Research Article

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Investigation of Solution Annealing Treatment Effect on Corrosion Resistance of AISI 304 Austenitic Stainless Steel for Oil Industry Application

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ABSTRACT

Effect of solution annealing on the corrosion resistance of AISI 304 stainless steel in hydrochloric acid media was studied. Corrosion tests were performed on AISI 304 stainless steel immersed in 0.5- 1.5M HCl and heated between 30 °C to 60 °C to simulate certain environments in the oil industry. Corrosion rate was determined for solution- annealed and un- treated samples using the weight loss measurement. Results show that corrosion rate of the samples increases with concentration and temperature of HCl medium; and that the annealed stainless steel samples show up to 36% corrosion resistance improvement than the untreated stainless steel samples.

Keywords

Austenitic, Stainless steel, Annealing, Corrosion resistance, Corrosion rate.

Introduction

The strong susceptibility of plain carbon steels to corrosion and oxidation led to the development of stainless steels in the beginning of 20th century [1]. Stainless steels are basically iron-based alloys containing more than 12% chromium for passivity [2]. Stainless steels are frequently classified according to their microstructure into five types: austenitic, ferritic, martensitic, duplex (ferritic- austenitic) and precipitation hardnable [2]. Due to the superior corrosion resistance, excellent mechanical properties and good weldability of austenitic stainless steels, they are used in oil and gas industries as well as in power generation industries [3-5]. Austenitic stainless steels represent about 60% of world's total stainless steel production [6,7]. They exhibit properties such as good corrosion resistance, high strength, toughness, good ductility and weldability [8]. These steels are used in applications which entail high stress level and/or exposure to corrosive atmosphere like in nuclear power plants, oil industries, marine environments, food and beverage manufacturing industries, chemical processing plants, bulk storage, aircraft structural parts and a wide variety of industrial applications [9,10].

Austenitic stainless steels are primarily divided into two basic categories: the 300- series in which nickel is added to (Fe- Cr) system as the major constituent for austenite stabilization; and the 200- series in which manganese is used to replace nickel to stabilize austenite structure [11]. Other alloying elements such as Ti, Mo, Cu, N, Cu and Nb etc are added to stainless steel composition to improve formability, mechanical and corrosion resistance of stainless steels.

The corrosion resistance of austenitic stainless steels is due to the formation of thin, nano- sized, passive oxide layer, which is composed of iron and chromium oxide [7,12]. The breakage of the oxide passive film due to aggressive ions, flaws and inclusions leads to pitting and intergranular corrosion attack. Pitting is a severe form of localized corrosion which is caused by a redox (reduction- oxidation) process initiated at certain points on the surface of a metal due to the non- uniformity of the passive layer, and which results in the formation and inward growth of microscopic holes on the steel surface, leading to the premature failure of components [9]. Intergranular corrosion occurs due to grain boundary depletion of corrosion resisting constituents (sensitization) especially chromium during slow heating or cooling in the temperature range of 450°C and 850°C or segregation of impurities at the grain boundary areas [7].

Intergranular and pitting corrosion are catastrophic in nature and can lead to premature failure of components of aircraft structures, power plants, marine vehicles and other structures where stainless steels are applied with disastrous consequences. Hence new insight to their mechanisms, propagation and control is of tremendous importance [13,14]. Solution annealing treatment causes changes in microstructure, grain sizes of materials and phase transformation in stainless steels and all these microstructural alterations affect the corrosion resistance of austenitic stainless steel materials. Loto et al. [7] studied the effect of heat treatment processes on corrosion resistance of AISI 301 austenitic stainless steel and observed from open circuit potential measurement large cathodic shift for quenched steels compared to anodic shift for untreated stainless steel materials. Taiwade et al. [11] reported that solutionizing stainless steel at higher temperatures and longer times increased grain sizes and that larger grains reduced the degree of sensitization of stainless steels when compared with smaller grains. Research by Azadi et al. [15] showed that precipitation of secondary carbides consisting of chromium carbides and ε-carbides on duplex stainless steel during ageing treatment affected the corrosion resistance of the alloy. Tukur et al. [16] observed that annealing of AISI 304 stainless steel at high temperature (above 1050 °C) causes sensitization of stainless steel and that this leads to the carbide precipitation at grain boundaries. They also reported that this affects the resistance of the steel material to intergranular corrosion. Designing annealing heat treatment process in stainless steels for optimal result in terms of microstructure, properties and performance poses a huge challenge. Solution treatment of austenitic stainless steel at high temperatures and longer times will lead to the dissolution of alloy carbides in the alloy system and formation of large grain sizes. Cooling to ambient condition can result in the precipitation of chromium carbide at grain boundaries, which leads to intergranular corrosion. Thus, the annealing of austenitic stainless steel can be both beneficial and/or detrimental to the steel's corrosion behaviour. This research work aims to investigate the effect of solution annealing on the corrosion resistance of AISI 304 stainless steel.

Experimental Method

AISI 304 stainless steel obtained from Curtix Metals Nigeria Limited, Port Harcourt was used for this study. Spark analysis was performed on the steel material at Delta Materials Testing Company limited, Delta State Nigeria and the chemical composition is presented in Table 1.

Table 1: Chemical composition of AISI 306 stainless steel sample.

Sample	C	Mn	P	S	Cu	Cr	Ni	Mo	Fe
AISI 304 SS	0.04	1.25	0.03	0.008	0.21	18.20	8.28	0.13	70.8

The stainless steel sample was machined to coupon sizes with surface area of 100 mm² ± 0.5 mm². The samples were solution treated in a muffle furnace at 1000°C and soaked for 1 hr. The samples were, thereafter, cooled in the furnace. The samples were abraded in sequence using silicon carbide of grit sizes 220, 320, 600, 800, 1000. Polishing was done with diamond liquid slurry to 6 μm and then finally washed with distilled water and sprinkled with acetone and then stored in a desiccator prior to corrosion tests. 0.5- 1.5M HCl was prepared from analytical grade of HCl with distilled water and heated to between 30°C and 60°C, in order to simulate working environment of some plants in a refinery [17]. Corrosion rate, CR was calculated from the following equation [15].

$$CR = \frac{87.6W}{DAT} \quad [1]$$

Corrosion resistance improvement (CRI) was determined as follows [18]

$$CRI = \left(\frac{CR_{unt} - CR_{ann}}{R_{unt}}\right) X\ 100 \quad [2]$$

where W is weight loss in mg, D is density of corroding sample in g/cm³, A is total surface area in cm², T is time in hours, unt and ann are untreated and annealed respectively.

Results and Discussion

The steel spontaneously undergoes electrochemical reaction in the HCl media resulting in the oxidative dissolution of iron as well as the deposition of Fe^{2+} ion in the solution and the release of H_2 as described by the following equation:

$$Fe + 2H^+ = Fe^{2+} + H_2 \quad [3]$$

The above electrochemical reaction (equation 3) consists of separate anodic and cathodic reactions. The anodic reaction involves iron dissolution and the generation of ferrous ions which passes into the solution; and the release of electrons according to reaction 4 [19]:

$$Fe = Fe^{2+} + 2e \quad [4]$$

Cathodic reaction involves hydrogen evolution reaction during which hydrogen ion is reduced to hydrogen gas as represented in reaction 5:

$$2H^+ + 2e = H_2 \quad [5]$$

And oxygen reduction reaction since the HCl solution is diluted in water, oxygen is present in dissolved amount [18]. The dissolved oxygen is reduced during corrosion process according to equation 6:

$$O_2 + 4H^+ + 4e = H_2O \quad [6]$$

The reactions occur at different locations on the steel surface especially on flaws, non metallic inclusions and defects etc.

Figures 1 and 2 show the effect of hydrochloric acid concentration on the corrosion rate of AISI 304 stainless steel under annealed and untreated (as- received) conditions at 30°C and 60°C respectively. As seen, the corrosion rate of the samples increases as the acid concentration was increased. A comparison between figures 1 and 2 shows that corrosion rate was higher when corrosion test was performed at a higher temperature. It can also be deduced from the experimental data that the annealed samples have higher corrosion resistance and showed up to 36% average corrosion resistance improvement (determined from equation 2) than the untreated stainless steel material. Increase in corrosion rate with acid concentration can be attributed to the ability of the chloride ions to penetrate the stainless steel- formed film (Cr_2O_3) on the surface of the steel material and the fact that to initiate, penetrate and sustain corrosion reactions is more drastic at higher concentration. The consequence of this was that of severe active corrosion reactions of anodic dissolution of the alloy. Moreover, in the presence of Cl^- ions, at high concentrations, the ability of the stainless steel to repair its film is drastically reduced and protection is hence lost [20].

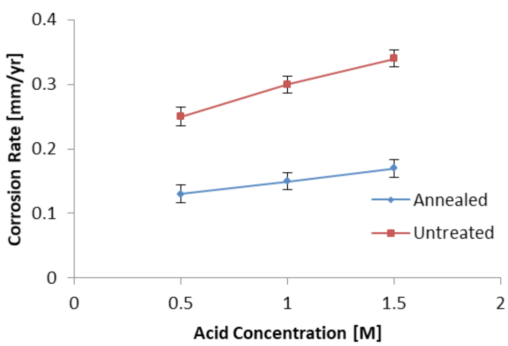

Figure 1: Effect of HCl concentration at 30°C on corrosion rate of AISI 304 stainless steel.

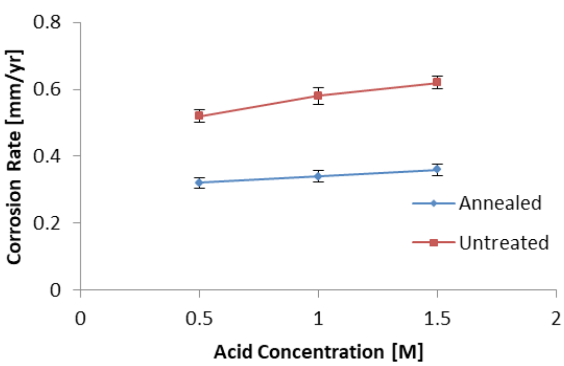

Figure 2: Effect of HCl concentration at 60°C on corrosion rate of AISI 304 stainless steel.

The effect of acid temperature on the corrosion rate of AISI 304 stainless steel in 0.5- 1.5M HCl solution is shown in figures 3 and 4 respectively for annealed and untreated samples. As can be observed, at constant acid concentration, the corrosion rate of the stainless steel samples increases as the temperature of the acid is increased. This is probably because when the temperature of the corrosive environment increased from 30 to 60 °C, the created films became thicker with higher porosities and led to film rupture and a decrease in the effectiveness of corrosion protection [21].

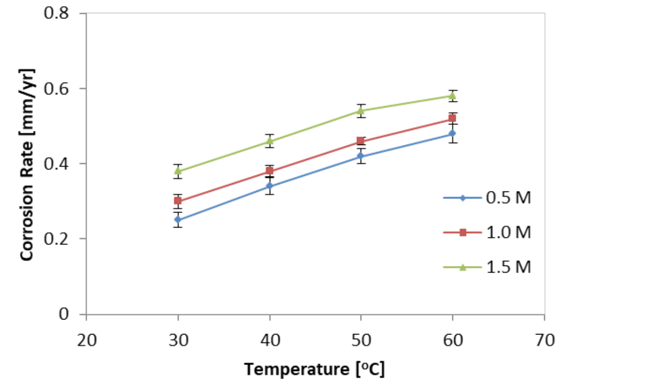

Figure 3: Effect of temperature on corrosion rate of un- treated AISI 304 stainless steel.

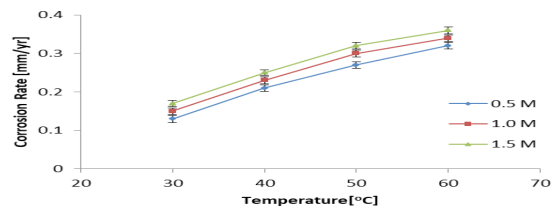

Figure 4: Effect of temperature on corrosion rate of annealed AISI 304 stainless steel.

The corrosion rate of the steel sample is generally higher for the un- treated sample than the annealed sample. For instance, it can be observed from figures 3 and 4 that the corrosion rate of the un- treated sample ranged between 0.25 mm/yr – 0.58 mm/yr in the 0.5M to 1.5M acid concentration and temperature conditions (30 to 60°C) whereas it (corrosion rate) ranged from 0.13 mm/yr to 0.36 mm/yr for the annealed sample for the same concentration and temperature conditions. This gives a significant corrosion reduction. Evaluation of empirical relationship between corrosion rate and acid temperature is presented in figure 5.

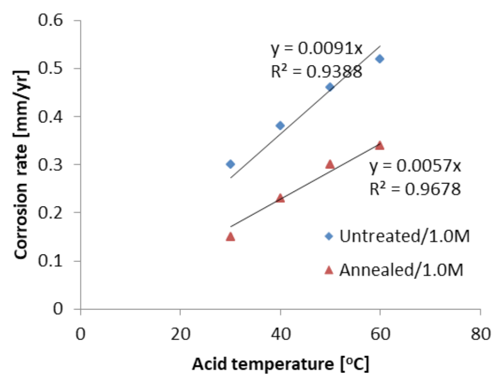

Figure 5: Empirical relationship between corrosion rate and acid temperature.

It can be deduced from regressional analysis (Figure 5) of experimental data that the corrosion rate (CR), of the AISI 304 in

1.0M HCl solution varies with acid temperature (T) according to the following empirical equations:

CR = KT [3]

Provided that the temperature of the medium does not exceed 60 °C. Where K is a kinetic parameter that expresses the change of corrosion rate with temperature. K depends on the corroding material (composition, microstructure and manufacturing) and environment (concentration, temperature and pH). K has been evaluated to be equal to 0.009 and 0.005 for the un- treated and annealed stainless steels respectively. From the best fit line and the coefficient of determination (R^2) that fits the experimental data, it can be seen that the empirical equation (equation 3) is able to describe the relationship between corrosion rate and medium temperature for AISI 304 stainless steel corroding in 1.0M HCl and heated to up to 60 °C. Higher corrosion resistance of the annealed samples is probably because during soaking at annealing temperature, greater proportion of alloying additions and inclusions are dissolved in the matrix along with other secondary phases and these in turn increases the corrosion resistance of the alloy [22].

Conclusions

This study investigated the effect solution annealing on the corrosion resistance of AISI austinitic stainless steel in 0.5- 1.5M hydrochloric acid solution and at varying medium temperature of 30- 60 °C. The following conclusions can be drawn: Annealed stainless steel showed up to 36% better corrosion resistance in the hydrochloric acid solutions than the untreated alloy. The corrosion rate of the austenitic stainless steel increases with the concentration of hydrochloric acid and with the temperature of the acid.

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