INVESTIGATION OF CORROSION BEHAVIORS OF A SUPER DUPLEX STAINLESS STEEL 2507 WELD IN CHLORIDE SOLUTION

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Abstract

In this paper work, its cover the important factors of material and environmental conditions on decomposition of super duplex stainless steels in chloridecontent were studied. Variations in the SDSS structural changes by heat treatment and cyclic heat treatmentsat 600°C were investigated in terms of deviations in the proportion of austenite and ferrite segments, phase morphology and intermetallic precipitation using SEM. Alloy structure and chemical composition of weldment and annealed weldment in chloride solution composition was also found to affect the corrosion performance and passivation of SDSS. Corrosion rates and susceptibility of DSS was observed with addition of chloride solutions.The result obtained after these experiments will have an important influence in relations of recognizing the composite structures, manufacturemethods, microstructures, and environmental situations that may be help to reduce to mitigate decomposition of SDSS in chloride solutions on weld regions.

Keywords: *Super Duplex steel 2507, Scanning Electrode Microscope, Filler material, Cyclic Heat Treatment , GTA welding, Pitting corrosion.*

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I. INTRODUCTION

Today there is additional concept accessible, austenitic-ferritic or, more normally, duplex stainless steel. Duplex stainless steel was established around 75 years ago, but the austenite-ferrite equilibrium was far from good in those days and manufacture was further an art than science. Duplex stainless steels, originally produced as low-nickel substitutes to austenitic stainless steels in uses wherever decomposition resistance, strength, and joining capability are of excessive concern, are resulting in the improvement procedure in the production of critical procedure constituents (e.g., welded pipes, pumps, etc.) in many manufacturing industries to includes the pulp and paper industry, offshore-gas and petroleum industry and chemical industry in wide-ranging [1-7]. In specific cases, super duplex stainless steels (SDSS) such as AISI 2507, with higher chromium percentage (25%) when compared with traditional 22% Cr duplex steels, are resulting more widespread application in destructive eroding environmental conditions, where characteristic functioning conditions can comprise of high levels of chloride attack with effective temperatures which is more than 50 °C

In the Modern world of industrialization the wear is eating the assets worth millions of dollars per year. The wear is in the form of corrosion, erosion, abrasion etc. which occur in the process industries like oil & gas, refineries, Cement plants, steel plants and dockyards. The combination of selective alloying components (Cr, Mo, N and Ni) helping the co-occurrence of ferrite and austenite phrase in equivalent quantities, has credited to this class of steels revealing advanced strength than austenitic SS, developed toughness than ferritic stainless steels, good welding ability, and more advanced corrosion behaviour in a range of destructive environmental conditions, that containing pitting, intergranular, and stress corrosion cracking [7].Super Duplex Stainless Steels 2507 are alloys which contains twin microstructure with same amount fraction of austenitic and ferrite phase content. However, these stainless steels also undergo many corrosion deformations in aggressive environment such like chloride solutions.Although corrosion behaviors of duplex stainless steels in chloride solution has been studied and well recognized in the works but the comparison of corrosion mechanisms for SDSS in base material and weld regions after annealing and cyclic heat treatment were not known. Microstructural variations through manufacture procedures affect the general corrosion exposure of these steels in Chloride solutions. Further environmental influences, like pH of the solution, temperature, and causing electrochemical potential also influence the corrosion susceptibility of super duplex stainless steels.

The variations in the SDSS structural changes by heat treatment and cyclic heat treatments were investigated in terms of deviations in the proportion of austenite and ferrite segments, phase morphology and intermetallic precipitation using image analysis, optical microscope and scanning electron microscopes. These specimens were then verified for overall and restricted corrosion exposure to recognize the fundamental mechanisms of corrosion occurred in SDSS in the chloride situations.

This experiments demonstrations that microstructural variations in super duplex SS due to heat behaviors could affect their corrosion exposure. Heat treatmentsof weldment region of this stainless steel samplesare conducted on 600°Cand water quenched samples were done to be resistant to chloride in chloride environment. Cyclic heat treatments at 600°C are done on weldment and heat treated of this steel sample. Alloy structure and chemical composition of base material, weldment and heat treated weldment in chloride solution composition was also found to affect the corrosion performance and passivation of SDSS. Corrosion rates and susceptibility of DSS was observed with addition of chloride solutions.

The result obtained after these experiments will have an important influence in relations of recognizing the composite structures, manufacturemethods, microstructures, and environmental situations that may be help to reduce to mitigate decomposition of SDSS in chloride solutions on weld regions as well as the base material specimen. The complete outcomes from this experimental works will facilitate in considerate the comparison of microstructure, heat treatment and mechanism of decompositionin chloride content on the weldment and annealed weldment of base material.

II. EXPERIMENTAL PROCEDURE

To determine the heat treatment and corrosion behavior susceptibility of SDSSs in chloride solutions, the following investigational processes were carried out.Before starting welding procedure it is essential to clean surface of base steel and groove edges, So as to remove the oil, dust ,rust and other unwanted particles can be removed properly otherwise these particles may lead to some types of defects in weld metal.The joint design used in this work is a single V groove which is selected according to the thickness of the base material. The groove angle will be approx 45°, root gap of 1 mm and root face of 1 mm is going to be used.

Table 1: Chemical Composition of SDSS grades used in this Analysis

Steel Grade	Composition (wt. Percent)									
Common Trade Name	UNS Number	C	Mn	S	Ni	Cr	Mo	N	Cu	Fe
2507	S32570	0.026	0.73	0.38	6.43	24.190	3.15	0.3	-	Balance

Before welding, the base metal plates were cut into130 mm × 50 mm × 4 mm dimensions.The samples were welded together by two permits with GTAW.After achieving the root pass two main weld passes are made with filler i.e. Duplex 2507 to fill the groove volume.The plates are cut into small pieces of 12 plates of required dimensions (40mm*30mm*4mm) from the welded samples where the samples are not affected by heat of welding. The plates are then shaped and grinded for make into required specimens for heat treatment.

Table 2: Parameters used for the welding the base steel material

Welding Parameters	
Current (I) (amps)	150
Voltage (V) (volts)	11.6
No of passes	1
Average Welding Speed (U) (mm/sec)	0.325
Heat input (kJ/mm)	1.05
Shielding gas	99.9% pure argon gas

The 12 samples are categorized into 4 set of 3 samples and base material recorded as 1 set of 3samples.In each specimen-code set, 1, 2 and 3 such A1, A2 and A3 represents 3 samples each are used for heat treatment and corrosion testing respectively.

The heat treatment temperatures for S32570 were 600°C. Heat treatment included exposing the 9 samples coded set (B, C and D) to the annealing temperature of about 600°C for one hour followed by air quenching. After conducting annealing treatment on 9 samples, cyclic heat treatments, i.e. 2^{nd} time of heat treatment were given to the 6 heat treated samples (C and D) at 600°C (for 3 hrs) followed by air quenching. Then the 3 samples coded set D are again heat treated for the 3^{rd} cyclic of heat treatment at same the temperature (600°C) for 3 hrs. Samples after heat-treated and cyclic heat treated (reheating) as well as base material and welded specimens without heat treat are sophisticated and etched in 40% NaOH solution to make known the ferrite/austenite segments.Micrographs were taken by means of aSEM.

After cleaning, the set of samples which are treated with or without heat are exposed to corrosion or exposure treatment in chloride environment medium.The pitting corrosion or exposure tests in the chloride environment are implement according to ASTM G48 standard where the chloride environment are carried out by reagent grade MgCl (Brine Salt) in water. The samples are implemented in chloride medium at temperature of 50°C (higher temperature at which corrosion occur on SDSS pipes) for a time period of 96 hrs.

At the end of corrosion test, the 4 sets of SDSS samples (A, B,C, and D) are washed and evaluated for the visual analysis of corrosion in the samples. After that, the samples are cleaned and polished for the measurement of weight and surface analysis.Cracks and pits may occur in the side and surface of the samples are the evaluated and analyzed by using Scanning Electrode Microscope (SEM).The change in the super DSSs microstructure are due to the outcome of heat treatment used in the investigation, made the steel samples sensitive to corrosion are analyzed by using SEM.The comparison of the microstructural variation and difference on the SDSS 2507 samples due to the heat treatment and pitting corrosion can be examined by SEM to evaluate about the initial crack occurred on the samples.

III. RESULT AND DISCUSSION

ASTM G48 TEST

The result of the pitting corrosion test is obtained the TABLE 3 which summarizes the corrosion treatments done to the SDSSs set of samples and the variation in the weight loss occurred on the welded part of samples with heat input will provide the rate of pitting corrosion.The graph obtained from the variation of weight loss due the pitting corrosion occurred on the 4 set of samples (Set A, B, C and D) with change in the heat input for 3 days, i.e. for about 72 hours is the result of pitting corrosion rate of SDSS weldment samples. The variation of pitting corrosion rate happened in the 4 set of samples is shown in the Figure 1.

It can be seen from the graph of pitting corrosion rate that loss of weight of samples are found to be increased with the increasing heat input per day. At higher value of heat input, the weld region of SDSSs sample accomplish sensitive range of temperature which lead to the formation of intermetallic phase segment such as secondary austenite occurs easily. Chromium (Cr) and Molybdenum (Mo) which are in very low content are present more in the secondary austenite. Therefore, these areas are the sites for pitting corrosion attack due to easy interruption of the passive film. According the ASTM G48 test, the constant pitting occurred or initiated when the reduction of weight is more than 1 g/(m^2.day).

For all 4 sets (A, B, C and D) of SDSS welded samples used for pitting corrosion test at the initially stage of the test, there is no evidence or indication of pitting as the loss of weight is less than 1 g/ (m^2.day). But there are evidence of pits and loss of weight in the 4 set (A, B, C and D) of sample weldment. The weight loss of sample weldment is more than 1 g/ (m^2.day) with time (day) and increase in the heat input.

Table 3: Pitting Corrosion Test result for SDSSs weldment

Heat input (Kj/mm)	Weight loss due to corrosion per day [g/(m^2.day)]			
	Set A	Set B	Set C	Set D
0.95	1.128	1.146	1.139	1.149
1.05	1.549	2.246	2.186	2.167
1.15	2.128	3.731	3.925	4.021

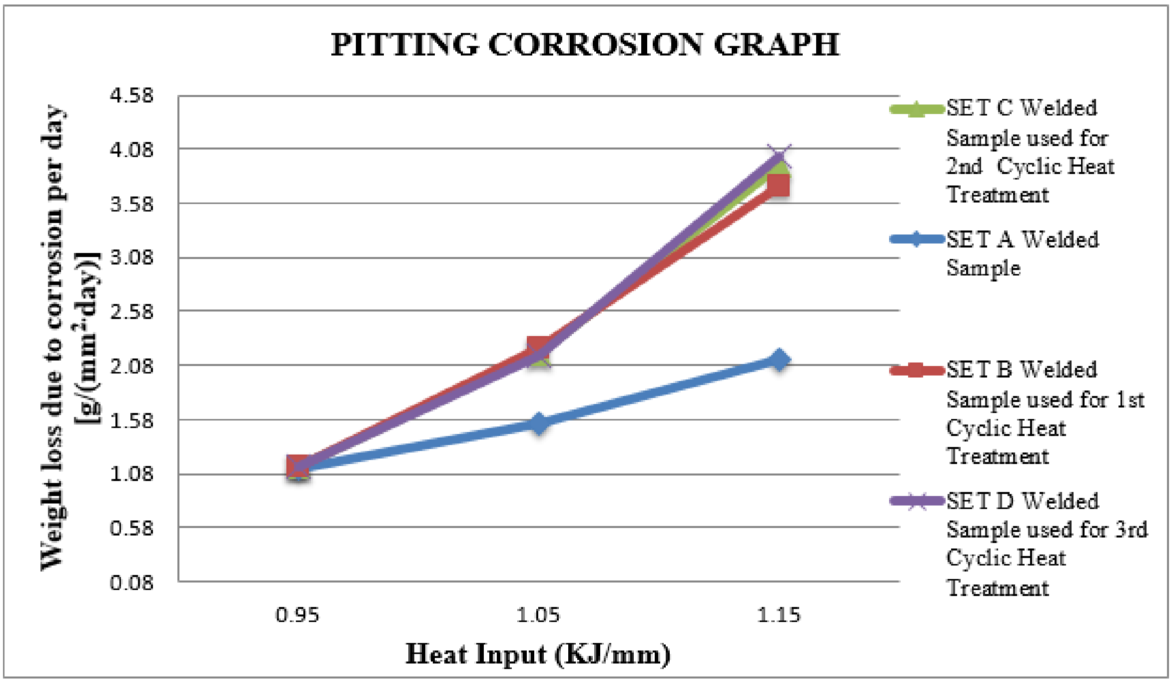


Figure 2: Variation of weight loss due to pitting corrosion with variable heat input.

The Set A (Welded Sample) is having less loss of weight due to corrosion per day compared to other sets (B, C and D). Set D (Welded Sample used for 3rd Cyclic Heat Treatment) sample is having more weight loss with heat input. The corrosion rate occurred on the Set D is more than other set of samples. The pitting corrosion rates of weldment in sets of sample are more than due to the loss of weight due to corrosion per day with heat input. The Set C (Welded Sample used for 2nd Cyclic Heat Treatment) is having more corrosion rate than Set B and Set A. This is large variation in the weight loss of Set A sample as compared to other sets of sample. Set B , Set C and Set D are having less variation in the loss of weight and hence in the corrosion rat occurred on the samples. There is very less different in the total average weight loss of Set B and Set C. The average corrosion rate of set of samples after the pitting corrosion test at 72 hrs is shown below.

3.2301 g/(m^2.day) > 2.4167g/(m^2.day) > 2.3743g/(m^2.day) >1.6017g/(m^2.day)

Set D > Set C > Set B > Set A

PITTING RESISTANCE NUMBER OF SDSS WELDMENT

The pitting corrosion resistance of SDSSs in chloride containing solution environment depends mainly on Nitrogen (N), Molybdenum (Mo) and Chromium (Cr) fraction. Hence, the decomposition corrosion resistance is correlated with the chemical composition of SDSSs and can be expressed in term of an empirical pitting resistance equivalent number (PREN) value is calculated by:

$$PREN = wt.\ \%\ Cr + 3.3\ wt.\ \%\ Mo + x\ wt.\ \%\ N$$

Where, the range of x value from 16 to 30.

When the PREN value is large, then it will give rise to more pitting resistance. InIndustrial sector,the value for x is normally take to be 16, while the researchers commonly use the value of x to be 20. This equation is taking into consideration the beneficial effect of Nitrogen (N), Molybdenum (Mo) and Chromium (Cr) only. However, rest of elements such as P, S andMn that shows deleterious result on the pitting opposition are ignored. Mo and Cr are partitioning in ferrite segment and of N and Ni in austenite segment have impact on the PREN values of both phases. Furthermore, the precipitation of secondary phase particle can cause compositional changes in the alpha (α) and gamma (γ) phases which results in the selective pitting corrosion of weak phases [25].

For Super Duplex Stainless Steel base material, the pitting corrosion resistance number (PREN) is

$$PREN = 25 + 3.3 * 4 + 20 * 0.030$$

$$= 25 + 13.2 + 0.6$$

$$= 38.8 \sim 39$$

If the value of x is 20, wt % of Cr is 25, wt % of Mo is 4 and that of N is 0.030, then the PREN value of the standard SDSSs sample is approximately 35 to 40. In SDSS base material sample, the PREN value is nearly to 39. That means the sample is having higher pitting corrosion resistance.

For Super Duplex Stainless Steel which is used as base material in this thesis work, the pitting corrosion resistance number is

$$PREN = 24.190 + 3.3 * 3.150 + 20 * 0.028$$

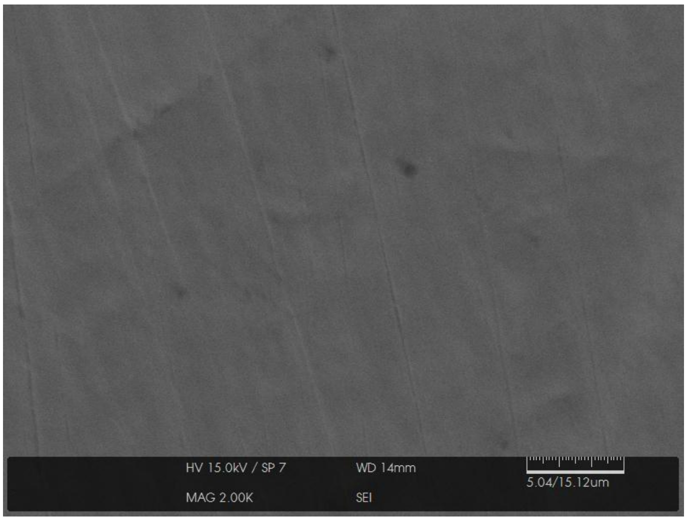
$$= 24.190 + 10.395 + 0.56$$

$$= 35.147 \sim 36$$

In this SDSS base material used in the thesis work is having the pitting resistance equivalent number to be 36 which comparatively less than the general super DSS material but it is in between the range of PREN value.

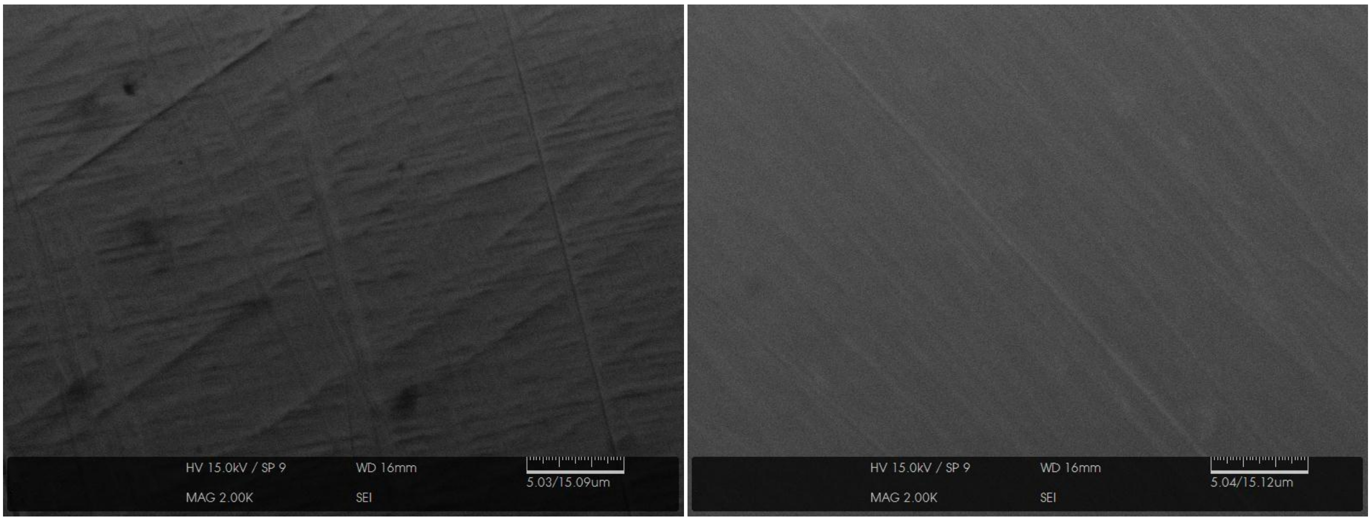
PIT MORPHOLOGY

In order to make sure the same surface situation are used, each specimens of all sets (A, B, C and D) are used for shaping and grinding the surface of the samples before the pitting corrosion ASTM G48 test. In this current thesis work, surface microstructures and morphology image of different sets of welded samples with cyclic heat treatment and welded sample were obtained by Scanning Electron Microscope (SEM). The SEM morphological images of all 4 set of samples (A, B, C and D) are obtainable in this thesis work. However, all set of samples had almost the same type of surface morphological image through the observation. Figure show the surface morphology image of all 4 set of sample (A, B, C and D) which are used in the experimental work before pitting corrosion test. It shows the magnified view of the polished weldment coupon surface. No significant corrosion damage is seen on the surface.



Set A

Set B



Set C Set D

Figure 2: SEM morphologies of all 4 set of samples before pitting corrosion test.

As is shown in this Figure 2, cross wise marks and scratches make happen by grinding can be observed noticeably, but there is no evidence of pit appears are presented on the external surface of the weldment of all 4 set of samples (A, B, C and D). However after the pitting corrosion test, there are a small number of pits appear existing on the surface of the weldment of all samples. Besides this, the scratches are become more evidently clear. It is theoretical that the places wherever scratches localise are more expected to decompose and corrode due to the presence of stress concentration. But the verification further about corrosion needs much more in future to survey about the size and number of corrosion pit.

After the completion of pitting corrosion test, the 4 set of SDSS samples are cleaned and etched in the solution to make the recognition of change in the microstructure phases. Cracks and pits may occur in the side and surface of the samples are the evaluated and analysed by using Scanning Electrode Microscope (SEM).

The SEM morphologies of Set A which welded sample without any heat treatment on the welded part of the samples after pitting corrosion are shown in the Figure 3. From the morphology of the Welded sample (Set A), the high magnification SEM images shows that there are staining or rusting effect on the polished welded region of coupon sample.

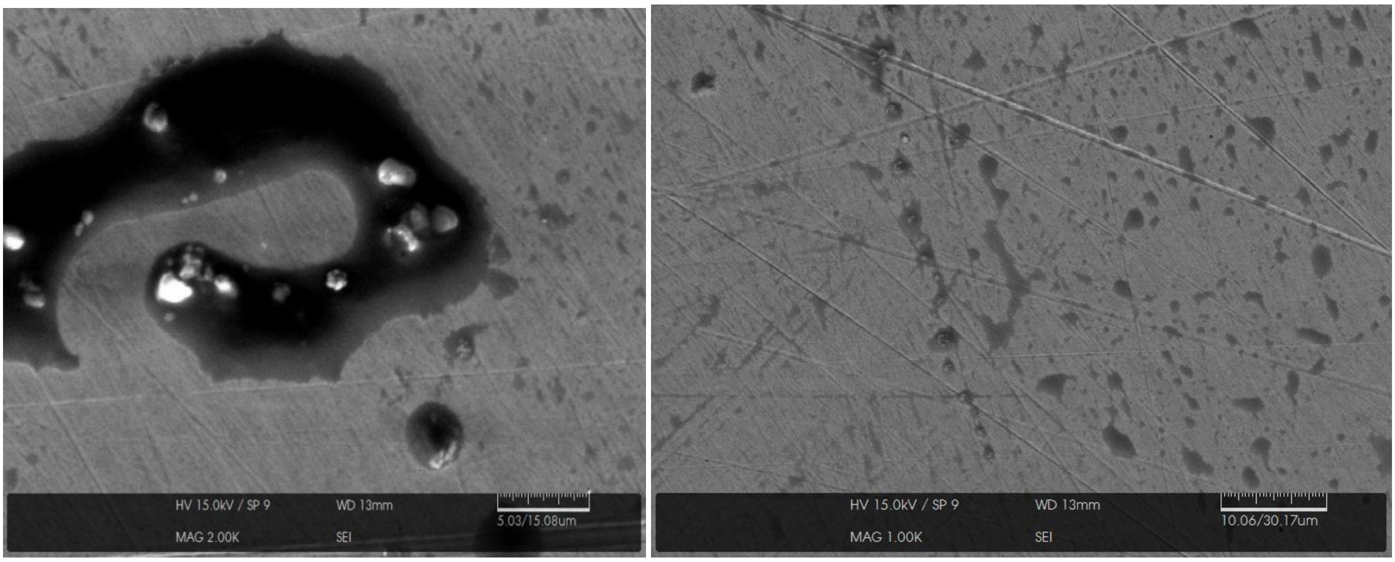


Figure 3: SEM morphologies of welded coupon sample without heat treatment (Set A) after Pitting Corrosion Test at different region of welded area

The Set B in which welded sample used for 1st cyclic heat treatment after pitting corrosion analysed by using SEM and are shown in the Figure 4. From the morphology, High magnification SEM images shows that there are shows shallow pitting damage on the polished welded coupon sample. Little deep pits are present in the surface of welded region of Set B.

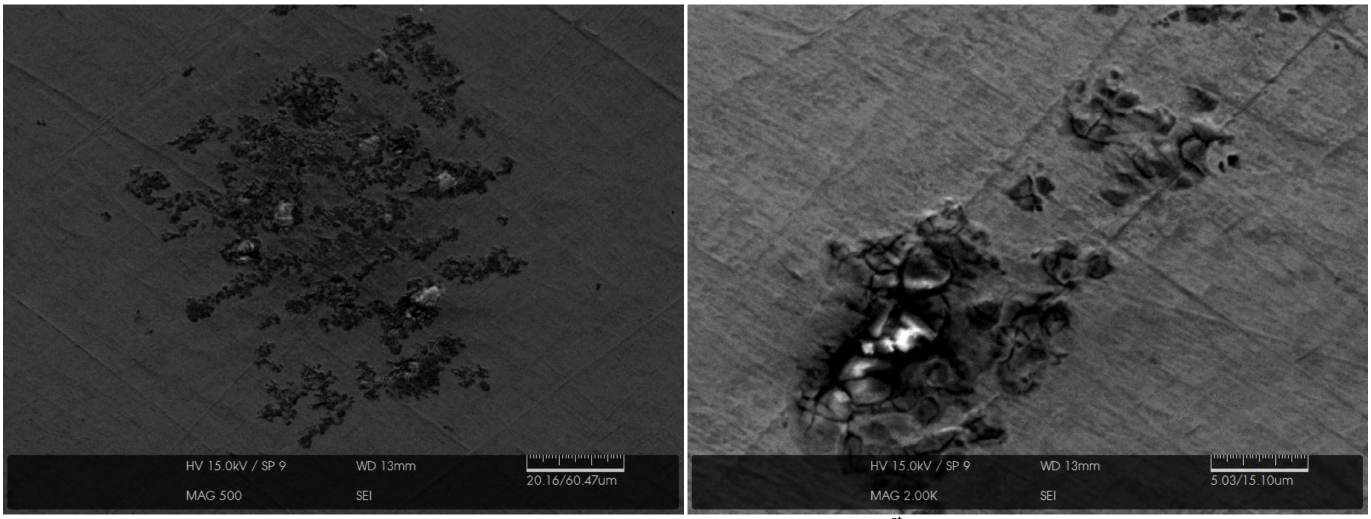


Figure 4: SEM morphologies of welded regions of welded coupon sample with 1st cyclic heat treatment (Set B) after Pitting Corrosion Test.

The SEM morphologies of Set C, which welded sample with 2nd heat treatment on the welded part of the samples after pitting corrosion, are shown in the Figure 5. From the morphology of the Welded sample with 2nd heat treatment (Set C), the high magnification SEM images shows that there are scattered shallow pitting damages on the surface of welded regions of the polished coupon sample. There are deep scattering pits which having only a short distance pits from the top to the bottom. The pits are scattered pieces with randomness.

Figure 5: SEM morphologies of certain region of welded coupon sample with 2nd cyclic heat treatment (Set C) after Pitting Corrosion Test.

The Set D in which welded sample used for 3rd cyclic heat treatment after pitting corrosion analysed by using SEM and are shown in the Figure 6. From the morphology, High magnification SEM images shows that there are large presents of scattered shallow pitting damage on the welded region of polished welded coupon sample. More depth pits are occurred on the surface of welded region of Set D. There are random occurrences of pits in large quantity on the surface of the weld region.

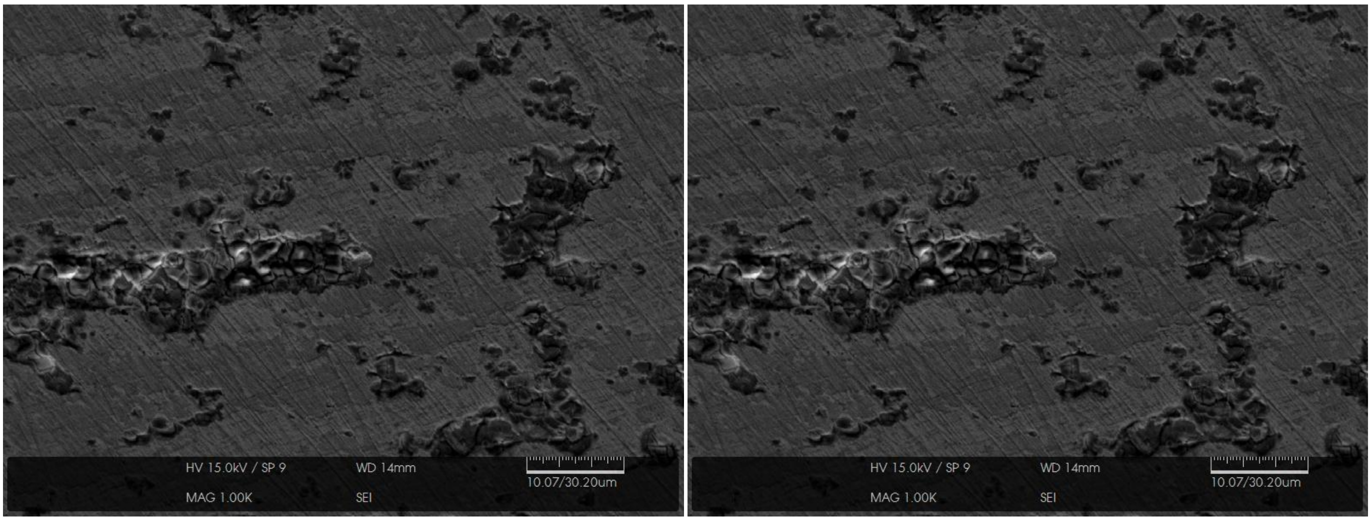


Figure 6: SEM morphologies of various region ofwelded part on welded coupon sample with 3rd cyclic heat treatment (Set D) after Pitting Corrosion Test.

IV. CONCLUSION

The main objective behind this research work was to examine the consequence on structure, composition, and environmental factors like solution composition and high temperature on the corrosion of super DSSs in chloride containing brine solutions. Ultimate motivations of this experimental works are to study the essential mechanisms required for the pitting corrosion in this system. In this chapter, main outcomes after these studies have been shortened and deliberated together and a mechanism of pitting corrosion in chloride solution has been recommended.

Based on the outcomes of the investigational part, it is likely to conclude the following:

1. After the cyclic heat treatment of all 4 set of samples (A, B, C and D), the samples which are used of cyclic heat treatment and sample without heat treatment are used for SEM analysis and the result obtained from the analysis shows thatcross wise marks and scratches make happen by grinding can be observed noticeably, but there is no evidence of pit appears are presented on the external surface of the weldment of all 4 set of samples (A, B, C and D).
2. Pitting corrosion test of ASTM G48 methodwas successfully done to illustratethe presence of pits and crackon the weld regions of all 4 set of 2507 grade SDSS samples.The graph obtained fromthe variation of weight loss due the pitting corrosion occurred on the 4 set of samples (Set A, B, C and D) with change in the heat input for 3 days from the second day of pitting corrosion test method, i.e. for about 72 hours is the result of pitting corrosion rate of SDSS weldment samples. The graph of pitting corrosion rate shows that losses of weight of samples are found to be increased with the increasing heat input per day. At higher value of heat input, the weld region of SDSSs sample accomplish sensitive range of temperature which lead to the formation of intermetallic phase segment such as secondary austenite occurs easily.
3. The Set A (Welded Sample) is having less loss of weight due to corrosion per day compared to other sets (B, C and D). Set D (Welded Sample used for 3rd Cyclic Heat Treatment) sample is having more weight loss with heat input. The corrosion rate occurred on the Set D is more than other set of samples. The pitting corrosion rates of weldment in sets of sample are more than due to the loss of weight due to corrosion per day with heat input.
4. In the SDSS base material used in the thesis work is having the pitting resistance equivalent number to be 36 which comparatively less than the general super DSS material but it is in between the range of PREN value ranging from 35 to 40.
5. From the SEM and optical morphology, the high magnification SEM images of Set A (Welded sample without heat treatment) shows that there are staining or rusting effect on the polished welded region of coupon sample, Set B shows that shallow pitting damage on the polished welded coupon sample, Set C and Set D shows that there are scattered shallow pitting damages on the surface of welded regions of the polished coupon sample. More depth pits and random occurrences of pits in large quantity are occurred on the surface of welded region of Set D.
6. GTA welding was accomplished successfully on the 2507 grade SDSS plates with thickness of 4mm. The effect of welding process such as heat input at 1.05 and appropriate temperature, PREN value, cyclic heat treatment and cooling rate on the weld region microstructure and pitting corrosion resistance were analysed. Based on the research and test outcomes, improved of corrosion resistance are increasing with the increase the number of cycle of heat treatment, faster cooling rate, heat input and temperature about 50^{o}C. Due the appropriate inter pass temperature, current , heat input (0.75-1.1) kj/mm and filler material, the rate of corrosion is lesser in the weld material.

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