Microstructural and Corrosion Characteristics of a Heat Treatable Stainless-Steel Weldments

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Abstract. In this research, AISI 316 stainless steel samples were welded and subjected to post-weld heat treatment (PWHT). Thereafter, they were evaluated for microstructural and corrosion behaviours. PWHT was carried out on some of the welded samples at 400°C for 12 minutes in the muffle furnace. Investigation of microstructure and chemical characterization of the samples was performed using scanning electron microscope, whilst also corrosion rate was evaluated by weight loss method in a controlled environment. The as-welded and post-welded samples were immersed in 0.5M of tetraoxosulphate (IV) acid at room temperature and then studied for 15 days with each sample removed at 3 days interval. It was evident that the post-weld heat-treated samples under controlled heating condition showed higher resistance to corrosion than the as-welded sample which is attributed to the fact that PWHT increases steel weldment resistance to stress corrosion cracking ultimately. Therefore, corrosion susceptibility of stainless steel in H2SO4 medium during exposure time could be a pointer to the aggressive sulphide ion (SO4-) which continuously breaks down the protective film on the metal sample. Two major carbide precipitates of Cr23M7 and CR7C3 were evident after the simulation of the research material, which could pose a detrimental effect on the general performance of the steel in engineering and other industrial applications.

Keywords: AISI 316; Arc Welding; Post weld heat treatment, H2SO4; Corrosion; SEM.
1. Introduction

Stainless steel was discovered in the early stages of the 20th century. Its classification was based on the constituent microstructure of the individual stainless-steel members. There are austenitic, ferritic, martensitic, and precipitation-hardenable (PH) stainless steels with the first two being the most common and abundant of the family. Austenitic stainless steels form the largest stainless-steel family with reference to alloys and usage in the steel industry. The basic alloying element in stainless steel is said to be the chromium compound. Others include nickel, molybdenum, nitrogen and carbon at different proportions. Applications at elevated temperature and aggressive media/environment in chemical industries made a new dimension and significant step on the discovery of stainless steel. Corrosion and oxidation resistant materials were introduced into the engineering sectors [1, 2]. A combination of the austenitic and ferritic stainless-steel species gives a much stronger and better variety of stainless steel. It is called duplex stainless steel (DSS). The modern development of the DSS grade was introduced which had improved welding properties strictly due to the nitrogen application in the alloy matrix [3].

The corrosion process has been right from the time a basic issue of concern in the metallic and engineering world. Its prevention and control as well have posed a substantial degree of retardation in the technological development of metallic compounds and materials [4]. The constant degradation of a material’s properties (both chemical and physical) due to interaction with the environment is a simple definition of corrosion [5]. A reduction in a system’s Gibb’s energy is the driving force for corrosion to occur i.e. the natural tendency for materials and metals to return to their natural state.

Heat treatment is defined as a sequence of heating and cooling that is designed to obtain some desired combination of properties on the material been heated [6]. Phase transformations as well as structural changes like Chromium rich carbide formation and dendritic grain formation which do take place around the welded joint thereby making the weldment susceptible to corrosion attack [7]. But post-weld heat treatments are the causes or reasons for changes in the properties of the material after treatment. The “principles of heat treatment” are factors that determine and control these changes. A substantial amount of residual stress occurs on weldment which is basically due to restraint by the parent metals of the steel component during weld solidification which can become a source of stress corrosion while in service [8].

Additionally, the presence of asymmetrical grains, element segregation, and thermal stresses lead to lower ductility in the material, and poor performance [9, 10]. An estimated US$4 trillion in global annual loss due to corrosion and corrosion protection expenses includes corrosion as well as everything related to its protection [11, 12]. Pitting during the corrosion in stainless steels is one of the primary failure mechanisms [13-16]. The heat treatment here was selected to eliminate the internal stress and get homogenized the microstructure after the welding processes [17]. This study is therefore geared towards validating the metallurgical and corrosion characteristics of AISI 316 weldment with a view to ascertaining specific scientific measures in curbing the possible menace welding could pose. All these stated challenges necessitated this research focus.

2. Experimental procedure

2.1. Materials and equipment

Materials used for the research work include the following; stainless steel sheet, acetone, distilled water, paper grits (80, 120, 220, 320, 400, 600, 800 and 1000 grit sizes), stainless steel electrodes (AWS A5.4-E316), tetraoxosulphate IV acid (H$_2$SO$_4$) solution while equipment include; Miller blue thunder series 443
SMAW Arc welding machine (Model: SKU MIL-029016236), JSM-F100 Schottky field emission scanning microscope (SEM), Electric furnace (Model: CHO-3.5/5.3.5/12.5), desiccators, digital weight balance and beakers.

### 2.2 Microstructure and chemical analysis

Both as-welded and post-welded samples were cut out and carefully examined for compositional characteristics in weight percentage as shown in Table 1.

| Element (wt.%) | C  | Mn  | Si  | S   | P   | Cr  | Ni  | Ti  | Al  | Fe |
|---------------|----|-----|-----|-----|-----|-----|-----|-----|-----|----|
| Parent Metal  | 0.540 | 1.730 | 0.480 | 0.008 | 0.008 | 18.530 | 8.4900 | 0.008 | 0.006 | Bal. |
| Welded Metal  | 0.680 | 1.280 | 0.750 | 0.002 | 0.001 | 17.310 | 7.2400 | 0.012 | 0.004 | Bal. |
| Post Welded Metal | 0.730 | 1.260 | 0.650 | 0.001 | 0.001 | 17.430 | 7.6000 | 0.010 | 0.003 | Bal. |

### 2.3 Welding and sample preparation

A size of 40mm x 30mm x 1.6mm specimens were welded in pairs to obtain ten different variations. The electric arc welding process was used to join the cut pieces together. The voltage and current passing through the welding machines were set at 22V and 100A respectively. A butt joint was utilized in the welding process. Stainless steel electrode was used for electric arc welding due to the thickness of the specimen.

### 2.4 Post-weld heat treatment

Post-weld heat treatment was done on five welded samples using controlled heating at 400°C for 12 minutes in a furnace. Thereafter, water was employed as the quenching medium to cooling.

### 2.5 Determination of corrosion behaviour

The weight loss system of corrosion studies was conducted as per the ASTM methods to determine the corrosion characteristics of the samples. Each of the prepared specimens was weighed before and after the immersion in order to determine the corrosion behaviour. The digital weight balance machine which measures a weight of 0.001g was used. The welded portion (the weldment and the HAZ) was cut out from the unaffected zones. This was then weighed and the result taken for all five samples. The samples were then wholly immersed in 0.5M of H₂SO₄ solution at ambient temperature. The post-welded samples were also immersed in the same solution for corrosion investigation.

### 3. Results and discussion

#### 3.1. Microstructure study

Scanning Electronic Microscopy (SEM) analysis was performed to study the surface morphology of the samples after immersion in the corrosive medium. The SEM micrographs of the specimen are shown in Figure 1 (a)-(g). A passive layer is formed due to corrosion in all samples that is; As-received sample
(Figure 1 (g)), As-welded sample (Figure 1 (a)-(c)), and PWHT sample (Figure 1 (d)-(f)). But the thickness of the layer is differing from sample to sample. There is more aggressive behaviour in the As-welded sample due to the effect of heat from the weld pool. This region is called the heat-affected zone (HAZ); it implies that the microstructure is greatly affected by the heat generated during welding as posited by Clark [18]. Many researchers have investigated the HAZ of the AISI 316 welding and came to a conclusion that there exist some features within the confined of HAZ region which are: the coarse grain (CGHAZ) which is fully austenite phase at a temperature range of 1000°C-1250°C, fine grain (FGHAZ) boundary layer which is the austenite phase and the inter-critical zone (ICHAZ) which is at 50% of austenite phase transformation. Due to these disturbances in phases of AISI 316 welded structure, precipitation is bound to occur which plays a significant role in the aggressive corrosive nature in the regions [19-23].

Also, presented in Figure 2 (a)-(d) are the optical micrographs of the tested parent metal studied for corrosion characteristics for 3 days and 7 days in 0.5M H₂SO₄ medium. Essentially, the parent metal composed of largely coarse austenite grains with tiny amounts of ferrite. Annealed twins were observed crossing the grain boundaries. The results in figure 2 (d) show significant damage occurred on the surface of the material after 7 days of immersion and a craggy-like terrain is observed. Evident from Figure 2 (b) and (d) are the pitting profiles of the steel which indicate the pattern of pitting on the material of experimentation. These observations are consistent with other researchers’ view on corrosion of AISI 316 stainless steel [24, 25].

![Figure 1 SEM Microstructure of: (a) As-welded stainless-steel joint at 500x, (b) As-welded stainless-steel joint at 1000x, (c) As-welded stainless-steel joint at 1500x, (d) Post-welded stainless-steel joint at 500x, (e) Post-welded stainless-steel joint at 1000x, (f) Post-welded stainless-steel joint at 1500x and (g) parent metal at 4000x magnifications](image-url)
Figure 2 Optical Microscopic images of the corroded samples of AISI 316 parent metal examined in 0.5M H₂SO₄ at Magnifications of: (a) 50x for 3 days (b) 200x for 3 days, (c) 50x for 7 days and (d) 200x for 7 days

It is observed that the post-weld heat treatment gives the low corrosive pitting than the as-received and welded samples with respect to pitting and surface roughness, pits distribution and size with a depth of cavities as shown in Figures 3 and 4.

Figure 3 Graphs of As-received samples, As-Welded samples and Post-weld Heat treatment for: (a) Maximum pit size, (b) Minimum pit size and (c) Average pit size of austenitic stainless steel
Figure 4  Mechanism of corrosion of As-received samples, As-Welded samples and Post-weld Heat treatment: (a) corrosion affected area (%), (b) number of corrosion pit/mm² and (c) surface roughness (µm)

3.2. Weight loss Analysis

Weight loss experiments were conducted in accordance with the ASTM methods described previously. Gravimetric measurements were carried out in an electrolysis cell equipped with a thermostat-cooling condenser. The carbon steel specimens used have a rectangular form of 40mm x 30mm x 1.6mm. After the immersion period, the specimens were cleaned according to ASTM G-81 and reweighed in order to determine the corrosion rate [26]. Duplicate experiments were performed in each case, and the mean value of the weight loss was reported. The weight loss mechanism gives room for the calculation of the mean corrosion rate as expressed in (mg.cm⁻².h⁻¹). It can be determined either by chemical analysis of dissolved metal in solution or by gravimetric method measuring the weight of specimen before and after exposure in the aggressive solution applying the following equation:

\[ W(\text{corrosion}) = \frac{M_i - F_i}{s t} \]  

Where \( M_i \), \( F_i \), \( s \) and \( t \) denote initial weight, final weight, the surface of specimen and immersion time, respectively.

3.3. Corrosion behaviour

The computed values for resistance to corrosion of both samples after 15 days at 3 days interval using weight loss method is tabulated below.

Figure 5 shows the corrosion rate (mm/yr) for AISI 316 steel specimen immersed in 0.5M H₂SO₄ for 15 days at 3 days intervals for each of the five samples. These five samples are functions of the time of their exposure to the corrosive medium at room temperature which ranges between 26 -33°C. Results obtained show an indication that the rate of corrosion as the days increased from 3 to 15 days for uniform concentration (0.5M) of H₂SO₄ acid, decreased moderately for
treated samples and significantly decreased for untreated samples. This is in agreement with the works of Arwati et al., [27] and Iliyasu et al., [28]. This unique behaviour of the medium is very much attributed to the corrosive or acidic condition of the acid at the onset of the procedure (first 3 days). As time goes on, acidity tends to decrease due to its reaction with the steel specimen. The graphs indicate a curve tending towards a horizontal position to be constant no matter how long it will take.

Figure 5 Corrosion rate (mm/yr) for AISI 316 steel specimens of untreated and treated weldment immersed in 0.5M H₂SO₄ for 15 days at 3 days intervals for each of the five samples.

3.4. Phase Study of research material.
Figures 6 and 7 are the graphs generated from the phase property diagram simulation using the JmatPRO software. From the simulated data, it can be seen that the major phases of ferrite are present in the steel generally and a negligible amount of cementite is existing. The austenite commences from around 750°C, this is the Ac1 temperature and the 100% transformation of austenite at 900°C, that is Ac3 temperature. Therefore, according to Kou [19], the CGHAZ is generated at 1300°C (Ac3<<); FGHAZ is estimated at 900°C (Ac3) and the ICHAZ is estimated at 850°C (<Ac1>). The Liquid phase is starting from around 1500°C. At 1400°C the austenite dissolved and transforms into delta ferrite (DF) as a result of the gamma alpha transformation, which is dangerous and leads to poor mechanical properties of the steel [29-30]. From the phase transformation study, it can be noted that precipitations can also be generated in these steels such as M23C6 and M7C3. The M23C6 precipitated carbides is the Cr23C6 with a mixture of
molybdenum richened carbides and the M7C6 precipitates are the Cr7C3 with a rich iron in the carbides.

![Figure 6](image)

**Figure 6** Phase diagrams of: (a) the simulated steel, (b) and (c) M7C3 carbides chemical composition

### 3.5 Carbon and chromium content

Presented in figure 7 (a) and (b) are the percentage of carbon and chromium present in the steel phases. Carbon is an interstitial alloying element in steel that can easily diffuse speedily through the structure and focus on the grain boundaries. The most challenging issues with carbon are the precipitation of chromium carbide that tends to deplete the grain boundaries of chromium, thereby posing a high risk to the steel in terms of its resistance-ability to corrosion. While the chromium content plays the role of toughens the steel and subsequently improves its resistance to corrosion at elevated temperature. These views are in agreement with the works of John and Tverberg [31]. The concentration of chromium and carbon is richer in the precipitated carbide of Cr23M7 and CR7C3. These precipitates are the main reason for creep and impact toughness enhancement improve the creep and impact toughness by blocking and creating the peening effect to stop/ slow down the dislocation movements [32-35].

Looking at this from a different perspective, as the welding progresses, the microstructure tends to distort invarious morphology with respect to their thermal effect that resulted in the HAZ formation. Precipitation of MC, Cr7C3 and Cr23C6 carbides are inevitable in this zone. Precisely, there is segregation of carbon and chromium from the ferrite and austenite matrix, giving rise to a more stable bond with chromium carbides. It was evident that there is a reduction
in the percentage of chromium and carbon elements in the welded sample of experimentation than the As-received sample, which resulted in corrosion aggression on the welded samples as shown in Figure 3 and 4. The corrosion aggression in the PWHT sample possesses less susceptibility than As-received samples. This due to the available time of heat treatment at 400°C to dissolve all precipitates in the matrix. It will be of great recommendation for PWHT for welded AISI 316 to enhance its corrosion resistance-ability properties.

Figure 7 The percentage of: (a) Carbon present in the steel phases and (b) the percentage of Chromium present in the steel phases.

4. Conclusion

The following conclusion can be drawn from this study;

i. The corrosion resistance of post-weld heat-treated samples of AISI 316 stainless steel welded metal is higher than that of its parent metal and As-received samples.

ii. Chromium content in AISI 316 seems to be a strong determinant of corrosion resistance of the steelweldment.

iii. The general resistance to corrosion due to its low ability to form insoluble (precipitates) compounds in the acid medium was not significantly affected by Mn additions.

iv. Additionally, the formation of insoluble molybdenum, Carbon, Chromium has increased the corrosion performance of the steel.
v. Controlled post-weld heat treatment improves corrosion behaviour of an as-welded sample.

vi. Corrosion susceptibility of stainless steel in H₂SO₄ medium during exposure period could be attributed to the aggressive sulphide ion (SO₄⁻) which continuously breaks down the protective film on the metal sample.

vii. Two major carbide precipitates of Cr₂3M7 and CR7C3 were observed after the simulation of the research material, which could pose a detrimental effect on the general performance of the steel in engineering and other industrial applications.

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Compliance with ethical standards
The authors declare that there is no conflict of interest in the course of this research article.

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