CHARACTERISATION OF 9% Cr-1% Mo FERRITIC STEEL WELDMENTS FOR SUSCEPTIBILITY TO CORROSION DURING HYDROGEN CHARGING AND REPAIR-WELDING

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ABSTRACT

Weldments of 9%Cr-1%Mo steel may become prone to accelerated localized corrosion attack if inadequately post-weld heat treated. The phenomenon is mechanistically identical to that observed in austenitic stainless steels where Cr-depleted regions along the grain boundaries are attacked in preference to the grain interiors. This paper describes a systematic study carried out to investigate the influence of initial microstructure, heat input, PWHT, accelerated ageing and repair-welding on the sensitization behaviour of 9%Cr-1%Mo steel weldments. It was found that repair-welding without PWHT resulted in the sensitization of the weld metal and base metal heat affected zones. Ageing of such repaired weldments led to the sensitization of both weld metal and HAZ. This study has also established the increased susceptibility of the sensitized regions to hydrogen induced cracking thus indicating that the region is microstructurally weaker, where cracking due to environment may initiate preferentially.

INTRODUCTION

In petroleum, chemical processing and fossil fuel fired power generating industries, 9%Cr-1%Mo steel finds extensive application. This variety of steel also finds application in the evaporators and super heaters for the advanced gas cooled nuclear reactors and is a candidate material for boiler/super heater circuit in liquid metal cooled fast breeder reactors. It has also been selected as the construction material for tubing in the reheater and super heater portions and as a thick section tube plate material in the steam generator of a 500 MWe Prototype fast breeder reactor which is proposed to be built at Kalpakkam. This material is also being considered as the construction material for sodium-air decay heat exchanger which will be used during emergency shut downs. In the normalized and tempered condition, the material exhibits high corrosion resistance, low propensity to carburization-decarburization in liquid sodium,

relatively high proof stress, ductility and good creep rupture strength. It is also readily weldable provided recommended welding conditions and proper post-weld heat treatment (PWHT) are followed. Delayed cracking, hot cracking and reheat cracking are some of the problems associated with the welding of these steels. In addition, sensitization and hence susceptibility to intergranular corrosion (IGC) has also been observed in these steels, if proper PWHT is not performed. Sensitization in Cr-Mo ferritic steel is defined as the change in microstructure leading to further lowering of chromium in certain regions of the heat-affected zone (HAZ) resulting in greater sensitivity to corrosion attack. Intergranular oxide penetration in caustic environment and poor stress corrosion cracking resistance of as-welded and inadequately post-weld heat treated welds have been attributed to the sensitization of weldments^{1,2} However, only limited information is available in literature regarding the sensitization arising due to welding of these steels. This is probably because of the fact that in airhardenable 9%Cr-1%Mo steel chromium depletion due to chromium carbide precipitation is not expected as chromium diffusion in martensite/ ferrite matrix is high. Inadequate PWHT, insufficient tempering and rapid thermal transients at and above 750°C are reported to be some of the situations leading to sensitization due to chromium depletion around chromium carbide precipitates34 This phenomenon has assumed great importance and needs to be examined thoroughly, because, the PWHT either may not be possible or may adversely affect the other desirable mechanical properties⁴. If improper PWHT is one of the causes of sensitization, the above repair-welding practice may lead to sensitization and subsequent exposure to service environment may result in IGC. As this phenomenon, though observed by several investigators, has not been properly under-

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stood in its entirety, a detailed investigation was carried out on 9%Cr-1%Mo steel to document the influence of (i) initial microstructure, (ii) heat-input, (iii) PWHT, (iv) accelerated ageing and (v) repair-welding on the sensitization behaviour.

The presently operating and planned fast breeder reactors in India are situated in the coastal areas; therefore, during various stages of fabrication, storage, time between installation and commissioning and during unplanned and unexpected shut downs, the components are exposed to marine atmosphere at ambient temperature which could result in corrosion damage. When corrosion takes place, the important cathodic reaction is hydrogen evolution. Hydrogen absorbed by ferritic steels frequently produces degradation of mechanical properties and facilities sub-critical crack growth. In this investigation Hydrogen Induced cracking (HIC) propensity of sensitized region was also examined by a potentiostatic technique.

Experimental

The material used in this investigation was a nuclear grade 9%Cr-1%Mo steel forging of a tube sheet (1000mm diameter and 300mm thick). The steel contains, in wt%, C-0.10%, Si-0.75%; Cr-9.27%; Mo-1.05%: Mn-0.63%; P-0.02%; S-0.001%; N-190 ppm; Ni-0.12%: Cu-0.10%; Fe-balance). The thick section forging had been subjected to austenitising at 950°C for 5h followed by quenching in water. The tempering heat treatment was carried out at 750°C for 8h followed by air-cooling. The material in the above heat treated condition is referred to as 'as-received'.

(i) Heat Treatment

From this material, two plates (250 x 150 x 10mm) were cut and the fol-

lowing heat treatments were carried out in order to study the influence of initial microstructure on the sensitization behaviour :

(i) 950°C-1h, ice quenched [IQ]

(ii) **950°C** C-1h, air-cooled + 750°C-1h, air-cooled [N&T]

[Henceforth, the samples subjected to the first heat treatment will be referred as 'IQ' and those to the second as 'N&T'].

To understand the influence of heat input on the sensitization, the IQ plates and N&T plates were subjected to autogenous GTA welding and SMA welding with matching consumable with heat inputs varying from 0.38 to 1.8 kJ/mm. A multipass weld also prepared with a heat input of 0.83 kJ/mm from the N&T plates. Table-I presents a summary of the welding conditions which were used to prepare the weld pads. All the 13 weld pads were subjected to PWHT at 750°C for 1h followed by air-cooling. The post weld heat treated pads were then subjected to accelerated ageing (to correspond to ageing at 500°C for 10 years) by exposing at 600°C for 9h. The multipass weldment was also subjected to accelerated ageing (to correspond to ageing at 500°C for 5, 10 and 20 years) by exposing at 600°C for 4.5h, 9h and 18h.

Table-I Summary of the Welding Parameters

Initial Microstructure	Welding Process	Heat input, kJ/mm
IQ	Single pass (autogenous GTAW)	0.48, 1.3, 1.8
N&T	Single pass (autogenous GTAW)	0.48, 1.3, 1.8
IQ	Single pass (SMAW)	0.38, 0.45, 0.66
N&T	Single pass (SMAW)	0.38, 0.45. 0.66
N&T	Multipass (SMAW)	0.83

Table-II Details of the Welding Conditions Used for Repair-Welding

Initial Heat Input in SMAW	Heat Input during repair-welding by GTAW
kJ/mm	kJ/mm
0.38	1.8
0.45	0.9
0.66	1.6
0.38	1.8
0.45	1.9
0.66	1.6
	Initial Heat Input in SMAW kJ/mm 0.38 0.45 0.66 0.38 0.45 0.45 0.66

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In order to simulate the effect of repair-welding on the properly PWHTed and service exposed samples, the SMA weldments (single pass) were subjected to repair-welding by overlapping another weld bead using autogenous GTAW for which the welding conditions are presented in **Table-II**.

The effect of ageing on repair-welded (without PWHT) samples was also investigated. The repair welded pads were subjected to accelerated ageing at 600°C for 9h (to correspond to ageing at 500°C for 10 years).

After every heat treatment viz. (i) PWHT, (ii) accelerated ageing, (iii) repair-welding, (iv) ageing after repair-welding, representative samples were cut for corrosion studies.

It has been reported that quenched and tempered microstructure of this steel is representative of certain region of HAZ of welds produced in N&T samples⁵. Hence, in order to simulate the thermal cycles experienced at the HAZ, samples of dimension 10 x 10 x 10mm were austenitised at 950°C for 1h, icequenched and then tempered for various durations ranging from 1 minute to 30 minutes.

(II) Specimen Preparation for Corrosion Studies

From the representative weld pads, weldments of 40 x 15 x 10mm size were sectioned for corrosion studies, these weldments and the samples which were given weld simulation heat treatments were mounted in epoxy resin with a brass rod in which M3 threading were made. The details of the mounting procedure have been described elsewhere⁶. The samples were polished up to a fine diamond finish for metallographic observation and electrochemical studies. In order to characterize the



Fig. 1. Anodic polarisation curves for 9%Cr-1%Mo steel in 16 wt.% H2SO4 solution

initial microstructure, samples were prepared both from the IQ and N&T sheets.

(III) Scanning Electron Microscopic Studies

The IQ and N&T samples were etched with Vilella's reagent (1g Picric acid + 5ml HCl + 100ml ethyl alcohol) for 15 seconds. In order to improve the contrast of the microstructure, the samples were gold coated by vapour deposition technique. Microstructure was characterised by conventional secondary electron imaging using Philips SEM (Model PSEM 501)

(IV) Potentiostatic Etching

In our earlier investigation, detailed potentiodynamic and potentiostatic experiments were carried out on weld-simulated samples (austeniti sed, ice quenched and tempered for various durations ranging from 1 to 30 minutes)⁷. The salient feature of the results are collectively presented in **Fig.1&2**. From the potentiody namic polarisation curves in 16 wt% H2SO4 (Fig. 1), it can be clearly seen that at + 100 mV (SCE), the sensitized sample (eg. austenitised. ice-quenched and tempered for 1 minute) containing chromium depleted zone dissolves at a rate which is several orders of magnitude higher than the non-sensitized sample (eq. austenitised and ice-quenched or tempered for longer durations) Therefore potentiostatic etching at + 100 mV (SCE) in this medium (Fig.2) will result in preferential dissolution of chromium depleted zones and thus sensitized zones can be clearly revealed in the weldments. Therefore, all the weldment samples (aswelded, post-weld heat treated, thermally aged, repair-welded. repairwelded and aged) were subjected to potentiostatic etching under the above conditions for 5 minutes. using Wenking potentiostat Model STP 84. These weldments were examined by optical and scanning electron microscope after potentio static etching for documenting the types of attack.



Fig. 2. Effect of tempering time on the current transients at constant potentials

(V) Surface Profilometric Studies

The potentiostatically etched weldments were examined with SLOAN DEKTAK (Model 3030) surface profilometer and the cross section was examined in each case Surface profilometric measurements were performed at a stylus pressure of 20 N/m2 to determine the width and depth of the HAZ next to the fusion line.

(VI) Hydrogen Charging by Potentiostatic Technique

All the weldment samples were charged with hydrogen at-1500 mV (SCE) for 12h in 0.5M H2SO4 containing 200ppm As_2O_3 . After charging, the samples were examined under microscope to study the nature of attack.

(VII) Estimation of Chromium Carbides by Electrochemical Extraction Technique

The weight precentage of chromium carbide in the samples which were

given weld simulation heat treatments was estimated using electrochemical extraction technique. The extraction of the carbides was carried out in a 10% HCl + methanol solution at a potential of 1.5 volts with respect to platinum electrode. By this technique martensite/ferrite dissolved leaving behind a residue containing carbides and undissolved inclusions. The residues were then collected by centrifuging, dried and weighed. The weight percent of carbides was calculated from the weights of the sample dissolved and residue collected.

The residue obtained by the electrochemical extraction process was analyzed by the XRD technique to identify the constituent phases.

Results and Discussion

When 9%Cr-1%Mo steel is austenitised and ice-quenched, complete transformation of austenite to martensite takes place. The asquenched microstructure contains lath martensite. The N&T sample contains tempered martensite and carbides. The scanning electron micrographs showing the initial microstructure of IQ sample and N&T sample are presented in Figs. 3(a and b) respectively.

Potentiostatic etching of the weldments of IQ as well as N&T samples in the as-welded condition, resulted in severe localised attack in the HAZ whereas the attak on weld metal and base metal was minimal, both in the GTA and SMA weldments. The corrosion attack was confined to a narrow band at the base metal-HAZ interface slightly away from the fusion line. The band of the selective attack appeared black to the naked eye and was well delineated from the other zones in the weldment. It was observed that the attack was more severe in the HAZ of weldments of IQ samples than that of N&T samples for each heat input. As the heat input was increased, the band of severe localised attack shifted away from the fusion line. Fig.4 represents the variation of distance of the sensitized zone from the fusion line as a function of heat input. It was noticed that under identical conditions of welding, the 'black' zone in the IQ samples was farther from the fusion line than that in the N&T samples. This indicates that the thermal cycles responsible for causing sensitization in the IQ samples are different from those for N&T samples. The reason for the above difference is not yet fully understood. Figs. 5(a-f) represent the macrographs obtained after potentiostatic etching of the GTA weldments and Figs. 6(a-f) pertain to those of the SMA weldments. Fig. 6(g) represents the macrograph of the multipass SMA weldment in the as-welded condition after potentiostatic etching. Because of the complex thermal cycles experi-



enced by various regions, multiple sensitized zones appeared in the multipass weldment.

Figs. 7(a-c) show the surface profiles taken over the etched weldments of IQ samples. When the surface profilimetric scans were analyzed it was found that in the



Fig. 4 Variation of the distance between fusion line and sensitization zone as a function of heat input (a) IQ, (b) N&T

case of weldments of IQ sample, the black zone showed considerable depth compared to the adjoining weld metal and base metal. The peaks and valleys in the surface profiles in the areas of HAZ indicate that the black zone is porous. Similar runs taken on weldments of N&T samples showed that the depth of attack was much less compared to the IQ samples.

The morphology of the corrosion attack was also studied using SEM. It was found that for weldments of IQ samples, the prior austenite grain boundaries had undergone severe attack which was continuous. The attack within the grain was also guite severe. This can be clearly seen in Figs. 8(a,b). In the case of weldments of N & T samples, both prior austenite grain boundaries and lath boundaries were attacked (Figs. 8(c,d). The attack along the prior austenite grain boundary was continuous whereas the inter-lath attack was discontinuous and highly localised.

It was found that when the weldments were subjected to proper PWHT (750°C-1h, air-cooled), there was no sensitization in the HAZ-base metal boundary. **Figs. 9(a,b)** repre-

sent the macrograph of SMA weldemnts of N & T sample and IQ sample respectively and Fig. 9(c) that of the multipass SMA weldment These results indicate that if proper PWHT is performed, there is no risk of sensitization.

It is well known that to simulate service temperature microstructure by higher temperature heat treatments in 9%Cr-1%Mo steel, Mansion-Mendelson parameter could be used⁸. When the properly PWHTed SMA weldments were heat treated at 600°C for 9h to correspond to 10 years of service at 500°C, there was no sensitization, both in the single pass and multipass weldments of IQ and N&T samples as indicated in **Figs. 10(a,b & c).**

Figs. 11(a & b) represent the macrograph of repair-welded samples of N&T and IQ plates after potentiostatic etching. Since PWHT was not performed after repair-welding, once again sensitization was observed at the HAZ metal boundary. Figs, 12(a & b) show the macrographs obtained after potentiostatic etching of SMA weldments which were aged at 600°C for 9h to correspond to 10 years of service at 500°C Both weld



Fig. 5 Macrographs of GTA weldments after potentiostatic etching at + 100mV (SCE) in 16 wt.% H2SO4 solution (a) IQ-0.43 kJ/mm, (b) IQ-1.3 kJ/mm, (c) IQ-1.8 kJ/mm (d) N&T 0.43 kJ/mm, (e) N&T-1.3 kJ/mm, (f) N&T-1.8 Kj/mm





Fig. 6 Macrographs of SMA weldments after potentiostatic etching at + 100 mV (SCE) in 16 wt.% H2SO4 solution

- (a) IQ-0.38 kJ/mm,
- (b) 1Q-0.45 kJ/mm,
- (c) IQ-0.66 kJ/mm,
- (d) N&T-0.38 kJ/mm,
- (e) N&T-0.45 kJ/mm,
- (f) N&T-0.66 kJ/mm,
- (g) Multipass (0.83 kj/mm)



Figs. 9 Macrographs of properly post weld heat treated SMA weldments after potentiostatic etching (a) N&T-0.66 kJ/mm, (b) IQ-0.66 kJ/mm, (c) N&T-0.83 kJ/mm (multipass)



Figs. 8 Scanning electron micrographs of the sensitized regions of the GTA weldments after potentiostatic etching (a,b) IQ-1.8 Kj/mm, (c,d) N&T-1.3 Kj/mm



Figs. 10 Macrographs of post weld heat treated and aged (equivalent to 500°C/10 years) SMA weldments after potentiostatic etching (a) N&T-0.66 kJ/mm, (b) IQ-0.66 kJ/mm, (c) N&T-0.83 kJ/mm (multipass)



Figs. 11 Macrographs of repair-welded samples (without PWHT) of SMA weldments after potentiostatic etching (a) N&T-0.66 kJ/mm, (b) IQ-0.66 kJ/mm



Figs. 12 Macrographs of repair-welded and aged (equivalent to 500°C/10 years) SMA weldments after potentiostatic etching (a) N&T-0.66 kJ/mm, (b) IQ-0.66 kJ/mm





kJ/mm), (b) N&T (GTAW-1.8 kJ/mm)

Fig. 13 Microhardness profiles (a) IQ (GTAW-1.8 Fig. 14 Hydrogen induced cracking of SMA weldments of IQ samples 0.66 kJ/mm) after charging with hydrogen potentiostatically at - 1500 mV (SCE) in 0.5M H₂SO₄ containing 200 ppm AS₂O

metal and HAZ were found to be sensitized after ageing. In other words, repair-welding (without PWHT) and ageing result in sensitization of both the weld metal and HAZ.

All the above observations can be summarized as follows : when 9%Cr-1%Mo steel weldment is subjected to proper PWHT either after welding or repair-welding, there appears to be no risk of sensitization, Moreover, if the correctly PWHTed sample is service exposed at 500°C, then also sensitization would not be observed in the HAZ up to 20 years of exposure. However, repair-welding without PWHT results in sensitization at HAZ-parent metal boundary and ageing at service temperature leads to sensitization of both the weld metal and HAZ.

Potentiostatic charging of the aswelded samples with hydrogen resulted in different types of attack at different locations depending upon

the strength/hardness. microhardness profile was taken using a load of 10gf and is represented in Figs. 13(a,b) for the GTA weldments of IQ and N&T samples. The sensitized zone is represented by the hatched area. From these plots it is very clear that the sensitized zone of the non-PWHTed welds did not correspond to the region of maximum hardness but corresponds to a region where the gardient in the hardness is maximum. Interestingly, it was also observed that a single sharp hydrogen induced crack was present in the sensitized region whereas hydrogen blisters were observed both at the base metal and weld metal. This is illustrated in Fig. 14. Since the sensitized zone coincides with the zone where the gradient in the mechanical properties is maximum, the risk of HIC is also maximum in that zone in addition to the susceptibility to IGC which arises due to sensitization.

Studies on Weld Simulated Samples

We have reported in our earlier work that insufficiently tempered samples showed instantaneous increase in the current values when potentiostatically kept at + 100 mV (SCE) in 16 wt.% H_sSO₂ solution⁷. When sufficiently tempered, i.e., greater than 3 minutes (Hardness > 250 VPN) such behaviour was not encountered. In potentiodynamic anodic polarisation curves, secondary active peak for insufficiently tempered samples was observed which was much higher than the primary active peak. Flade potential and current density at Flade potential increased with increase in tempering time and reached maximum at 1 minute and 2 minutes respectively and after that started decreasing. It was attributed to the Cr., C, precipitation and concomitant Cr depletion around the precipitate at the initial stages of tempering. Being a BCC matrix, further tempering results in



Fig. 15 variation of wt% of carbides vs tempering time at 750°C **Fig. 16** Diffractograph of the percipitate obtained in electrochemical extraction

quick homogenization. The variation of wt. % of carbide with tempering time confirmed our earlier proposed mechanism. It can be seen from Fig. 15, that as the tempering time increases, there is a sharp increase in the wt.% of carbide. This will result in chromium depletion around the carbide precipitate and hence sensitization. After 3 minutes, there is not much difference in the wt.% of carbides and hence homogenization of chromium by diffusion might have set in. That is why after 3 minutes of tempering, there is no risk of sensitization.

The Diffractograph (Fig. 16) obtained for the residue clearly indicates that the major peaks correspond very well to the $Cr_{23}C_6$ carbides as also given in Table-III.

The microstructure obtained after potentiostatic etching of the weld simulated samples are presented in Fig. 17. The as-quenched sample did not get etched in potentiostatic etching, because there is no carbide precipitation and hence no chromium depletion. When tempered for 1 minute, severe intergranular attack was observed (Fig. 17a) which is almost identical to that shown in Fig. 8a corresponding to the sensitized zone od the non-heat treated weld. As the tempering time increased, the severity of the localised attack along austenite grain boundaries and lath boundaries decreased indicating the on-set of the chromium homogenization of process.

Proposed Mechanism and Practical Significance

The results from this study have unambiguously established that 9%Cr-1%Mo steel weldments undergo sensitization if improper PWHT is given or if insufficiently tempered after normalizing. Certain regions in the HAZ of the weldment exprience thermal cycles which induce rapid chromium carbide precipitation, this leads to chromium depletion in the immediate vicinity of carbide particles. Because of the difference in the chromium content between matrix and depleted zone, passivation potential of these two regions will be different. Therefore, in corrosive media, preferential attack leading to IGC can take place in zones leaner in chromium. If sufficiently tempered, chromium diffusion from matrix to depleted zone may desensitize the HAZ. The practical implication of above result is that if improper PWHT or insufficient tempering is the reason for the sensitization, repair welding of 9%Cr-1%Mo steel without PWHT may be a cause of concern as the sensitized HAZ may become prone to severe IGC during service. Since sensitized zone is the zone where the gradient in the strength/hardness is maximum, HIC propensity is also high in these regions.



Fig. 17 Scanning electron micrographs of weld simulated sample after potentiostatic etching; austenised, ice-quenched and tempered for (a) 1 min, (b) 2 min, (c) 3 min, (d) 4 min, (e) 5 min, (f) 8 min, (g) 15 min, (h) 30 min, (i) 60 min

S.No.	2 0	Intensity %	hkl	d-spacing °A	Reciprocal lattice spacing °A ⁻¹
1	10.9994	9.5	*	8.04379	0.7811
2	18.4971	6.7	*	4.79672	1.3099
3	27.8497	3.9	*	3.20350	1.9614
4	30.4998	3.3	*	2.93092	2.1438
5	33.7476	4.7	*	2.65592	2.3657
6	37.1114	5.5	Cr ₂ N(110)	2.42255	2.5936
7	37.8595	33.7	M ₂₃ C ₆ (420)	2.37638	2.6440
8	40.2618	5.1	Cr ₂ N(002)	2.23996	2.8050
9	40.3485	5.5	•	2.23535	2.8108
10	40.5009	4.8	*	2.22729	2.8210
11	41.5973	35.2	M ₂₃ C ₆ (422)	2.17108	2.8940
12	42.4490	9.8	Cr ₂ N (111)	2.12948	2.9506
13	44.2835	100	M ₂₃ C ₆ (333,511)	2.04543	3.0718
14	48.4476	22.7	$M_{23}C_6$ (440)	1.87891	3.3441
15	50.7 984	22.8	M ₂₃ C ₆ (531)	1.79734	3.4950
16	51.5730	10.3	$M_{23}C_6$ (442)	1.77215	3.5455
17	54.599 5	4.0	M ₂₃ C ₆ (620)	1.68086	3.7381
18	55.87 2 2	3.5	Cr,N (112)	1.64555	3.8183
19	55.9980	3.8	*	1.64215	3.8262
20	56.7804	4.0	*	1.62137	3.8752
21	57.505 7	9.6	M ₂₃ C ₆ (622)	1.60263	3.9205
22	66.8978	4.0	Cr ₂ N (300)	1.39864	4.4924

Table-III Results of the X-ray diffraction studies carried out on the electrochemical extracts of weld-simulated samples Source Wave Length = $1.541038^{\circ}A$

CONCLUSIONS

- During welding of 9%Cr-1%Mo steel, certain regions in the HAZ experienced thermal cycles which lead to chromium carbide precipitation. Chromium depletion surrounding the carbides, results in selective attack in corrosive environments.
- 2. The depth and width of the sensitized zone increases with increase in heat input and it moves away from the fusion line with increase in heat input.
- 3. A properly post-weld heat treated 9%Cr-1%Mo weldment is not susceptible to sensitization. The risk of sensitization is absent even when such a microstructure is exposed to a simulated heat treatment corresponding to extended periods of exposure to serves temperature.
- If the repair-welded samples (without PWHT) are exposed to ageing at service temperature then both weld metal and HAZ become prone to IGC.

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- In the case of multipass weldments, multiple sensitized zones appeared as a result of the complex thermal cycles experienced by the various regions of the weldment
- The sensitized region is more prone to HIC indicating that the region is microstructurally weaker and hence cracking due to environment may initiate preferentially.

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