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Smart assessment of environment assisted cracking of grade 92 material using different test solutions

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Abstract

The Environment Assisted Cracking (EAC) of grade P92 was investigated by employing a new testing method. The main aim is to understand the mechanism of the EAC. The specimen was subjected to a static load of approximately 90% of the material’s yield strength. The experimental work was carried out under the various corrosive environments such as NaOH, KOH, HCl, CuSO₄, and CH₄N₂S test solutions until it gets fractured, which takes a duration of one week (168 h). These specimens were subjected to the Scanning Electron Microscope (SEM) analysis for obtaining its microstructural characterization. Further, the obtained microstructural images can be utilized to understand the failure mechanism. It can be witnessed that the majority of failures appear in the region around the coarse-grained HAZ. Subsequently, the results indicate that the passive path corrosion mechanism seems to be the preferred one as P92 welds are vulnerable to EAC.

1. Introduction

Chromium-molybdenum steels is an attractive material for use in steam piping, header, and boiler applications due to its superior high-temperature strength and excellent creep behavior in the range of 600°C–650°C [1–5]. These steels have been regarded as the potential replacement for the stainless steel in fossil and nuclear power plants because of their unusual high temperature creep strength, high thermal conductivity, low thermal expansion coefficient, high corrosion resistance, and good weldability, high resistance to Stress Corrosion Cracking (SCC) and good oxidation resistance. However, still, there is a high demand to increase efficiency with enhanced operated temperature, which leads to the development of newly creep-resistant steels [6–10]. It has been reported that the stress rupture properties of the common grades of Cr-Mo steels such as 2.25Cr-1Mo, 9Cr-1Mo, and 12Cr can be improved by the addition of tungsten (W), vanadium (V) and niobium (Nb) for their use in high-temperature application [11]. The continuous effort has been taken to achieve better creep strength of 9Cr-1Mo steel by selecting an optimal amount of the above-mentioned carbide forming elements and designated as modified Grade P91 (X10CrMoVNb 9-1) steel [12]. However, the performance of welds for long term circumstances in newly developed grades has been a significant concern, since they are prone to hydrogen embrittlement and SCC. These are the most common threat to the structural integrity of these alloys [13–15]. The boiler power plants are subjected to SCC and hydrogen assisted cracking (HSC) [16–22]. Still now, most of
the researchers have carried out the SCC susceptibility and HSC tests separately, but in this paper, both tests are carried out simultaneously. In order to carry the project, it aims to develop a new setup based on an improved understanding of both tests. This paper elaborates on the study of environment assisted cracking of modified grade 92 material.

1.1. Chemical composition
The chemical composition of P92 steel is listed in table 1, which confirms the ferritic-martensitic steel alloyed with niobium and vanadium elements. Further, to carry out the welding, the electrodes were baked at 350°C for 2 h (table 2).

1.2. Implant test
The implant test employed in our work was initially developed by Granjon at the French Welding Institute \cite{17}. Figure 1(a) shows the Granjon implant test machine, and figure 1(b) illustrates the distance between implant specimen and borehole. Further information regarding the test setup, an operation can be found here \cite{23–25}.

A cylindrical specimen is notched around its circumference at one end and inserted into a backplate, which is shown in figure 2. A weld bead was deposited over the specimen such that fusion zone and notch lies in coarse-grained HAZ. Besides, time taken for cracking is determined for a range of applied stresses to access the hydrogen cracking susceptibility \cite{25}.

![Figure 1. The Granjon implant test machine.](image)

| Table 1. Chemical composition of grade 92 material. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| C | Mn | P | S | Si | Cr | W | Mo | V | Nb | N | B | Al | Ni |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Min | 0.07 | 0.3 | 0.02 | 0.01 | — | 8.5 | 1.5 | 0.3 | 0.15 | 0.04 | 0.03 | 0.001 | — |
| Max | 0.13 | 0.6 | — | — | 0.5 | 9.5 | 2 | 0.6 | 0.25 | 0.09 | 0.07 | 0.006 | 0.04 |

| Table 2. Weld parameters used for the experiment. |
|---|---|
| Current | 145 A |
| Voltage | 22.25 V |
| Electrode Type | E9218 |
| Diameter of electrode | 4 mm |
| Electrode baked at | 350°C for 2 h |

Figure 1. The Granjon implant test machine.
procedure:

1. Preparation: The implant specimen shall be prepared by turning and the backplate prepared by drilling.

2. Pre-heat: Both test specimens and backplate shall preheat by a suitable method such that the temperature difference between them should not differ by 5 °C.

3. Deposition: Run on and run off plates may be attached for welding. Each weld bead should be deposited in the flat position, in one direction and a single pass. Fusion penetration shall be such as to locate the notch in the coarse HAZ of implant specimen.

4. Loading: Subject to static tensile load, the load is applied after the end of welding before the temperature reaches 100°C or as soon as the holding temperature is reached. The specimen should be loaded slowly, and the load should reach in between the 20 s and 90 s. The load should be set with an accuracy of ±1% and maintained constant over the test period. The specimen should be free from bending, torsion, or shock loading.

5. Test result: The implant test specimen may fracture while the load is maintained, in that case, record the load and time to fracture.

figure 3 shows the cross-section of a test sample for macro and microanalysis. The SCC test and hydrogen-induced cracking test (implant test) are carried out simultaneously by using the newly developed setup. In the implant test, the specimen is loaded and kept in the ambient atmosphere free from any corrosive environment.

2. Experimental work

The following steps are involved during the experimentation of environment assisted cracking (EAC) of grade P92 using this newly developed method.

1. Preparation of the setup

2. Preparation of the samples

3. Measurement of yield stress

4. Preparation of solutions
5. Specimen loading

6. Examination of the loaded specimens

The newly developed experimental setup was prepared with high strength P92 material to withstand the load, which is shown in figure 4.

The samples were prepared with suitable dimensions as per the setup. The machined samples were threaded at its top and bottom portions in order to increase the stress concentration at the top portion such that the crack initiation is easy and the bottom portion for loading the specimen in the setup, which is shown in figure 5. The major diameter and minor diameter of the specimen were 8 mm and 6 mm, respectively.

The measurement of yield stress is done based on the trial test by using a strain gauge instrument and dial gauge instrument, which is shown in figure 6. The yield strength is measured for 0.17 mm elongation of the specimen, if the load is removed at this point the elongation value becomes zero from the 0.17 mm elongation, simultaneously the strain is measured by the strain gauge measurement. The strain at the yielding was around 1400 microns.

The environments used to experiment are CH₄N₂S, copper sulphate, hydrochloric acid, sodium hydroxide, potassium hydroxide. The loading of the sample is quite difficult, and proper care should be taken while applying the load. The load applied is below its yield strength of the material, i.e., 90% yield strength of the material approximately (467.7MPa). The loading is applied by measuring the elongation of the specimen by using the dial gauge i.e. 0.17 mm. The samples were loaded in the above-discussed environments for baked and unbaked electrodes were kept under observation, if the crack initiated, the time taken to fracture will be calculated and then the samples will be characterized through for the macro and microanalysis. Finally, the specimen was kept inside the setup exactly flat to the surface and then perform the welding operation with baked electrodes and unbaked electrodes. The total setup is quenched into the water in order to hold the hydrogen...
content inside the weld pool i.e. the quenching provides sudden cooling. The escaping hydrogen from the weld pool and HAZ found to be difficult due to sudden quenching. After loading the specimen, the whole setup is immersed in the environments mentioned above with baked electrodes and unbaked electrodes. The samples were cleaned ultrasonically before mounting the jobs. The mounted jobs were polished and etched with Vilella’s (picric acid + HCl + methanol) reagent. The fractured surface was cleaned for the SEM and EDS analysis.

3. Results and discussions

The experiment was carried by using two parameters, i.e., welding done by using baked and unbaked electrodes. The specimens were loaded up to 0.14 mm elongation, i.e., around 85% of the yield strength of the material. Among all loaded specimens, the specimens immersed in copper sulfate were unbroken for both baked electrode and unbaked electrode, and the CH$_4$N$_2$S environment for baked electrode was unbroken. The results are noted on an average of three trials for each specimen. The baked and unbaked conditions are portrayed in table 3.

Further, to carry the metallographic examination, the specimens were broken by applying the load. The specimen in the copper sulphate is broken at 0.42 mm and 0.68 mm elongation for unbaked and baked electrodes, respectively. The specimen kept in the CH$_4$N$_2$S environment for the baked electrode is broken at 0.26 mm elongation. Now, the total elongation at which the specimen failed was 0.56 mm and 0.82 mm for copper sulphate environment unbaked electrode and baked electrode, respectively, and 0.40 mm for CH$_4$N$_2$S environment with the baked electrode. Now, stress was calculated for the elongation of 0.56 mm, 0.82 mm, and 0.40 mm, and strain values for the high elongation were 1500, 1630, and 1750, respectively. The stress calculation was presented for different test solutions (CH$_4$N$_2$S and CuSO$_4$) with and without baked electrodes,
Figure 7. Comparison of baked and unbaked specimen’s time to fracture in different corrosive environments.

Figure 8. Baked sample in CuSO₄ (a) macroscopic image, (b) microscopic image, and (c) fractography of SEM image.

Table 4 Stress calculation for the specimen in CH₄N₂S and CuSO₄ environment.

| Environment | Electrode condition | Elongation for the load of 90% of yield strength | Elongation up to fracture from a yield point | Total elongation | Stress for total elongation |
|-------------|---------------------|-------------------------------------------------|------------------------------------------|-----------------|---------------------------|
| CH₄N₂S      | Baked               | 0.14 mm                                         | 0.26 mm                                  | 0.40 mm         | 507 N mm⁻²                |
| CuSO₄       | Unbaked             | 0.14 mm                                         | 0.42 mm                                  | 0.56 mm         | 552 N mm⁻²                |
| CuSO₄       | Baked               | 0.14 mm                                         | 0.68 mm                                  | 0.82 mm         | 592 N mm⁻²                |
which suggested that baked CuSO₄ exhibited superior elongation. The exact elongation values were provided in table 4. The total time taken for testing all the specimens with different solutions until it gets fractured is illustrated in figure 7.

The cracks were propagated from HAZ for the unbaked specimens tested in NaOH solution (figure 15(b)) and CH₄N₂S solution (figure 17(b)). Figure 8 portrays the microstructure of the CuSO₄ baked sample, which reveals that the crack occurs in the coarse-grained heat-affected zone. These cracks occur due to induced hydrogen. Besides, from the microimages, it can be seen very clearly that the crack is initiated between the weldment and heat-affected zone. The fractography of the SEM image is displayed in figure 8(c) and 9(c), respectively. Further, the fracture is found to be quasi-cleavage intergranular in nature, which has been charged with hydrogen when tensile stress is applied it will induce small cracks, quasi-cleavage occurs where the crack initiates (stress are high), and the fracture changes to intergranular as the crack grows and relieves the hydrogen pressure.

The unbaked specimens of CuSO₄ it can be seen that the cracks occur in the heat-affected zone, primarily it occurs at the coarse-grained heat-affected zone. It shows that the development of crack is due to SCC; moreover, the superficial intergranular corrosion penetrations were observed along the surface of the crack. The SEM image of this specimen suggested that intergranular cleavage fracture. Crack initiates from where stresses are high while crack grows the stresses relieves out. Figure 10 shows the baked HCl specimens, developed stress corrosion cracks, with branches, which can be observed in the weld metal region. It shows that crack is due to SCC. Besides, the SEM image revealed that delicate river patterns are witnessed, and also they show the tilt boundaries, where the grains are merely tilted concerning each other [18]. Finally, it is concluded that the fracture is cleavage intergranular cracking. Figure 11 shows unbaked HCl specimens, revealed the development of SCC, are initiated from the heat-affected zone and propagated towards the weldment. The texture of the surface was not typical of hydrogen assisted cracking. The specimens of KOH baked, exhibited visible crack along the coarse-grained HAZ (figure 12). Further, the texture of the crater was rough and contained smaller

**Figure 9.** Unbaked sample in CuSO₄ (a) macroscopic image, (b) microscopic image, and (c) fractography of SEM image
Figure 10. Baked sample in HCl (a) macroscopic image, (b) microscopic image, and (c) fractography of SEM image.

Figure 11. Unbaked sample in HCl (a) macroscopic image, (b) microscopic image, and (c) fractography of SEM image.
Figure 12. Baked sample in KOH (a) macroscopic image, (b) microscopic image, and (c) fractography of SEM image.

Figure 13. Unbaked sample in KOH (a) macroscopic image, (b) microscopic image, and (c) fractography of SEM image.
Figure 14. Baked sample in NaOH (a) macroscopic image, (b) microscopic image, and (c) fractography of SEM image.

Figure 15. Unbaked sample in NaOH (a) macroscopic image, (b) microscopic image, and (c) fractography of SEM image.
unknown particles. A type of gas pockets and porosity appears due to improper welding, and the density of pits is also high.

The same observation was noticed for KOH unbaked specimens (figure 13). Besides, as observed from the SEM image, the river markings are prevalent, and the surface flaw features are away from the ridges and towards the centre pits. The same was noticed for NaOH baked (figure 14) and unbaked specimens (figure 15). The corrosive environments of NaOH, KOH, HCl, CuSO4, and CH4N2S for 168 h test results of micro-macro examinations and fractography analysis helps to understand the failure mechanism. The metallographic analysis indicates that the failures have arisen at the coarse-grained HAZ region. The micro-crack initiation sites are developed at a coarse-grained HAZ region due to that silicon enriched and oxide inclusions. In CH4N2S corrosive environments, the SEM result confirms that the micro-cracks which are nucleated at the coarser grain in the HAZ region have propagated through α-ferrite grains as transgranular manner.

No evidence of failure has been found in the copper sulfate environment; the samples are not broken due to the effectiveness of copper sulfate decreases as uniform distribution of inclusion increases. The SEM/EDS analysis confirms that a more predominant corrosion mechanism is a passive path corrosion mechanism. Hence, the P92 alloy with low inclusions such as oxides, hard inclusions, or inclusions with uniform distributions would be desirable for resistance to EAC. Based on the above discussion, the microstructure of P92 alloy plays an essential role in the EAC initiation and propagation [25]. The nature of the EAC cracks would follow the transgranular crack propagation, which is very similar to SCC. Moreover, the phase of the microstructural constituent encompasses various precipitates, intermetallic phase, and inclusion size. The SEM images of the CH4N2S treated environment shows the transgranular cracking failure mode. This present investigation on environment assisted cracking of grade 92 material concludes that the inclusion size and distribution become determinative factors for the failure [26, 27]. Figure 16 shows the macro, micro and SEM images of the CH4N2S baked sample. The macro image can be concluded that some of the cracks are propagated towards the weldment and crack is initiated from the coarse grain heat-affected zone. The crack has branches; it means that the cracks are stress corrosion cracks only. From the fractography of the sample, it reveals that the fracture is mixed type fracture i.e. both ductile (dimple) fracture and cleavage fracture. The ductile fracture

![Figure 16. Baked sample in CH4N2S (a) macroscopic image, (b) microscopic image, and (c) fractography of SEM image.](image)
surface is observed that the fractured surface with dimples and cleavage fracture also evident from the same fractography image. Figure 17 shows the macro, micro and SEM images of the CH$_4$N$_2$S unbaked sample. The macro and micro images show that it is evident that the crack is severe of stress corrosion cracking, and the crack is initiated from the coarse grain heat-affected zone propagated towards the weldment and also the images show corrosion pits upon the surface. From the fractography of the SEM image, it can be observed river patterns at some of the places and also can be visible at corroded portions. The hydrogen is a major cause of these failures and the sample fails because of hydrogen embrittlement [24].

4. Conclusions

The experiment was carried by using two parameters, i.e., welding done by using baked and unbaked electrodes. The specimens were loaded around 90% of the yield strength of the material.

- Among all loaded specimens, the specimens immersed in the CuSO$_4$ environment, both baked and unbaked electrodes, and the CH$_4$N$_2$S environment baked electrode were not broken. It is indicating that more predominant corrosion mechanism is a passive path corrosion mechanism.
- Macro images reveal that most of the failure occurs in the coarse-grained heat-affected zone due to stress corrosion.
- Cleavage fractures are observed in all the fractography samples due to the combined effect of stress corrosion cracking and hydrogen cracking.
- The electrodes without baking were failed within a short time than the baked electrodes due to the moisture content present in the electrodes. The effect of baking in terms of diffusible hydrogen is evident from the comparison of baked and unbaked samples.
P92 welds are more susceptible to environmental corrosion so that hydrogen in the consumables and residual stress during welding requires to be controlled to reduce the environment based cracking.

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