

Study on Effect of Failure Modes in Hydrogen Embrittlement of Austenitic Stainless Steel

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Abstract-----Austenitic stainless steel is a good choice of material when needed in harsh environment. Even though the alloy content of material makes it suitable for harsh environment, some premature in-service failures have been recorded. The reason behind this type of failures is due to the degradation of mechanical properties. This study investigates the effect plastic deformation, solution concentration and current on the hydrogen embrittlement of pre-strained as well as unstrained specimen. The specimen is subjected to electrochemical charging of hydrogen continuously for 8hrs. After hydrogen charging the specimen is subjected to tensile test and corresponding change in mechanical properties of the specimens were recorded. The hydrogen charging decreased the yield and percentage elongation of the specimens.

Key words: hydrogen embrittlement, tensile test, electrochemical charging, current, solution concentration.

1 INTRODUCTION

Hydrogen in metals will cause a change in mechanical properties of the material. This kind of effect in metals caused by hydrogen is called as Hydrogen embrittlement. Hydrogen embrittlement is a mechanism and is an important subject for many applications. Austenitic stainless steel is most widely used material in automotive as well as household appliances, because of its low cost, price stable and corrosion-resistant steel. The austenitic stainless steel is immune to chloride-induced stress-corrosion cracking when compared to the other grades of stainless steel. The study deals with analysing the effect of hydrogen in austenitic stainless steel in the chlorine contained environment (to simulate the sea water environment) and identifying its failure modes. The hydrogen charging is carried out using electrochemical charging technique. The effect of concentration of hydrochloric acid, charging current and strain rate analysed in a detailed manner.

2 COMPOSITION OF AUSTENITIC STAINLESS STEEL

The below table shows the composition of austenitic stainless steel used in this study and the percentage contribution of each elements are also listed.

TABLE 1
COMPOSITION OF AUSTENITIC STAINLESS STEEL

Element	C	Si	Mn	P	S	Cr
Wt. %	0.017	0.68	0.88	0.019	0.0057	11.5

3 EXPERIMENTAL SETUP

The specimen is prepared based upon the standard recommended to conduct tensile test. As per the American society of testing material the standard for conducting tensile test is ASTM E8M-04. The specimen is polished with emery paper up to 1200 grit and kept it in a free air space for 24hrs. The specimen is exposed to free air for 72Hrs to form the oxide layers. The specimen preparations are carried out in EDM wire cut in order to get accuracy in dimensions to avoid errors in size and shape of the specimen.



Fig.1.work specimen

The electrochemical charging is carried out in a constant supply of power. The electrolyte used for carrying out the electrochemical process is Hydrochloric acid. The hydrochloric acid is used in three various concentrations (0.1m, 0.3m and 0.5m). The charging current density also gets varied in three ranges ($30mA/cm^2$, $40mA/cm^2$ and $50mA/cm^2$). Three different specimens are created by applying different amount of plastic strain namely 0%, 4% and 8% at slow displacement rate of 1.2m/min. the strain is applied in the axial direction. Hydrogen charging was carried out using electrochemical charging technique consist of regulated power supply and two electrodes.

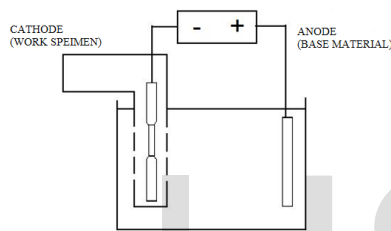


Fig.2. electrochemical charging

The specimen is connected to the cathodic terminal of the regulated power supply and base material acts as the reference electrode and it is connected to the anode of the regulated power supply. The charging duration is kept constant throughout the entire experiment and it only 8hrs at room temperature. The working electrode of austenitic stainless steel is charged at three ranges of current density in three different concentration of hydrochloric acid. The hydrogen charged specimen is subjected to tensile test and pulled to failure. Based upon the percentage of elongation the effect of hydrogen is analysed.

4 RESULTS AND DISCUSSION

After hydrogen charging the specimen is subjected to tensile test. The test was carried out in universal testing machine having a capacity of about 400KN. The test was carried out in ambient temperature.

From the below result it is evident that the elongation is very low for the 8% prestrained specimen and the yield strain of the cold worked

specimen is increased when compared to the specimen which is not subjected to the cold work. The specimen that is prestrained is most brittle in nature when compared to the other specimen, from this it is evident that ductility property of the material gets reduced due to the inducement of hydrogen into the specimen. The hydrogen inducement results in degradation of mechanical properties.

TABLE 2
 TENSILE TEST RESULTS

Sample no	Concentration of electrolyte (X ₁)	Charging current (X ₂)	Pre-strain (X ₃)	Yield strength	Elongation
Units	Mol.	mA/cm ²	%	Mpa	%
1	0.1	30	0	320.9	58
2	0.3	50	0	316.5	59.2
3	0.5	40	0	357.5	61.6
4	0.3	30	4	372.8	57.6
5	0.1	40	4	404	56
6	0.1	50	8	459	54
7	0.3	40	8	441.6	54.4
8	0.5	30	8	440.3	52.8
9	0.5	50	4	388.7	59.2

To identify the contribution factor and the rank of the parameter design of experiments is carried out. Regression analysis was used to evaluate the result of the experiments. The regression analysis is evaluated for each parameter and their responses. The regression analysis is carried out between concentrations of electrolyte, charging current and strain rate to the yield strength. Similarly the regression analysis is carried out between elongation to the concentration of electrolyte, charging current and strain rate. The following tables stated the result of taguchi analysis.

$$\text{The regression equation for Yield Strength} = 311 + 2.2 \text{ Concentration of electrolyte (X}_1\text{)} + 0.503 \text{ Charging current (X}_2\text{)} + 14.4 \text{ Pre-strain (X}_3\text{)} \quad (1)$$

The R-Sq value obtained from the experiment through taguchi analysis is 92.8% and the R-Sq (adj) value obtained is 88.4%.

TABLE 3
REGRESSION ANALYSES FOR YIELD STRENGTH

Predictor	Co-eff.	Se. co-eff.	T	P
Constant	310.58	32.25	9.63	0.000
Concentration (X ₁)	2.17	36.13	0.06	0.955
Charging current (X ₂)	0.5033	0.7226	0.70	0.517
Pre-strain (X ₃)	14.417	1.806	7.98	0.000

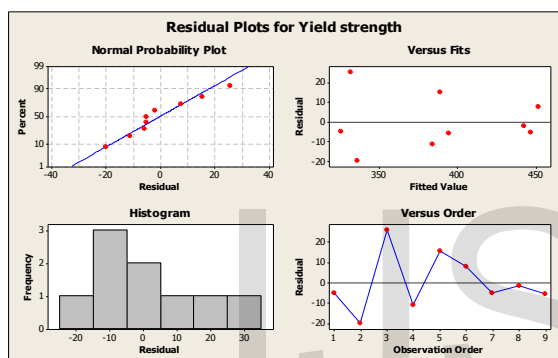


Fig.3.residual plot for the response yield strength to the process parameters.

TABLE 4
REGRESSION ANALYSES FOR ELONGATION

Predictor	Co-eff.	Se. co-eff.	T	P
Constant	55.844	2.167	25.97	0.000
Concentration (X ₁)	4.667	2.428	1.92	0.113
Charging current (X ₂)	0.06667	0.04856	1.37	0.228
Pre-strain (X ₃)	-0.7333	0.1214	-6.04	0.022

The regression equation for ELONGATION = 55.8 + 4.67 Concentration of electrolyte (X₁) + 0.0667 Charging current (X₂) - 0.733 Pre-strain (X₃) (2)

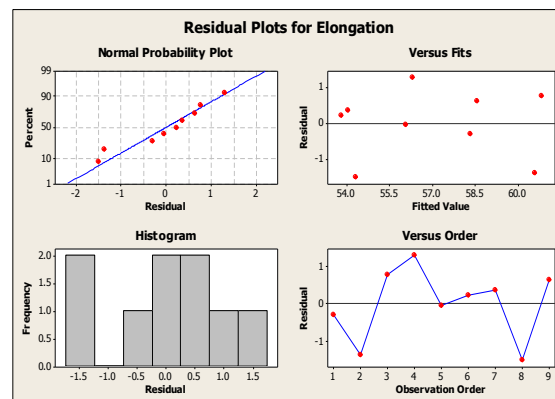


Fig.4.residual plot for the response elongation to the process parameters.

The R-Sq value obtained from the experiment through taguchi analysis is 89.4% and the R-Sq (adj) value obtained is 83%.

Ranking of the parameters based on their influence over the yield strength is mentioned in the table 5.

TABLE 5
PARAMETER RANKING BASED ON THEIR INFLUENCE ON YIELD STRENGTH

Parameters	Percentage contribution	Rank
Concentration (X ₁)	12.98	3
Charging current (X ₂)	15.20	2
Pre-strain (X ₃)	71.82	1

Table 5 clearly indicates the pre-strain or percentage cold has significant impact on the strength followed by the charging current. It is also found that concentration was the least significant factor was due to the high corrosion resistance properties of austenite stainless steels.

From the table 5 and table 6 it is to be stated that pre-strain has the highest influence over the percentage elongation followed by charging current and solution concentration.

TABLE 6
 PARAMETER RANKING BASED ON
 THEIR INFLUENCE ON PERCENTAGE
 ELONGATION

Parameters	Percentage contribution	Rank
Concentration(X_1)	19.70	3
Charging current (X_2)	14.5	2
Pre-strain (X_3)	65.80	1

4 CONCLUSION

The finding from this investigation is summarised as follows.

- The use of Taguchi L9 experiments significantly reduced the number of experiments required to study the hydrogen embrittlement of austenite stainless steels
- The adequacy of the first order models were assessed using the R-Sq. values and found both models were adequate.
- The parameter pre-strain (X_3) has the highest contributing factor towards both the responses.
- The parameter solution concentration has the least contributing towards the responses.

It is better to avoid the usage of severely cold worked specimens in the environment where hydrogen charging is suspected.

REFERENCES

- [1] Lonyuk.B, Hop.R, et.al, (2013), "A study of hydrogen embrittlement in automotive fastener steel", oral/poster reference: icf100386or
- [2] Arup Mallick, et.al, (2013), "Internal reversible Hydrogen embrittlement leads to failure of cold drawn wires" Journal of Engineering failure analysis, vol.1, pp.139-143.
- [3] Shasaku takagi, et.al, (2013), "Hydrogen embrittlement evaluation methods for ultra-high strength steel sheets for automobiles", International journal of automotive engineering vol.2, pp.7-13.
- [4] Fassina.P, et.al, (2011), "Influence of hydrogen and low temperature on pipeline steels mechanical behaviour", Procedia engineering, vol.10, pp: 3226-3234.
- [5] Diblikova.L, et.al, (2014), "Mechanical and electrochemical evaluation of organic inhibitors effect on mild steel damage by hydrogen", Procedia engineering, vol.74, pp: 303-308.
- [6] Capelle.J, et.al, (2014), "Evaluation of electrochemical hydrogen absorption in welded pipe", Procedia material science, vol.3, pp: 550-555.
- [7] Chunjie Ye, et.al, (2013), "Experimental study of hydrogen embrittlement on AISI 304 stainless steels and Rayleigh wave characterization", Journal of Engineering failure analysis, vol.5, pp.228-234.
- [8] Cwiek.J, (2010), "Prevention methods against hydrogen degradation of steel", Journals of achievements in materials and manufacturing engineering, vol.43, issue.1,pp:214-221
- [9] J. Toribio, (1991)," Effects of strain rate and notch geometry on hydrogen embrittlement of AISI type 316L austenitic stainless steel", Fusion Engineering and Design ,vol.16, pp.377-386.
- [10] Motomichi koyama, et.al, (2014), "Hydrogen embrittlement associated with strain localization in a precipitation – hardened Fe-Mn-Al-C light weight austenitic steel", International journal of hydrogen energy, vol.39, pp.4634-4646.
- [11] P.Rozenak and D.Elizezphase, (1987), "Changes related to hydrogen-induced cracking in austenitic stainless steel" Acta metal, Vol. 35, No. 9, pp. 2329-2340.
- [12] C. San Marchi et.al, (2008), "Effect of alloy composition and strain hardening on tensile fracture of hydrogen-pre charged type 316 stainless steel" International journal of hydrogen energy, vol.33, pp. 889-904.
- [13] J.Toribio,et.al, (2010), "Effects of manufacturing-induced residual stresses and strains on hydrogen embrittlement of cold drawn steels", Procedia Engineering, vol.10, pp. 3540–3545.
- [14] Laura verganii, et.al, (2014), "Hydrogen effect on fatigue behaviour of a quenched tempered steel" Procedia Engineering, vol.74, pp. 468-471.
- [15] Arnaud Macadre, et.al, (2014), "Ultra-grain refinement effect on tensile and phase transformation behaviour in a metastable austenitic steel charged in hydrogen gas" procedia material sciences, vol.3, pp.350-356.
- [16] J. Cwiek, (2010), "Prevention methods against hydrogen degradation of steel", journals of achievement in materials and manufacturing engineering, vol.43, issue1.
- [17] Chunjie ye, et.al, (2013), "Experimental study of hydrogen embrittlement on AISI 304 stainless

steel and Rayleigh wave characterization”
Engineering failure analysis, vol.34, pp.228-234.

- [18] M.B.Whiteman and A.R.Troiano, “*Hydrogen embrittlement of austenitic stainless steel*”.

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