can resist at far less levels than others (Uhlig, 1971; Guma and Durami, 2019). The consequences of corrosion are many and varied and the effects of these on the safe, reliable and efficient operation of equipment or structures are often more serious than the simple loss of mass of metal. Failures of various kinds and the need for expensive replacements can occur even though the amount of material destroyed is quite small (Goel *et al.*, 2010; Shymma, 2014).

Out of all corrosive environments, acids and/or chloride salts are outstandingly corrosive to steel. That is why most standardized short-time corrosion tests and other laboratory corrosion tests of steels are based on using media that contain higher levels of acids and/or chlorides than in natural or artificial environments such as water, soil, atmosphere, and some industrial solutions. Such tests are generally much more economical and easier to conduct than field and service tests which require long-time exposure of materials in terms of years in the natural or service environments before meaningful corrosion evaluation of the materials can be achieved. The accelerated or short-time tests are used to screen steels and other materials for corrosion resistance based on the understanding that if a material judgmentally performs better than another material in such media, then it is indicative of better corrosion resistance than the other material in natural or artificial environments (Money and Kain, 1988; Guma and Daniel, 2019). Although acids are generally known to be more corrosive to steel than chlorides, chlorides are also great agents of steel corrosion with capability to promote corrosion current and attack and breakdown passivating protective barriers that do naturally form on the surface of the alloy (Kar, 2010; Ulaeto *et al.*, 2012; Guma and Daniel, 2019). Chlorides abound in some natural environments such as surf beaches, sea water, seafront atmospheres, and industrial polluted atmospheric environments. Chlorides can also exist inadvertently alongside acids in industrial solutions as a result of partial reactions of acids with alkalis. They can therefore synergize with acids to aggravate corrosion. Acidic chloride environments abound naturally in the atmosphere, soils and sea environments of many maritime tropical countries like Nigeria (Ramananda *et al.*, 2011; Matjaz and Jackson, 2014; Guma *et al.*, 2014; Guma and Daniel, 2019).

One method of controlling corrosion of steel is through heat treatment. Heat treatment is the process of favorably altering the microstructures, physical and mechanical properties and corrosion resistances of metals for better engineering applications through controlled heating and cooling without changing material size. Heat treatment has comparative advantage in terms of inbuilt control permanence in the material compared to other common methods of corrosion control such as inhibition, cathodic protection, and coating methods which need to be renewed from time to time for their continued effectiveness (Youngblood and Raghavan, 1978; Cotterel, 1985; Rajan *et al.*, 1997; Ayodele and Nenuwa, 2013; Guma *et al.*, 2019). Although heat treatment is a costly method of mass-controlling corrosion due to cost of energy required for mass-heating, and cooling and handling of parts some which may be awkward and heavy, it can be used in special or customized needs such as innovative improvement of some work parts or components. It can also be used for inhibiting corrosion of structural parts where accessibility is difficult for inspecting and maintaining them during their service lives and those that can wear by rubbing contact (Guma *et al.*, 2019).

300M steel is a low-alloy vacuum melted steel often used in applications where high strength, ductility and fracture toughness are required. Essentially it is a modified AISI 4340 steel with silicon, vanadium and slightly greater carbon and molybdenum content than the AISI 4340 steel. 300M steel is used in applications that require strength in the 2000-2070MPa range and fracture toughness such as aircraft landing gear, high strength bolts, and airframe parts (Hornbach and Prevey, 2007). 300M steel is however very susceptible to SCC and corrosion fatigue which can lead to devastating catastrophic consequences especially for aircraft landing gear (Youngblood and Raghavan, 1978; Hornbach and Prevey, 2007). Because of the significance and corrosion behavior of the steel, a number of researches such as those by Ryder and Pickel (1978), Padmanabhan and Wood (1985), Kerr et al (1987); Awasthi (2012) have been conducted in the last few decades to improve its corrosion resistance with respect to SCC susceptibility in a number of environments or find alternative better materials for it in its areas of application but the utopia is yet to be reached.

The aim in this paper is to present conducted laboratory SCC tests of as-received (un-heat-treated), tempered and normalized samples of 300M steel in various aqueous media that contained different concentrations of both hydrochloric acid (HCl) and sodium chloride (NaCl) in equal measures up to 17.5% under separate constant temperatures of 60 oC and 100 oC and tensile stress of 90% of the yield stress of the steel.

## **2. MATERIALS AND METHODS**

### **2.1. Materials**

The materials used for the work were un-heat-treated modified AISI 4340 (300M) steel which was bought in the form of seven rods of about 1000 mm length and 18 mm-diameter from a commercial dealer (Hartzog limited) in Kaduna metropolis in Nigeria; concentrated hydrochloric acid, purchased from Emma Continental Enterprise Kaduna metropolis; unadulterated sodium chloride also purchased from Emma Continental Enterprise; and red oil. The equipment employed for the research were a Vecstan electric furnace model E0F2 at the Department of Mechanical Engineering, Nigerian Defence Academy (NDA), Kaduna; procured plastic cylindrical container of about 500 mm internal diameter and 400 mm depth for preparing the test acidic chloride media; Anton Paar Abrasion machine with Dino-lite digital microscope No 20~220\* AM7391SMZT at Liquefied Natural Gas laboratory of Ahmadu Bello University Zaria, in Nigeria; procured Amazon-made magnifying eye glass of optical magnification 20; Amen Optical Emission Spectrometer of Model No PDA7000 at Defence Industries Corporation of Nigeria (DICON), Kaduna; a digital weighing scale; electric ring boiler; emery abrasive cloth of various grades for cleaning the steel samples; dead weight loads in kilogrammes and grammes for stressing the samples which were available at the Department of Mechanical Engineering workshop, NDA; and a mercury-in-glass thermometer with a scale range of up to 150 °C for monitoring the temperatures of the prepared acidic chloride media.

### **2.2. Methods**

#### **2.2.1. Analysis of steel samples**

Analysis of nominal composition of the procured 300M steel rods was conducted using the Amen optical emission spectrometer (OES) to ascertain and accept them as 300M steel or discard them. This was done by cross-checking the analyzed compositions of the rods with the acceptable composition for the 300M steel type according to the UNS and AISI specifications for the steel. A piece of about 20 mm length was cut at both ends of each of the seven procured 300M steel rods and separately analyzed in accordance to the spectrometer's manual. The obtained elemental compositions of the pair pieces from each rod were averaged by elemental types and recorded. Any rod as per its averaged elemental compositions that was found to deviate from the UNS, and AISI nominal specification for the 300M steel by at least  $\pm 5\%$  for even one element or contained a significant amount of an element that is not included in the UNS and AISI

specifications for 300M steel was not considered. In that way three rods were confirmed as correct 300M steel and the rest were discarded.

### 2.2.2 Production of samples

The ascertained 300M steel rods were used produce (using a lathe) 56 samples of dimensional specification according to the ASTM E-8 standard (Ajibola et al., 2012) shown in Figure 1.

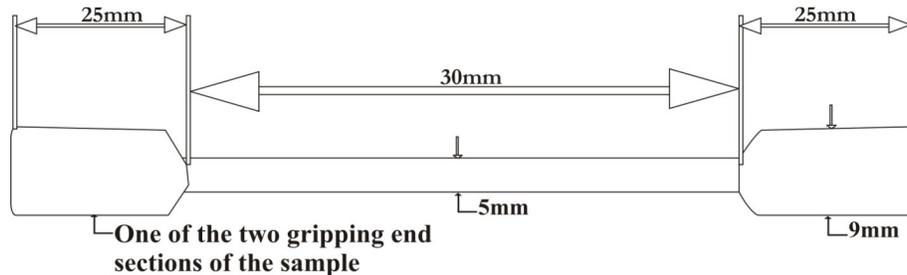


Figure 1: Dimensional specifications of the ASTM E-8 standard sample

A smooth file was used to scrub out machining burrs on the samples and smoothen their cut ends. One gripping end section of each sample was similarly externally threaded to facilitate fixing the end sections interchangeably in similarly threaded opposite holes produced on a mild steel support bar for holding two samples as cantilevers for bending them to induce the required tensile stress in the samples for the SCC test. A 4 mm-diameter hole was also drilled on the opposite gripping end section of each sample with its edge 1 mm from the edge of the section to facilitate hanging the required load with a steel wire of diameter 4 mm to induce the required bending tensile stress in each of the two cantilevered samples as shown in Figure 2.

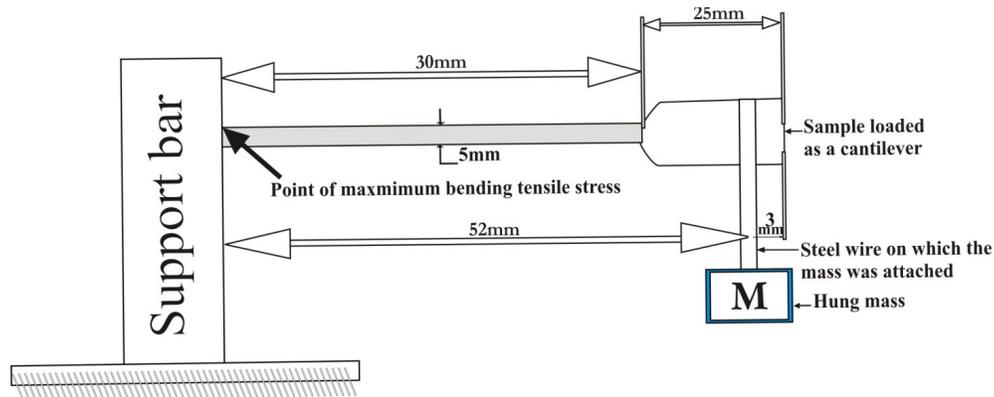


Figure 2: Principle of loading sample in tension by cantilevering

### 2.2.3. Production of sample support bar

A solid mild steel support bar of length 100 mm and diameter of 80 mm was produced by mechanically sawing it out from a procured parent mild steel bar. Two holes each of depth 25 mm and diameter 9 mm in accordance with the diameter specification of the gripping end sections of the ASTM E-8 standard sample (Ajibola et al., 2012) shown in Figure 1 were drilled on opposite sides of the bar diameter at 10 mm from the top end of the bar. The holes were threaded with the same thread sizes used to thread the gripping end sections of the samples to facilitate screw-fixing the samples on the bar. The other end of the mild steel bar was arc-welded onto a procured and prepared square mild plate of 180 mm by 180 mm by 2 mm thickness

at the middle of the plate to stabilize the bar's stand in the vertical position when loaded with samples as shown in Plate I. In this way, the support bar was made to hold two samples loaded on it as cantilevers for inducing the required maximum bending tensile stress in them for the SCC test.



Plate I: Principle of cantilevering and stressing samples on the support bar for the SCC test

#### 2.2.4. Heat treatment of the produced 300M steel samples

Out of the 56 produced samples, 28 were not heat-treated and 28 heated treated by heating them to a temperature of 900 °C and holding them there for 120 minutes in a Vecstan furnace which had a maximum heating temperature of 1100 °C. Out of the 28 heated samples, 14 samples were taken out of the furnace and normalized by allowing them to cool in still air while the other 14 samples were quenched into oil at room temperature. After quenching, the samples were tempered by heating them to a temperature of 500°C for two hours. Plates II, III, and IV show samples in the furnace and as were being normalized by cooling in the air, and quenched for tempering respectively.



Plate II: Samples in the furnace



Plate III: Samples as were being cooled in still air for normalizing



Plate IV: Samples as were being quenched in oil during the tempering process

### 2.2.5. Micro-structural analyses of the produced samples

In order to ensure comparability of test results among the samples, both the heat-treated and un-heat-treated 300M steel samples were procedurally cleaned manually to smooth uniform shiny surfaces with abrasive emery cloths of grits 60, 80, 220 and 320 in that order. The samples were thereto wiped with lint-free towel, cleaned with acetone, rinsed in distilled water, and dried in air for one hour; all in accordance to ASTM G1 standard method for cleaning metallic materials for corrosion tests (Guma and Daniel, 2019).



Plate V: Some of the polished heat-treated and un-heat-treated samples



Plate VI: Piles of semi-polished heat-treated and un-heat-treated samples

The cleaned samples were handled with clean gloved hands and stored in moisture-free desiccators prior to use. Plate V shows a pile of some of the produced samples after cleaning them, while Plate VI shows appearances of some of the semi-cleaned heat-treated and un-heat-treated samples. Micro-structures of the as-received un-heat-treated, normalized and tempered samples were then analyzed using Anton Paar Abrasion machine with Dino-lite digital microscope at the Liquefied Natural Gas laboratory of the Ahmadu Bello University Zaria in Nigeria after which micrographs of each sample types taken.

### 2.2.6. Determination of the tensile stress

The same tensile stress was applied on each sample and was worked out from 90% of the yield strength of 300M steel (1586MPa) given by Kuehmann *et al* (2008) to be 1427.4MPa in accordance with the ASTM G36-99 procedure (Guma *et al.*, 2014).

#### 2.2.6.1. Determination of the load to be applied on the cantilevered samples

The tensile bending stress on the sample was determined from (Ryder, 2003).

$$\sigma = \frac{32mgl}{\pi d^3} \quad (1)$$

Where m = mass of the hung load at the free end of the cantilevered sample, g = acceleration due to gravity, l = 52 mm = distance of the applied load from the fixed end of the cantilevered specimen, d = diameter of the gauge length of the cantilevered sample = 5 mm,  $\sigma = 1427.4$  MPa was the intended stress to be applied to each sample under test. Using Equation (1), m was evaluated to be 34.36 kg.

### 2.2.7. Preparation of the acidic chloride media

Seven aqueous acidic chloride media (Nos. 1-7) were prepared using pure water, the procured hydrochloric acid and sodium chloride. The percentage compositions of pure water, acid and chloride in the corrosive media are as shown in Table 1. Water (48 kg) was used to work out the mass percentages of hydrochloric acid and sodium chloride to be admixed in the container. In each case, the masses were determined using a weighing scale. Also, in each case water was first poured into the container, then the required hydrochloric acid was added atop and thoroughly stirred with a glass rod and lastly the sodium chloride was added and also stirred to mix up the whole content.

Table 1: Percentage compositions of pure water, hydrochloric acid, and sodium chloride by mass

Medium No	Water (%)	Sodium chloride (%)	Hydrochloric acid (%)
1	100	0	0
2	97	1.5	1.5
3	93	3.5	3.5
4	86	7.0	7.0
5	77	11.5	11.5
6	72	14.0	14.0
7	65	17.5	17.5

### 2.2.8. SCC test of the prepared samples

The support bar was loaded with two samples. One heat treated sample by normalizing was fixed on the bar by screwing its threaded end into one of the produced holes on the bar, and one un-heat-treated (control)

sample similarly fixed on the bar in the other opposite hole on it. Different standard steel dead weights of total mass 34.36 kg were hung together at the free end of each of the two samples. One end of a steel wire of diameter 4 mm and length 50 mm whose other end had previously been centrally used to knot the 34.36 kg-mass together was used to hang the mass on sample by inserting the end into the 4 mm-made hole on the sample and knotting the wire. After loading the samples on the support bar as cantilevers, the bar was immersed in vertical position with its assembly in medium No. 1 which had been prepared. The medium was then heated electrically to 60 °C and thermostatically maintained at that temperature for one hour using the electric ring boiler. The temperature was monitored by the mercury-in-glass thermometer. Plate VII shows the top view of the medium in the container as it was being heated during the test. During the test duration a pair of the Amazon-made magnifying glasses was used to see through the bowl content to monitor any observable sample failure or cracks on them. At the end of the test duration the sample support bar was removed and the samples dismantled from it for detailed micro-examination for cracks. This procedure was repeated using one tempered heat-treated sample and one un-heat-treated (control) sample. These were repeated using media No 2, No 3, No 4, No 5, No 6, and No 7 in that order.



Plate VII: Top view of the medium content as it was being heated during the test

The foregoing procedure was repeated with each medium heated to a constant temperature of 100 °C using the remaining 28 samples and similar pairing.

### 2.2.9 Micro-structural examination

Although the tested samples were visually checked for cracks alongside the Amazon magnifying glasses of magnification no crack was detected on any of them by the methods. Each sample was further checked for cracks by micro-examination using Anton Paar Abrasion machine with Dino-lite digital microscope after which micrographs of all cracked samples were taken.

## 3. RESULTS AND DISCUSSION

### 3.1. Chemical Composition

Result of the analyzed chemical composition of the 300M steel used for the study is shown in Table 2. Results from Table 2 were compared with AISI specification of 93.4-94.8% Fe, 1.65-2.0% Ni, 1.45-1.80% Si, 0.70 -0.95% Cr, 0.65-0.90% Mn, 0.40- 0.46% C, 0.035% P, 0.30-0.45% Mo, 0.050% V, and 0.040% S for 300M steels (UNS K44220) as well as and , ASTM A646 (300M-8) specification (Carvalho and Lima, 2012; Arindam et al., 2016). From this, it was evident that the steel used for the tests met the UNS and ASTM specifications for 300M steel as can be observed.

Table 2: Analyzed chemical composition by elemental (Wt) of the 300M steel used for the study

Element	Fe	C	Cr	Mn	S	V	Si	Mo	Ni	P
Wt (%)	94.516	0.4	0.73	0.6	0.08	0.06	1.5	0.4	1.68	0.031

### 3.2. Micro-photographic Analyses of Tested Samples for SCC

Results of micro-structural analyses of the 300M steel which were found to be distinct from the others are shown in Plates VIII to XII. It can be observed that the normalizing heat treatment refined and homogenized the microstructure and shortened the inter-grain boundaries of the as-received un-heat-treated 300M steel. The matrix structure of the as-received steel also changed from more or less martensitic-cementite-austenitic type to seemingly ferritic-pearlitic type due to normalizing process as can be observed from the two plates. The tempering processes also altered the microstructure of the steel but to more or less martensitic-cementite-lath type of more uniform composition with no clear identifiable inter-grain boundaries as can be observed from Plates VIII and X. From Plates XI and XII, it is evident that corrosivities of media No. 6 and No. 7 at 100°C disfigured the microstructures of the as-received un-heat-treated samples with various sizes of cracks of trans-granular and to some extent random orientations alongside with more or less craters.

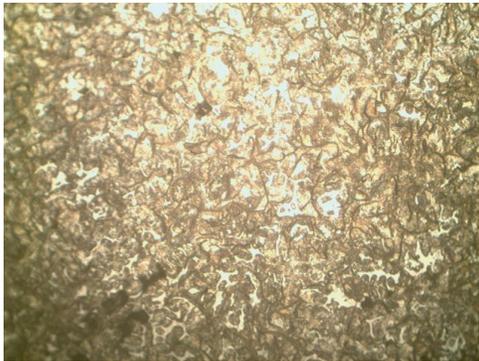


Plate VIII: Microstructure of the as-received 300M steel (250  $\mu\text{m}$ )

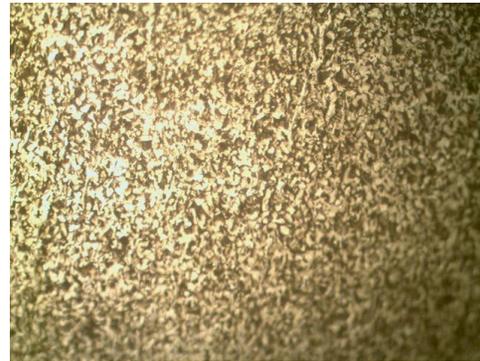


Plate IX: Microstructure of the normalized 300M steel (250  $\mu\text{m}$ )

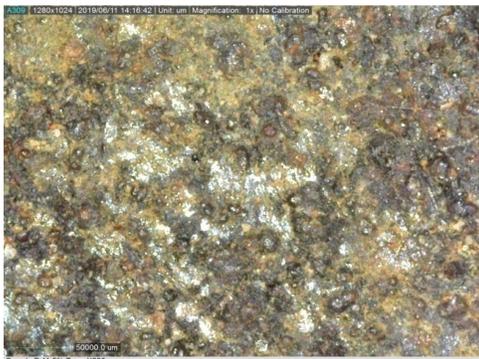


Plate X: Microstructure of the tempered steel samples (250  $\mu\text{m}$ )

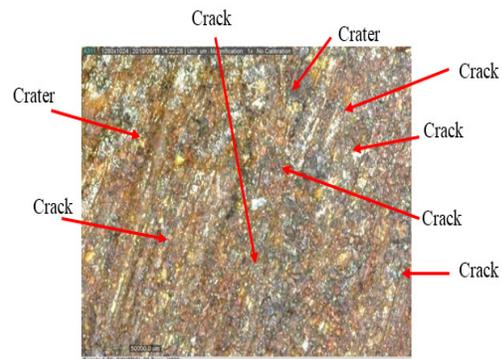


Plate XI: Microstructure of the control steel samples that cracked in No 6 medium which contained 14% HCl and 14% NaCl (250  $\mu\text{m}$ )

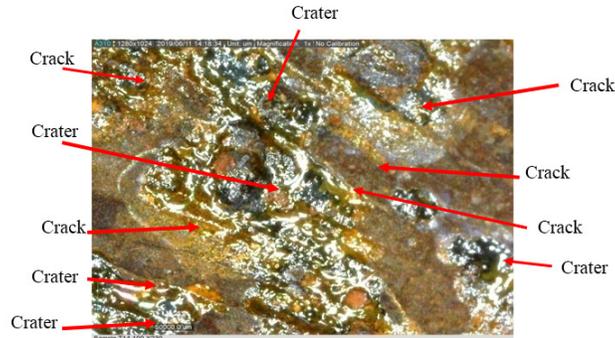


Plate XII: Microstructure of the as-received steel samples that cracked in No 7 medium which contained 17.5% HCl and 17.5% NaCl (250  $\mu\text{m}$ )

### 3.3 SCC Corrosion Test Assessment

The detailed results from micro-examinations of the tested samples are presented in Tables 3 and 4. From Table 4, it is evident that all the heat-treated and un-heat-treated (control) samples of the test 300M steel survived SCC in all the seven acidic chloride test media at 60 °C. One reason that was attributed to this is the low test temperature of 60 °C and exposure time of only one hour even though the applied stress value of 1427.41 MPa on the samples was high and the corrosivity levels of some test media such as media Nos. 5, 6, and 7 were adjudged to be very high. The test stress value and corrosivity levels of the media are much higher compared to what are practically obtainable in service stress corrosion cracking where much less stress loads and corrosivity levels can cause SCC of susceptible steels after several years of exposure to some natural environments within temperature range of less than 10 °C to 60 °C at different locations around the globe due to fatigue (Mildrexler et al., 2006; Gulbrandsen, 2012; Guma et al, 2014; Scambos et al, 2018). However when the test temperature of the acidic chloride media was raised to 100 °C, all the un-heat-treated (control) steel samples experienced SCC in medium No. 6 which contained 72% water, 14% hydrochloric acid, and 14% sodium chloride; and medium No. 7 which contained 65% water, 17.5% hydrochloric acid, and 17.5% sodium chloride as can be seen from Plates XI and XII as well as Table 4. The degree of SCC increased from the steel samples tested in medium No. 6 to those tested in medium No. 7 by multiple and greater trans-granular cracks and craters that propagated in the matrix structure to the surfaces of the steel as can be observed from Plates XI and XII. Although most of the cracks were trans-granular few were random and not clearly visible especially for Plate XI as can be observed. All the normalized and tempered samples of the steel however survived SCC in all the media at the media's temperature of 100 °C as can also be observed from Table 4. The control samples of the steel however survived SCC in media Nos. 1 to 5 which were less corrosive than medium Nos. 6 and 7 as can be seen from Table 4. From the foregoing it can be appreciated that the normalizing and annealing heat treatments of the steel would have alleviating effects on SCC behavior of the steel in acidic chloride media as by indications of results from the accelerated corrosion tests with hydrochloric acid-sodium chloride media.

Table 3: SCC assessment of heat-treated and un-heat-treated (control) samples of 300M steel after one-hour test exposure at 60 °C

Medium No.	Normalized	Control	Tempered	Control
1	NCD	NCD	NCD	NCD
2	NCD	NCD	NCD	NCD
3	NCD	NCD	NCD	NCD
4	NCD	NCD	NCD	NCD
5	NCD	NCD	NCD	NCD
6	NCD	NCD	NCD	NCD
7	NCD	NCD	NCD	NCD

NCD =No crack detected

Table 4: SCC assessment of heat-treated and un-heat-treated (control) samples of 300M steel after one-hour test exposure at 100 °C

Medium No.	Normalized	Control	Tempered	Control
1	NCD	NCD	NCD	NCD
2	NCD	NCD	NCD	NCD
3	NCD	NCD	NCD	NCD
4	NCD	NCD	NCD	NCD
5	NCD	NCD	NCD	NCD
6	NCD	NCD	NCD	CD
7	NCD	NCD	NCD	CD

NCD =No crack detected, CD = Crack detected

#### 4. CONCLUSION

SCC behaviors of as-received un-heat-treated (control), normalized, and tempered 300M steel samples under constant applied stress load of 90% of their yield stress (1427.41 MPa) for one hour at 60 °C and 100 °C in seven aqueous media that contained different levels of hydrochloric acid from 0 to 17.5% and sodium chloride from 0 to 17.5% were separately investigated. Analyses of the tested samples by structural micro-examinations indicated that the normalizing and tempering heat treatments can have alleviating effects on stress corrosion behavior of 300M steel under such high tensile stress and exposure temperatures up to 100 °C in environments with each of acid and chloride contents up to 17.5%.

#### 5. ACKNOWLEDGEMENT

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#### 6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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