RESEARCH ARTICLE

Investigating the effect of solution annealing on corrosion resistance and hardness property of AISI 1036 welded steel in chloride environment using electrochemical noise method

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Article Info	Abstract
Article history: Received 02.12.2023 Revised: 27.01.2024 Accepted: 11.03.2024 Published Online: 28.06.2024 Keywords: Carbon steel, Solution Annealing, Corrosion Resistance, Electrochemical Noise, Hardness property, Chloride Environment	The outcomes derived from the investigation into the influence of solution-annealed AISI 1036 welded steel on the corrosion resistance and hardness properties in a chloride environment are presented in this paper. The solution-annealed steel samples were austenitized at a temperature of 990 °C and soaked for 2 hours to ensure complete homogenization and thereafter cooled in water. The control and the heat-treated samples were exposed to a chloride solution for corrosion investigation. Corrosion rates were examined for at the parent metal (PM), heat affected zone (HAZ), and welded zone (WZ) using the Potentiostatic measurement method (PMM), and hardness tests were conducted using Vickers microhardness testing (VMT). The results obtained from the hardness testing showed that the solution-annealed samples have higher hardness properties than the control samples at the welded zone, heat-affected zone, and the parent metal. When exposed to corrosion, the obtained corrosion rate showed that the solution-annealed samples. The corrosion resistance of the samples was observed to decrease from PM to the HAZ, and the WZ exhibited the least resistance. From the results obtained, the solution-annealed welded steel specimens seemed to be better suited for application in a chloride environment since they showed superior resistance throughout the exposure period.

1. Introduction

The fabrication and maintenance of oceanic facilities is one of the fundamental points to the sustainable improvement of the ocean [1]. Metallic materials such as carbon steel constitute important components of marine facilities [2]. Carbon steels, find widespread use across various industries owing to their notable properties such as wear resistance, high toughness, and strength. [3]. Carbon steel materials are predominantly used for ships, flow lines, transmission pipelines, and downhole tubulars in the oil and gas industry, most possibly owing to their low cost [4-6]. Furthermore, they are employed in diverse applications, including but not limited to axles, spindles, bolts, light gears, connection rods, crankshafts, worms, and various other aerospace and automotive products [3].

Consequently, the utilization of carbon steel for these applications entails a variety of shapes and sizes, ranging from simple to intricate. Engineers must apply their ingenuity in fabrication to accommodate diverse environmental conditions. Welding stands out as one of the most widely employed methods for fabricating carbon steel components. In essence, it involves the fusion of two or more metal pieces through the application of heat and, at times, pressure [7]. Welding comprises of a broad range of scientific variables such as welding speed, power input, electrode, temperature, and time [8-10].

However, when the carbon steel is exposed to a chloride environment they are susceptible to pitting corrosion, threatening the safety of the facility. Therefore, the need for adequate corrosion inspection techniques and maintenance measures. The durability of engineering marine facilities greatly hinges on its life-cycle maintenance and corrosion detection is the basis of this maintenance [11]. Conversely, the aggressive nature of the chloride environment is an essential factor affecting their durability. Thus, developing an electrochemical technique adequate enough to determine the corrosion degree of metallic materials and the corrosion type is essential. The electrochemical noise (EN) method has been adequately utilized in corrosion detection, most especially in marine environments because its detection equipment setup is portable and simple. In corrosion research, it is recognized as a reasonably mature electrochemical technology [12-13], since it has distinctive insitu, nondestructive detection [14]. The discovery by Iverson [15], that a metal undergoing corrosion at EOCP generates random ruptures of charges as a result of steady state fluctuations in potential and current at the interface of the metal/solution, has gained significant attention in recent years [16-19]. Electrochemical noise investigation is a remarkable technique that allows the determination of the occurrence of metal susceptibility to corrosion at EOCP devoid of the need for external potential perturbation [20]. Electrochemical noise usually occurs in the metallic electrode as a result of changes that take place at the surface states, for instance, the failure and repair of a protective surface film [21-25]. It offers valuable data pertaining to intricate electrochemical reactions mostly in a dynamic state. This fact allows the technique to be useful in the investigation of environment-assisted flaws [26-28], pitting corrosion [29-31], grain boundaries corrosion [32], and general or uniform corrosion [28, 33]. The analysis of EN signals has

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been considered to provide useful data about the corrosion rate and its type [34-36]. The electrochemical noise technique has gained the attention of researchers around the world. [37] showed that electrochemical current noise (ECN) is an effective technique that is closely related to corrosion rate. Investigation by [38], revealed that sudden peak amplitude of ECN signals depicts pitting corrosion. This finding is also in agreement with [39], who also observed that pitting corrosion was initiated by sharp fluctuation of current noise signals. [40], further revealed that pitting corrosion is considered to occur when isolated signals or events of high peak spike amplitude occur. [41] discovered that there was a steady state fluctuation with the electrochemical noise signals. This observation was attributed to the regular and synchronized depassivation and repassivation of the brownish formation of an iron-oxide film on the surfaces of the sample. The study by [40] showed that uniform corrosion is considered to occur when the instantaneous fluctuation signals are uniform and continuous. A higher standard deviation indicates a higher amplitude of ECN signals and a higher corrosion rate [42-43].

The effect of solution annealing on different materials on corrosion has been extensively researched. Most of these researchers however worked on different steel materials in various environments, but investigation on the impact solution annealing would have on the corrosion resistance using the electrochemical noise method on welded medium carbon steel in a chloride environment is lacking.

With the objective of investigating the effect of solution annealing on the corrosion resistance of welded AISI 1036 steel in chloride solution, the present study used the SEM-EDX analysis to assess the impact of solution annealing on the steel and electrochemical noise measurements to examine the resistance of the annealed steel to corrosion.

2. Materials and methods

2.1. Materials

A medium carbon steel substrate of 16mm diameter was used for this investigation. A spectrometer analyzer was utilized to analyze the chemical composition of the steel. Table 1 present the result obtained from the analysis.

Table 1. Chemical composition of the medium carbon

steel		
Element	% Composition	
С	0.36	
Si	0.28	
Mn	0.87	
Р	0.05	
S	0.056	
Cr	0.13	
Ni	0.10	
Cu	0.23	
Nb	0.01	
Al	0.01	
В	0.003	
W	0.03	
Mo	0.01	
V	,0.0001	
Ti	< 0.0001	
Fe	97.90	

2.2. Methods

2.2.1. Preparation of samples

The design welding specifications of the steel samples were obtained by machining. A power hacksaw was used to cut the samples into two equal parts. The V-edge design was adopted with a bevel angle of 30° in preparation for butt welding which is in line with the American Welding Society [44]. This welding geometry was selected based on the submission of [7] that acceptable welding penetration could easily be achieved by butt sample preparation. A summary of the various stages of the experiment is shown in Figure 1.

2.2.2. Welding procedure

The prepared steel samples were clamped firmly together with a root gap of 2 millimeters on a welding table. The steel samples were joined manually in pairs. The welding operation used for this research is the shielded metal arc welding (SMAW) technique. To achieve the thickness level of the welded steel during the welding process, the electrode was passed through the butt. On completion of the welding operation, the welded specimens were permitted to cool in the ambient air, and subsequently, an iron brush was employed for the removal of slag. The welding machine and electrode which were used for the experiment at the Department of Welding Engineering and Offshore Technology, Petroleum Training Institute, Effurun, Nigeria, are illustrated in Figure 2a and 2b respectively.



Figure 1. A flowchart showing the various stages of the experiment

2.2.3. Post-weld heat treatment

The welded samples were stress-relieved to remove internal stresses that were induced on them in the course of welding [45-46]. A muffle furnace was used to perform the solution annealing heat treatment for the heat-treated samples. The samples were austenitized at 990 °C and soaked for 2 hours to ensure complete homogenization and thereafter the samples

were cooled immediately in water. Some welded and unwelded samples were solution annealed while some were not solution annealed which were used as control samples. The heat treatment furnace which was used for the experimrnt at the Department of Metallurgical and Materials Engineering, Delta State Polytechnic, Ogwashi-Uku, Nigeria, is illustrated in Figure 2c.





(c) Figure 2. (a) Welding machine, (b) welding electrode and (c) muffle furnace

2.2.4 Hardness testing

In order to examine the effect of solution annealing heat treatment on the mechanical properties of the steel, the hardness test was carried out according to standard ASTM A384/89 (1990), [47-48]. The samples were prepared to give it a flat, smooth, and mirror-like surface. The hardness measurement was done with the aid of a Vickers Microhardness Testing Machine, with a microhardness scale of HV 0.02, a test force from a diamond pyramid of 0.1961N, with a dwell time of 10 (ten) seconds. The indentation was examined over the entire sample surface which cut across the PM, HAZ, and WZ. A camera was swung over the square impression produced by the diamond pyramid, the diagonals were measured between knife edges, and the reading was taken with the aid of a digital counter. The hardness values are obtained from the hardness testing machine which is equipped with an electronic sensor that permits the direct readout digitally from the machine.

2.2.5 SEM-EDX evaluation

Scanning Electron Microscope fitted with a digital camera and a computer was used to evaluate the Energy Dispersive Xray spectrometry for the solution annealed and the control samples.

2.2.6 Environmental preparation

For a comprehensive corrosion evaluation of the materials, the corrosion environment was established by dissolving 58.44g

of sodium chloride salt in 1000 ml of distilled water. The resulting solution was stored in a 20-liter container, appropriately labeled.

2.2.7 Electrochemical corrosion measurement evaluation

The electrochemical corrosion susceptibility of the examined steel was investigated using potentiodynamic potentiostat/galvanostat (VoltaLab) with methods. А VersaSTAT 4 software was used for data gathering and analysis of the corrosion, and the electrochemical measurements were done after the tool was properly calibrated. The measurements were done with three-electrode cells with a platinum electrode acting as the counter electrode and the reference electrode is a saturated (Ag/AgCl) calomel electrode (SCE). The epoxy resin was used to properly seal the samples acting as the working electrodes. The samples (working electrode) were wet ground and polished with a 1.5 µm diamond paste, properly washed in 95.6% ethanol distilled water, and air dried. Silica gel sealant was used to seal the interfaces between the samples and the resin in order to prevent crevice corrosion, and air dried. The total surface area of the exposed working electrode was 45.26 cm². Polarization investigations were done at a scan rate of 0.5 mV/s in 1.0M NaCl solution (electrolyte) at room temperature (26 + 1°C). The corrosion susceptibility was examined after 1 hour of immersion. The corrosion susceptibility was examined by open circuit potential (OCP) monitoring for 600 seconds. The EN measurement was done under OCP and the result was evaluated by the software. This measurement is in accordance with [49-50].

3. Results and discussion

3.1. Hardness test results

The hardness distribution in the different regions of the unheat treated and solution-annealed samples as presented in Figure 5a-i are summarized as shown in Figure 5j. The results showed that the hardness increases from the WZ, HAZ to the PM for both samples. This variation in properties across the welded samples could be attributed to the different rates of cooling across the sample. These findings correspond with [46, 51]. The welded metal zone was observed to have the lowest hardness value and this finding aligns with [52], and this could be attributed to the loss of quenching elements from the WZ, their coarse structure [53-55] and also due to grain growth [56]. The occurrence of coarse grain size in the WZ can also be attributed to the heat cycle, low dislocation density, and dissolution of precipitation [57]. The parent metal was observed to have the maximum hardness for both samples and this finding aligns with [52]. These could be attributed to the finer grain size [55]. The solution-annealed samples showed higher hardness than the un-heat-treated samples. This implies that the carbides dissolution in the matrix of the solution-annealed samples has helped to stretch the matrix of the steel resulting in volumetric expansion with martensite formation and also impede dislocation movement which resulted in increased hardness of the solution-annealed samples. This finding is in close agreement with [58-63]. It was also reported by [64-68] that the hardening mechanism in steel is due to the martensitic structure (phase). The higher hardness associated with the solutionannealed samples over the as welded samples can also be attributed to the existence of retained austenite in the structure. This finding aligns with [46, 69]. [70], they observed that retained austenite enhanced hardness property as it prevented the generation of pitting holes at the grain boundary.



Figure 5. (a) Hardness crop image of the un-heat treated sample, (b) Hardness indents image of the un-treated sample, (c) hardness raw plot indents image of the un-treated sample, (d) Hardness spectrum of the un-heat treated sample, (e) Hardness crop image of the solution annealed treated sample, (f) Hardness indents image of the solution annealed treated sample, (g) Hardness raw plot indents image of the solution annealed treated sample, (i) SEM indent image of the samples, (j) Hardness plot of the solution annealed treated samples.

3.2 SEM-EDX evaluation result

Figures 6a-b show the result of SEM-EDX of AISI 1036 steel solution annealed and un-heat treated parent metal and weld metal of the samples. It is found that Fe concentration gave

a very high spectrum indicating that it is the predominant element for both the solution annealed heat-treated and the unheat-treated samples. The extrapolated Figure 6b showed a remarkable difference between the solution-annealed and unheat-treated samples. It was observed in the un-heat treated samples from the parent to the weld metal zones that there are spikes of alloying elements C, Si, S, Cr, and Mn indicating that the alloying elements are not homogeneously dispersed in the matrix of the samples, while in the solution annealed samples there were no spikes of alloying elements indicating that the alloying elements are homogeneously dispersed in the matrix of the samples. It was also observed in the solution-annealed samples that the carbon is fully solutionized in the matrix of the samples which is one of the major reasons of solution annealing heat treatment and this finding is in good agreement with [48-49, 71]. They observed in their studies that solution annealing is effective in causing the dissolution of the precipitated intermetallic phase (Fe₃C phase).



Figure 6. (a). EDX spectrum of welded 0.36%C steel solution annealed and un-heat treated parent metal and weld metal of the samples (b). Extrapolated EDX spectrum of welded 0.36%C steel solution annealed and un-heat treated parent metal and weld metal of the samples

3.3. Electrochemical current noise results

The ECN method was reported as an effective technique that is closely related to corrosion rate [37]. This finding was observed in Figure 7a-f which shows the ECN plots for the control and solution annealed PM, HAZ, and the WZ of the samples after 1 hour of immersion in 1.0M NaCl Environment. The ECN plot in Figure 7a of the control PM sample, showed the spontaneous fluctuation with time of the that electrochemical current noise passing through the sample surface maintained a steady state fluctuation with a peak spike at 191.7 seconds of immersion at a current of -8.815E-08 (A) and thereafter a steady state fluctuation signals was observed with exposure time. This peak fluctuation observed in this study can be attributed to pitting corrosion taking place as a result of film failure and hence fresh sample surfaces were exposed to further corrosion until a passive film was formed. This finding is in agreement with [38-39]. This observation is also in strong agreement with [40], they observed that pitting corrosion is considered to occur when isolated signals or events of high peak spike amplitude occur. This peak fluctuation indicated that there is a tendency for pitting corrosion to take place in the control sample. The film failure observed in this study could be due to chloride ions in the environment. This finding is in agreement with [72], who reported that chloride ion-induced corrosion led to passive film failure and repair on steel surfaces. The steadystate fluctuation later observed could be attributed to the electrochemical events associated with regular and synchronized depassivation and repassivation of brownish iron oxide film formation on the sample surfaces. This finding is in strong agreement with [41]. The ECN plot in Figure 7b of the solution annealed PM sample, shows that the spontaneous

fluctuation with time of the electrochemical current noise passing through the sample surface maintained a steady state fluctuation with no apparent significant peak spike throughout the period of exposure. The steady fluctuation observed in this study can be attributed to uniform corrosion. This finding corresponds with [40]. This steady fluctuation can be ascribed to the homogeneous dissolution of the carbides in the matrix of the solution-annealed PM sample. This finding is in good alignment with the SEM-EDX result. The ECN plot in Figures 7c and 7e of the controlled HAZ and the WZ, were observed to follow the same pattern of corrosion tendencies as observed with the PM with more increased peak spikes from the HAZ to the WZ. The plots in Figure 7d and 7f of the solution annealed HAZ and the WZ were also observed to follow the same pattern of corrosion tendencies as observed with the PM. Pit initiation associated with sudden high peak current signals and reparation of the metastable film was not observed on the solutionannealed steel samples in the chloride environment. This observation is in strong agreement with [73-75]. This observation is in good agreement with [76], who in their research observed that solution annealing increased the resistance of their samples to pitting corrosion as a result of the dissolution of carbides and secondary phases in the matrix of the steel which would have been precipitated at grain boundaries.

In summary, from the ECN investigation carried out, the control samples were observed to be more susceptible to pitting and uniform corrosion while the solution annealed samples were observed to be only susceptible to uniform corrosion. These findings can be attributed to the homogeneous dissolution of the carbide in the matrix of the solution-annealed samples. These findings are in strong agreement with [77-78].



Figure 7. Electrochemical Current Noise Plots for: (a). Control PM (b) Solution annealed PM (c) Control HAZ (d). Solution Annealed HAZ. (e) Control WZ (f) solution annealed WZ

3.4 Corrosion rate results from tafel slope

Figures 8a-c show the Tafel plot of electrode potential against the current density for the control and solution-annealed samples in 1.0M NaCl environment. The Tafel plot in Figure 8a, shows that the anodic current of the control PM samples gave the highest anodic potential of -0.72V (SCE) compared to the solution annealed PM sample that gave an anodic potential of -0.70V (SCE). This finding depicts that the solution-annealed samples are more noble and that the control samples are

corroding more than the solution-annealed sample. This finding could be attributed to the homogenous dissolution of the carbides in the matrix of the solution-annealed PM, which resulted in its low anodic potential. This is in accordance with the findings of [79], who reported that low anodic potential results in low corrosion rates and that high anodic potential results in an increase in the corrosion rate in chloride medium. [80] and [81] also reported that low anodic potential favored corrosion rate reduction. The Tafel plot in Figure 8b, shows that the anodic current of the control heat-affected zone sample gave the highest anodic potential of -0.73 V (SCE) compared to the solution-annealed HAZ sample that gave an anodic potential of -0.71V (SCE). This finding depicts that the solution-annealed samples are more noble and that the control HAZ samples are corroding more than the solution-annealed HAZ samples. This finding can be attributed to the homogenous dissolution of the carbides in the matrix of the solution-annealed samples which is in agreement with [71], and also to the presence of carbides at the grain boundaries in the HAZ of the control samples. This finding aligns with [78], who reported that the presence of carbides at the grain boundaries in the HAZ was responsible for its high susceptibility to corrosion.

The Tafel plot in Figure 8c, shows that the anodic current of the control WZ samples gave the highest anodic potential of -0.73V (SCE) compared to the solution annealed WZ samples that gave an anodic potential of -0.71V (SCE). This finding depicts that the solution-annealed samples are more noble and that the control WZ samples are corroding more than the solution-annealed WZ samples. This finding can be attributed to the homogenous dissolution of the carbides in the matrix of the solution annealed samples which is in agreement with [50].



(c)

Figure 8. Tafel Polarization Plot for: (a).CPM and SAPM of the samples (b). CHAZ and SAHAZ of the samples (c). CWZ and SAWZ of the samples

4. Conclusion

From the results of the investigation carried out, the following conclusions were made:

- 1. The optimum heat treatment conditions (temperature and holding time) for solution annealing treatment of AISI 1036 steel were 990 °C and 2.0 hours.
- 2. The hardness distribution in the different regions of the sample was found to increase from the WZ, HAZ, and PM for both control and solution annealed samples. However, the solution-annealed samples were found to have higher hardness across the different regions. The control samples

were found to have lower hardness values of 148 - 174 HV at the WZ, 174 - 187 HV at the HAZ and 187 - 252 HV at the PM. The solution-annealed samples were found to have higher hardness values of 186 - 255.8 HV at the WZ, 255.8 - 325.6 HV at the HAZ, and 325.6 - 535 HV at the PM.

- 3. The heat-treated steel samples were less susceptible to corrosion when compared with the control samples.
- 4. The control samples were observed to be susceptible to pitting and uniform corrosion, while the solution-annealed samples were observed to be only susceptible to uniform corrosion.

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Author contributions

Basil Obimma Onyekpe provided guidance and supervision during the research work, mainly in the interpretation and discussion of results. The material characterization, research investigation, data collection, analysis, writing, and editing of the manuscript were carried out by Oghenerobo Awheme.

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