

Experimental Analysis of Corrosive Impact of Oxidizing and Non-oxidizing Environment on Sensitized Welded and Unwelded Samples of AISI 316

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Abstract: An experimental analysis was carried out to investigate the corrosivity of sensitized welded and unwelded austenitic stainless steel AISI 316, in oxidizing (H₂SO₄) and non-oxidizing (HCL) media. The selected samples were cut into several equal pieces. To induce sensitization, the samples were heated and soaked at 750°C at different soaking time intervals such as 30 minutes, 60 minutes, 180minutes, 300 minutes and 600 minutes followed by water quenching. The sensitized welded and unwelded samples each were subjected to immersion duration test in the oxidizing and non-oxidizing media for 5, 10, 15, 20, 25, 30, 35, 40, 45, and 50 minutes, respectively. It was concluded from the results obtained that corrosion rate of welded and unwelded decreases as soaking time and immersion duration increases at constant soaking temperature, in non-oxidizing medium of hydroChloric acid (HCL); but the decrease in corrosion rate is more in the unwelded samples. In the oxidizing medium of sulphuric acid (H₂SO₄); corrosion rate of the samples decreased as immersion duration/soaking time increases. However, the welded samples experienced erratic behaviour in the oxidizing medium, having a lower corrosion rate than their unwelded counterpart surprisingly, at immersion duration between 25-50 minutes as soaking time increases at constant soaking temperature of 750°C.

Keywords: Austenitic Stainless Steel AISI 316, Corrosion Rate, Sensitization, Immersion Duration, Oxidizing and Non-oxidizing Media, Soaking Temperature, Soaking Time, Welded and Unwelded

1. Introduction

Corrosion of materials [2] is the most common and most important failure mechanism in industry. Stainless steels retain its special qualities in structures because of their strength, stiffness, toughness and tolerance at high temperatures [3]. Their usage in Catalytic Reformer Unit, Reactor Scallop, Vacuum Distillation Unit, Heat Tubes at Port Harcourt Refinery; process and utility areas of Mobil producing Nigeria unlimited and Chevron Escravos gas to liquid plants all in Nigeria, is a demonstration of their importance [1]. They are widely used in harsh oil and gas production environments. Austenitic stainless steel is widely used in caustic environments [4-9].

These steels usually in welded form to achieve the desired design, have been observed to experience various debilitating forms of localized corrosion like pitting, crevice and stress corrosion cracking [10-16], when exposed to chloride containing solution environments; leading to loss of millions of dollars each year [17, 18]. Although one of the main reasons why stainless steels are used is corrosion resistance; they do suffer from certain types of corrosion in some environments; for instance, austenitic stainless steel is sensitive to chloride-induced stress-corrosion cracking (SCC) [19, 20]. Therefore, care must be taken to select a grade which will be suitable for certain type of application [21]. Time, temperature and environment therefore, significantly influences this unusual complex/sensitized microstructure of

the steels and hence its properties [22].

The harsh acidic environment at the sensitized regions can be too aggressive to allow passivity to be maintained by; being too reducing, in some acidic media (e.g. Hydrochloric acid); so that passivating species cannot form [23] and being too oxidizing (e.g. Hot dilute nitric acid or H₂SO₄) and thus make oxidized species that normally affect passivity unstable. Weld decay has been observed to be common in sensitized stainless steel structures [24].

These unavoidable circumstances results in heterogeneous microstructure with small amount of delta ferrite and significant segregation of major as well as minor alloying elements at the phase interfaces. This steel alloys usually have many constituent elements and many thermodynamically possible phases, complicated with the facts that none of these possible phases are necessarily uniform in their composition. Interestingly, it is the composition of the alloy in contact with the specific environment at any microscopic point that determines the corrosion resistance of that particular point. It is not unusual therefore, that one can find out erratic behaviour of some alloys, especially at the steel weldment in a particular environment; the alloy displaying both resistance and susceptibility to corrosive attack.

This research was set out to study the influence of environments on the corrosivity of sensitized welded and unwelded stainless steels {AISI 316}; to assist in addressing some of the worrisome and disturbing cases despite numerous researches. The inferences from the experimental results could assist in establishing a relationship on various factors (oxidizing and non-oxidizing environment, alloy composition, microstructure, Temperature, immersion Time, etc). This understanding would assist in mitigating the

menace of corrosion in a susceptible environment as low as reasonably practicable.

2. Methodology

2.1. Materials

AISI 316 stainless steel was selected for this investigation. The sample is a commercial pure sample and was available in form of pipes and was collected from the ware house of Port Harcourt Refinery Company Ltd, Alese Eleme, Port Harcourt, Rivers State-Nigeria.

2.2. Equipments

Equipments used for the study included the following: Welding machines, Grinding machines, Cutting machines, Universal milling machine, Manual hand Hacksaw with blade, heat treatment furnace, Oxford instrument XRF spectrometer model X-Met 7000 with a serial number 711150, Crown technology inc. CT 1000 corrosion meter [capable of reading corrosion rate], Epoxy Resin Ring, Emery Paper.

2.3. Experimentation

2.3.1. Chemical Composition

The chemical composition of the selected welded and unwelded steel samples were determined at Turret Engineering Services Ltd., Port Harcourt using an Oxford instrument XRF spectrometer model X-Met 7000 with serial number 711150. The detailed chemical compositions of the samples are as shown in figures I and II below.

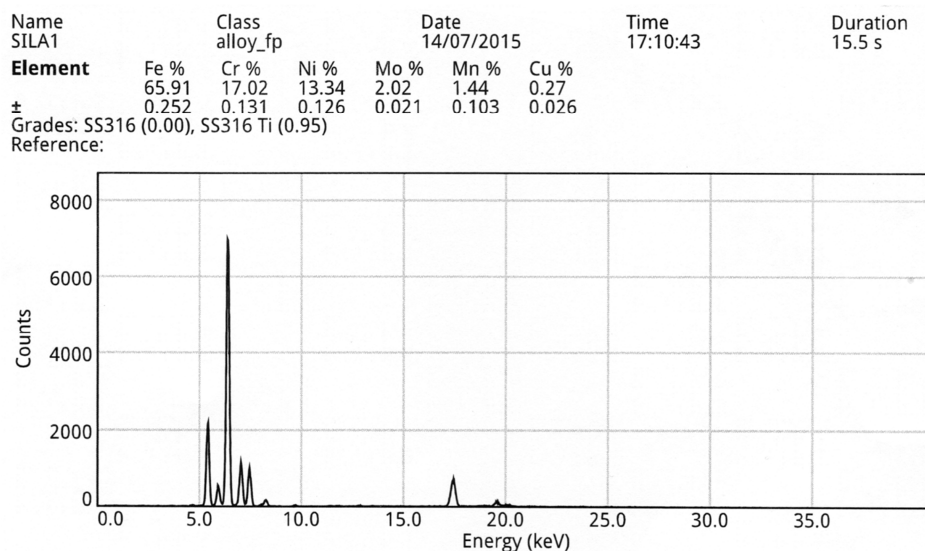


Figure 1. Chemical composition of Austenitic Stainless Steel AISI 316 (Unwelded sample).

Name	Class			Date	Time			Duration
SILA1 W	alloy_fp			14/07/2015	17:12:12			15.5 s
Element	Fe %	Cr %	Ni %	Mo %	Mn %	Cu %	Co %	Ti %
±	65.69	17.41	12.57	2.13	1.62	0.30	0.17	0.10
	0.240	0.126	0.116	0.020	0.101	0.023	0.040	0.033

Grades: SS316 (0.00), SS316 Ti (0.69)
Reference:

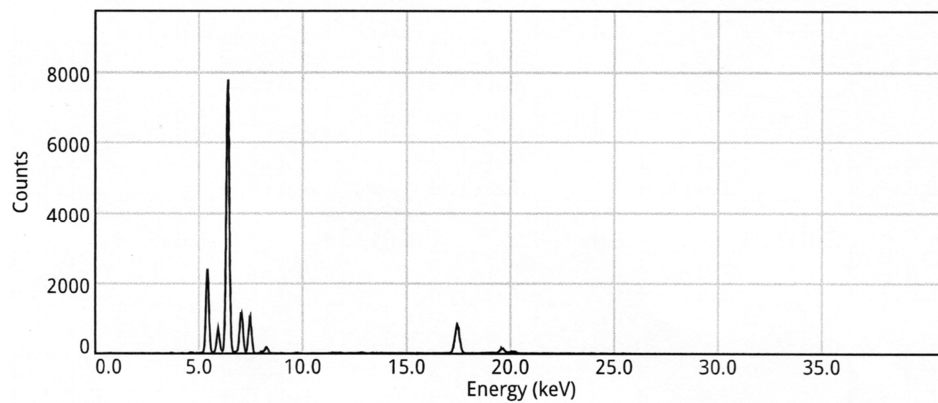


Figure 2. Chemical composition of Austenitic Stainless Steel (AISI 316) Weldment.

2.3.2. Heat Treatment

The chosen grade of stainless steel was procured and welded; following all necessary Global Practices/standards and code requirements. The weldment and the unwelded samples were cut to sizes and subjected to heat treatment using solution annealing for 1 hour at 1000°C, followed by quenching in water to dissolve the precipitate phases, if any.

The samples were then prepared by cutting into several equal pieces. To induce sensitization, the samples were soaking at 750°C at different time intervals such as 30

minutes, 60 minutes, 180 minutes, 300 minutes, 500 minutes, 400 minutes and 600 minutes followed by water quenching. This type of heat treatment schedule by varying the holding times was suggested by Ghosh et al.

Consequently, corrosion study was carried out on the prepared samples from the weldment and the unwelded samples of the sensitized steels; by immersion at different time intervals in corrosive media {HCL=non-oxidizing and H₂SO₄=oxidizing {H₂SO₄}. The tabulated results and graphical representations/discussions are as shown in below.

3. Results/Graphical Representations

Table 1. The results and their graphical representations are as shown below {Tables 1-6 and Figures 3-8}. Corrosion rate(COR) of unwelded AISI 316 samples{1A, 2A, 3A, 4A, 5A, 6A, 7A}, soaked at 750°C at variable soaking time of 30, 60, 180, 300, 400, 500 and 600 minutes respectively and immersed in non-oxidizing medium (HCL) at variable immersion duration.

Immersion duration (Minutes)	1A COR (mpy)	2A COR (mpy)	3A COR (mpy)	4A COR (mpy)	5A COR (mpy)	6A COR (mpy)	7A COR (mpy)
5	3.60	3.53	3.20	19.45	9.50	7.74	7.72
10	3.09	3.30	3.17	7.90	6.98	6.95	3.61
15	4.41	2.86	2.86	2.72	3.45	3.06	3.05
20	3.33	2.69	2.90	4.04	3.54	3.19	3.57
25	2.78	3.09	3.32	3.13	3.33	3.14	3.04
30	2.77	2.82	3.10	4.60	1.28	4.28	3.45
35	2.73	2.63	2.97	3.44	3.88	2.03	2.29
40	2.88	2.71	3.06	2.94	2.29	2.57	2.20
45	2.80	2.73	2.54	2.55	2.77	3.24	3.71
50	2.39	2.56	2.66	3.28	2.63	2.10	2.14

Table 2. Corrosion rate(COR) of welded AISI 316 samples{1AW, 2AW, 3AW, 4AW, 5AW, 6AW, 7AW}, soaked at 750°C at variable soaking time of 30, 60, 180, 300, 400, 500 and 600 minutes respectively and immersed in non-oxidizing medium (HCL) at variable immersion duration.

Immersion duration (Minutes)	1AW COR (mpy)	2AW COR (mpy)	3AW COR (mpy)	4AW COR (mpy)	5AW COR (mpy)	6AW COR (mpy)	7AW COR (mpy)
5	9.51	16.17	9.69	7.70	4.97	5.47	5.13
10	8.56	9.03	8.92	4.98	5.17	5.33	5.20
15	7.79	8.07	9.17	5.55	5.00	5.17	5.44
20	8.27	8.33	7.27	5.19	5.03	4.97	4.74
25	7.31	7.36	8.31	4.56	4.70	4.43	2.12
30	6.91	7.23	6.41	4.45	4.72	4.96	4.11
35	6.50	5.00	6.55	3.75	3.75	3.69	3.72
40	5.92	6.60	5.98	3.61	3.43	3.55	3.50
45	5.52	5.47	5.41	3.36	3.28	3.42	3.33
50	5.19	4.88	4.63	3.30	3.27	3.56	3.59

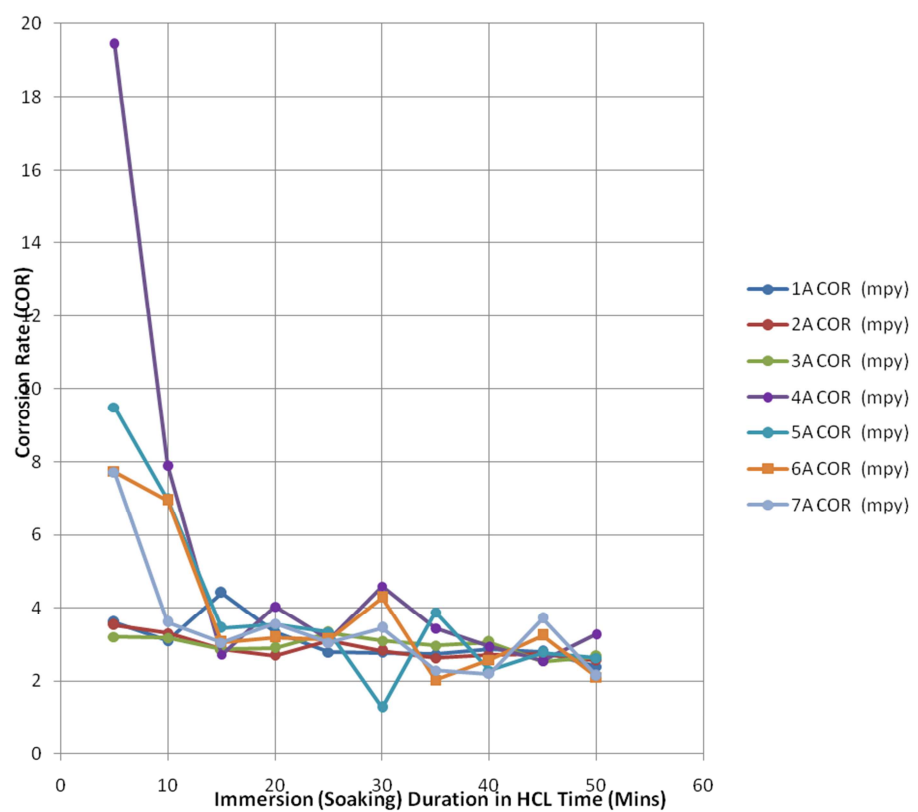


Figure 3. Graphical relationship of immersion duration in non-oxidizing medium (HCL) versus corrosion rate (COR) for various samples in table 1.

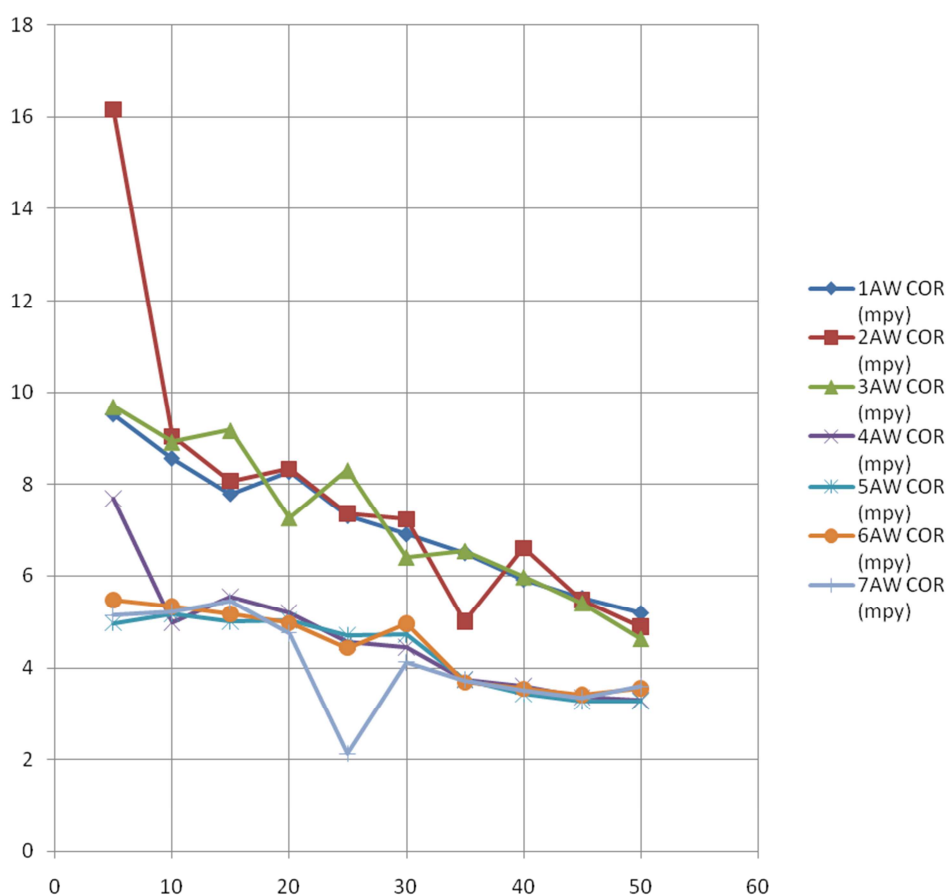


Figure 4. Graphical relationship of immersion duration in non-oxidizing medium (HCL) versus corrosion rate (COR) for various samples in table 2.

Table 3. Corrosion rate (COR) of unwelded AISI 316 samples {8A, 9A, 10A, 11A, 12A, 13A, 14A}, soaked at 750°C at variable soaking time of 30, 60, 180, 300, 400, 500 and 600 minutes respectively and immersed in oxidizing medium {H₂SO₄} at variable immersion duration.

Immersion duration (Minutes)	8A COR (mpy)	9A COR (mpy)	10A COR (mpy)	11A COR (mpy)	12A COR (mpy)	13A COR (mpy)	14A COR (mpy)
5	2.87	1.82	1.80	11.00	9.12	8.18	7.97
10	3.43	2.37	2.90	7.37	8.14	7.17	6.93
15	2.46	2.71	2.45	7.40	6.75	6.61	6.11
20	2.26	2.45	2.99	1.26	0.97	0.91	3.91
25	3.74	2.90	2.68	1.96	2.47	2.31	2.12
30	3.45	2.26	2.41	0.72	0.84	0.26	1.09
35	2.16	2.53	2.50	1.46	1.15	0.55	0.63
40	2.58	2.32	2.61	0.76	0.77	0.70	0.73
45	2.73	2.22	2.17	0.54	0.49	0.43	0.68
50	2.58	2.48	2.15	0.53	0.41	2.87	2.96

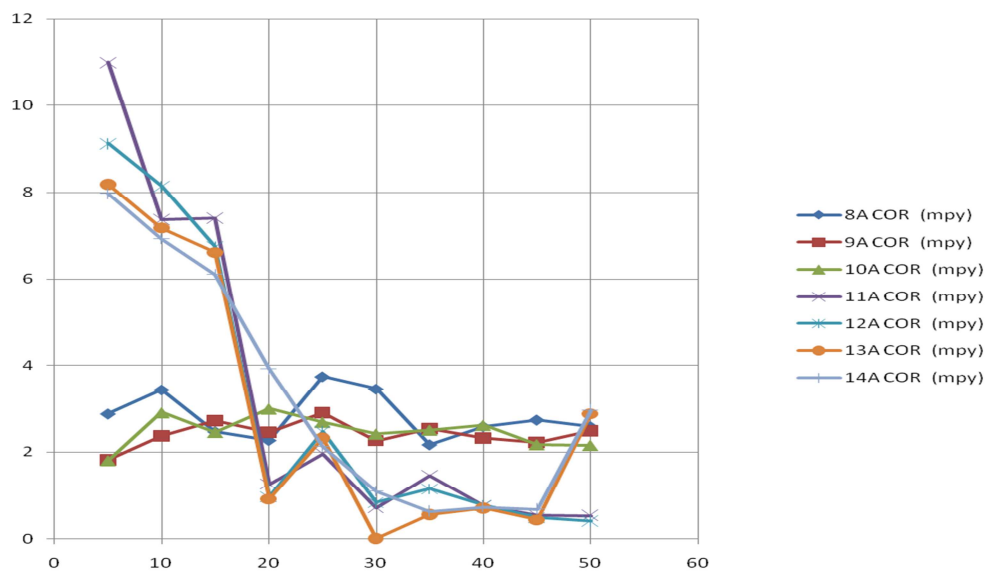


Figure 5. Graphical relationship of immersion duration in oxidizing medium {H₂SO₄} versus corrosion rate (COR) for various samples in Table 3.

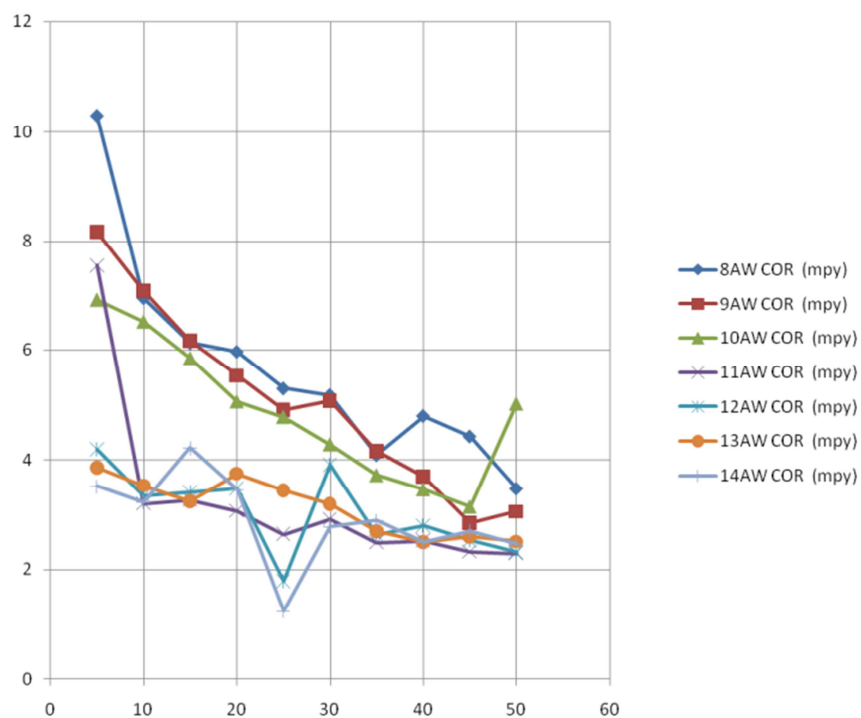


Figure 6. Graphical relationship of immersion duration in oxidizing medium (H₂SO₄) versus corrosion rate (COR) for various samples in table 4.

Table 4. Corrosion rate (COR) of unwelded AISI 316 samples {8AW, 9AW, 10AW, 11AW, 12AW, 13AW, 14AW}, soaked at 750°C at variable soaking time of 30, 60, 180, 300, 400, 500 and 600 minutes respectively and immersed in oxidizing medium {H₂SO₄} at variable immersion duration.

Immersion duration (Minutes)	8AW COR (mpy)	9AW COR (mpy)	10AW COR (mpy)	11AW COR (mpy)	12AW COR (mpy)	13AW COR (mpy)	14AW COR (mpy)
5	10.29	8.16	6.92	7.55	4.19	3.87	3.52
10	6.95	7.08	6.52	3.20	3.34	3.54	3.23
15	6.13	6.17	5.85	3.27	3.41	3.25	4.22
20	5.97	5.53	5.06	3.07	3.48	3.76	3.46
25	5.30	4.91	4.78	2.64	1.79	3.45	1.23
30	5.18	5.07	4.28	2.92	3.91	3.20	2.78
35	4.09	4.16	3.72	2.49	2.64	2.70	2.89
40	4.80	3.70	3.46	2.53	2.80	2.50	2.50
45	4.43	2.85	3.14	2.32	2.53	2.60	2.70
50	3.48	3.06	5.02	2.29	2.32	2.51	2.46

Table 5. Average corrosion rate of AISI 316 samples of unwelded {1A, 2A, 3A, 4A, 5A, 6A, 7A} and welded {1AW, 2AW, 3AW, 4AW, 5AW, 6AW, 7AW}, soaked at 750°C at variable soaking time of 30, 60, 180, 300, 400, 500 and 600 minutes respectively and immersed in non-oxidizing medium (HCL) at variable immersion duration.

Unwelded 1A-7A		Welded 1AW-7AW	
Immersion duration (Minutes)	COR (mpy) {Average}	Immersion(Soaking) duration in HCL Time (Mins)	COR (mpy) {Average}
5	7.82	5	8.38
10	5.00	10	6.78
15	3.20	15	6.60
20	3.32	20	6.26
25	3.12	25	5.54
30	3.19	30	5.54
35	2.85	35	4.17
40	2.66	40	4.66
45	2.91	45	4.26
50	2.54	50	4.06

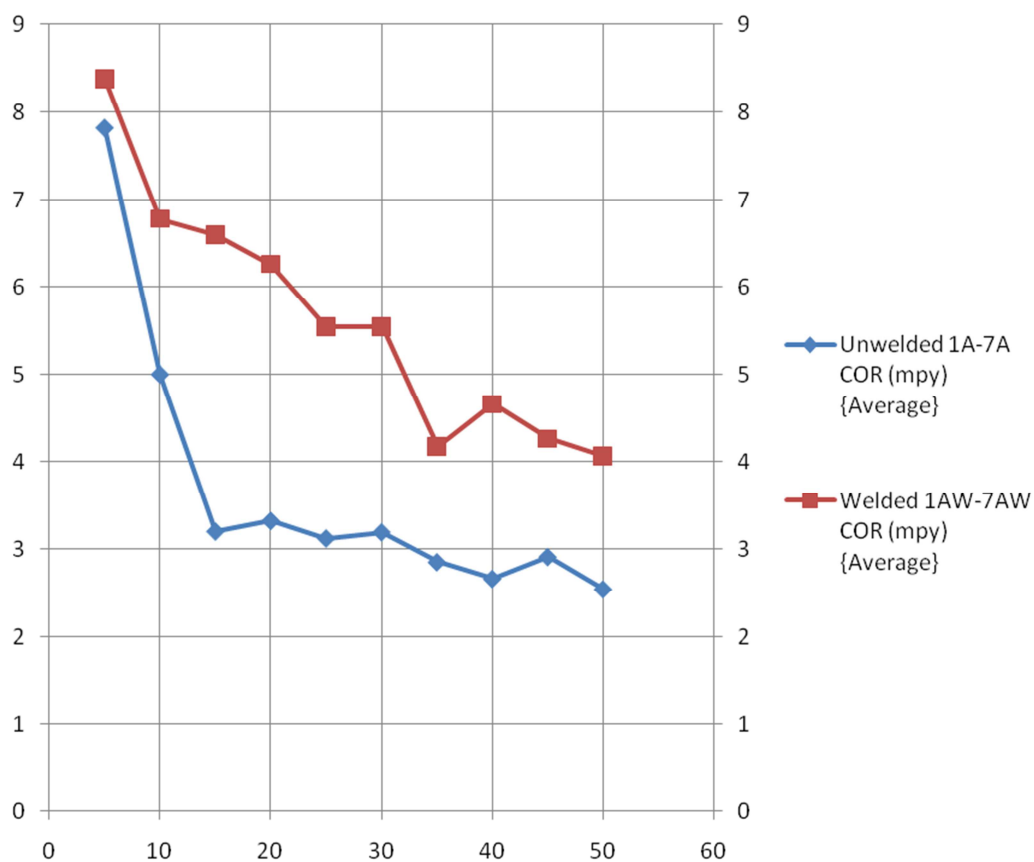


Figure 7. Graphical relationship of immersion duration in non-oxidizing medium (HCL) versus average corrosion rate (COR) for various samples of welded and unwelded as shown in table 5.

Table 6. Average corrosion rate of AISI 316 samples of unwelded {8A, 9A, 10A, 11A, 12A, 13A, 14A} and welded {8AW, 9AW, 10AW, 11AW, 12AW, 13AW, 14AW}, soaked at 750°C at variable soaking time of 30, 60, 180, 300, 400, 500 and 600 minutes respectively and immersed in oxidizing medium (H₂SO₄) at variable immersion duration.

Unwelded (8A-14A)		Welded (8AW-14AW)	
Immersion (Soaking) duration in {H ₂ SO ₄ } Time (Mins)	COR (mpy) {Average}	Immersion (Soaking) duration in {H ₂ SO ₄ } Time (Mins)	COR (mpy) {Average}
5	6.11	5	6.36
10	5.47	10	4.84
15	5.00	15	4.61
20	2.11	20	27.36
25	2.60	25	3.44
30	1.68	30	3.91
35	1.57	35	3.24
40	1.50	40	3.18
45	1.32	45	2.94
50	1.20	50	3.02

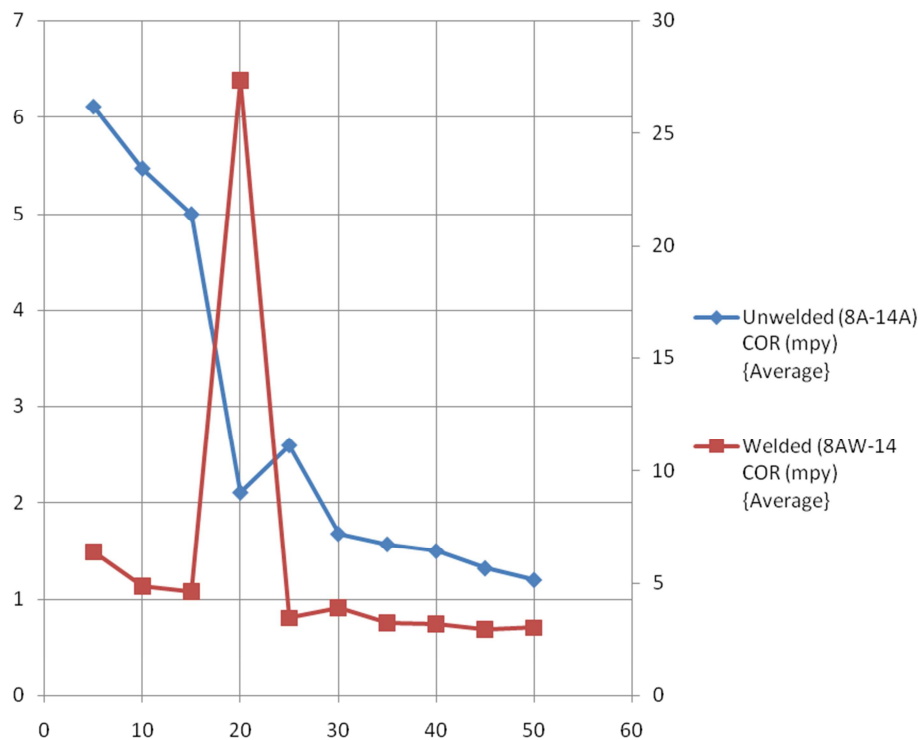


Figure 8. Graphical relationship of immersion duration in oxidizing medium {H₂SO₄} versus average corrosion rate (COR) for various samples of welded and unwelded as shown in table 6.

4. Discussions

From table 1 and figure 3 above, sample 4A has the highest corrosion rate within five minutes of immersion duration in non-oxidizing medium (HCL); but decreased sharply to 2.72mpy as immersion time increased up to 15 minutes; then fluctuates gradually as immersion time increases. Corrosion rate of 4A, 5A, 6A, 7A have an initial sharp decreases; but fluctuates gradually at lower corrosion rate as immersion duration increases. 5A has the lowest corrosion rate, while 4A has the highest. Careful study showed that corrosion rate of 2A, 3A and 5A are more stable and decreases at increasing soaking time and immersion duration in HCL. One thing is common in all the samples; the

resistance to corrosion increases and sensitivity to fluctuation in corrosion rate decreases as immersion duration/ soaking time is increased. This could be attributed to the positive recovery in the sensitized steel [25-26].

From table 2 and figure 4 above, sample 2AW has the highest corrosion rate within five minutes of immersion time in non-oxidizing agent (HCL); but decreased sharply as immersion time increases up to 10 minutes; then gradually decreases as immersion time increases. Careful study of the table 2 and the figure 2 below showed that sample 7A become erratic at lower corrosion rate, between 20 and 30 minutes of immersion time; decreasing and increasing sharply at lower corrosion rate. Also corrosion rates of samples 4A, 5A, 6A, 7A are smaller than the corrosion rates of 1A, 2A and 3A, at all immersion duration, but

significantly corrosion rates of samples 4A, 5A, 6A stably decreases as immersion duration increases. From the table and the graph, one thing is common; corrosion rate tend to decreases with increasing immersion duration /soaking time in the non-oxidizing agent.

From table 3 and figure 5 above, samples 11A, 12A, 13A and 14A have very sharp drop in corrosion rate (from very high to very low corrosion rate), as immersion duration in oxidizing agent (H_2SO_4) increased up to 30minutes of immersion duration. Samples 8A, 9A, 10A have relatively initial lower corrosion rate than 11A, 12A, 13A and 14A in the oxidizing agent (H_2SO_4), but fluctuate at higher corrosion rate, as immersion duration/soaking time increases.

Careful study of the table 3 and the figure 3, showed that corrosion rate of samples 11A, 12A, have lower corrosion rate than samples 8A, 9A, 10A, as immersion duration is increased between 20-50 minutes. Sample 13A has the lowest corrosion rate among the samples at 30minutes. Also, samples 13A and 14A have lower corrosion rate than 8A, 9A, 10A between 20-40minutes of immersion duration; but tend to arise sharply as immersion duration increase.

From table 4 and figure 6 above, the corrosion rates of samples 12AW, 13AW and 14AW, fluctuates at a lower corrosion rate than samples 8AW, 9AW and 10AW. Sample 11AW had sharp corrosion rate decrease, from 7.55mpy to 3.2mpy within the first 5minutes of immersion duration in H_2SO_4 . Samples 11AW, 12AW 13AW and 14AW fluctuates at a lower corrosion rate than samples 8AW, 9AW and 10AW between 10-50 minutes of immersion duration in H_2SO_4 . Careful study of the table 4 and figure 4 showed that Samples 8AW, 9AW and 10AW, tend to decrease steadily as immersion duration/soaking increases, but at a higher corrosion rate than 11AW, 12AW 13AW and 14AW.

Careful study of table 5 and figure 7 above, showed that average corrosion rate of the unwelded samples 1A-7A and welded samples 1AW-7AW, decreases with increase in immersion duration and soaking time in the non-oxidizing agent of HCL; but the rate of decrease is higher in the unwelded samples than that of the welded samples, as immersion duration and soaking time increases.

Careful study of table 6 and figure 8 above showed that average corrosion rate of the unwelded samples 8A-14A decreases with increase in immersion duration /soaking time in the oxidizing agent (H_2SO_4). However, erratic behaviour is observed in the welded samples 8AW-14AW. The average corrosion rate of the samples rose sharply in the oxidizing agent (H_2SO_4), between 15 and 20 minutes of immersion duration; and decreased sharply almost at the same rate within additional 5 minutes; but generally has a lower corrosion rate than the unwelded samples between 25-50 minutes of immersion duration and variable increasing soaking time.

5. Conclusions

From the results and graphical analysis, the following may be concluded:

- a) Corrosion rates of unwelded and welded samples of AISI 316 decreases as soaking time and immersion duration increases in non-oxidizing medium of HCL.
- b) What must have happened? Thermodynamically, when the steels were subjected to intermediate homogenous temperature [27, 28] of $750^\circ C$, at variable increasing soaking time, recovery may have occurred, from possible presence of Cr-rich alpha prime (α') precipitates/formation [24, 29], a key material degradation properties that limits its upper service temperature and general mechanical properties.
- c) The rate of corrosion rate decrease in hydrochloric acid (HCL) medium of the unwelded samples of AISI 316 with increase in immersion duration/ soaking time is greater than that of their welded counterparts at the same constant soaking temperature.
- d) The unequilibrium heat cycles, which resulted in complex reactions/phase transformation of the steel (AISI 316), during welding activities sensitized the weldment differently which significantly resulted in different corrosive impacts.
- e) Similarly, the unwelded and welded samples of AISI 316 subjected to variable soaking rate and immersion duration in oxidizing medium (H_2SO_4); have one common characteristic with their corrosion rate decreasing as soaking time and immersion duration increases at constant soaking temperature.
- f) However, the welded samples of AISI 316 in oxidizing medium of H_2SO_4 , experienced erratic behaviour at immersion duration between 15 and 25 minutes. The corrosion rate increased sharply between 15 and 20 minutes of immersion duration and sharply decreased approximately at the same rate within additional 5minutes of immersion duration. Between 25-50minutes, the corrosion rate of welded samples of AISI 316 is lower in the oxidizing medium (H_2SO_4) than that of their unwelded counterpart at the same immersion duration and soaking time.
- g) Finally, the differences in sensitization of the steel weldment and the unwelded counterpart significantly affected their reaction in oxidizing and non-oxidizing media which led to the differences in their corrosion rates. Recovery during soaking time significantly contributed to the improved corrosion resistance of the steel in both oxidizing and non oxidizing environment.

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