HYDROGEN ASSISTED CRACKING SUSCEPTIBILITY OF MODIFIED 9Cr-1Mo STEEL

S.K. ALBERT, V. RAMASUBBU and T.P.S. GILL

Materials Joining Section, Materials Technology Division,
Indira Gandhi Centre for Atomic Research, Kalpakkam 603 102

Presented at International Welding Conference (IWC-2001) on Advances in Welding & Cutting Technology held on 15th–17th February, 2001 at New Delhi.

ABSTRACT

Hydrogen Assisted Cracking (HAC) susceptibility of modified 9 Cr-1 Mo (T91/P91) steel has been studied using UT-Modified Hydrogen Sensitivity Test. Autogenous bead-on-plate welds were made on specimens clamped in a copper fixture using Gas Tungsten Arc Welding process. Hydrogen was introduced through the shielding gas and pre-heating of the specimens was done by heating the copper fixture to the desired temperature. Immediately after welding, specimens were strained to a known strain level in a straining jig for 24Êh and subsequently checked for cracking. The critical preheating temperature, above which no cracking occurred was measured for different hydrogen levels and strain levels. The results indicated that without preheating cracking occurs in this steel even at hydrogen levels as low as 0.25 vol.% in the shielding gas. The critical preheating temperature was found to be a strong function of strain level, while for a given strain level the critical preheating temperature did not vary appreciably with hydrogen content in the shielding gas. Microstructural observation of the cracked samples showed that in this steel cracking occurs both in the weld metal and coarse-grained heat affected zone, in contrast to that obtained in the 9Cr-1Mo steel where cracking is predominantly confined to weld metal. Thus, the present study showed that modified 9Cr-1Mo steel is highly susceptible to HAC and cracking can occur even at very low hydrogen levels.

INTRODUCTION

Modified 9Cr-1Mo steel is used widely in both fossil power plants and petrochemical industries for fabrication of components exposed to temperature in the range of 550-630 °C in service because of its very good creep resistance. This steel is used in normalised and tempered condition and in the as-normalised state. Structure is fully martensitic which is tempered during the subsequent heat treatment. Welding is one of the important processes extensively used in fabrication of various components. For Cr-Mo steels welding is carried out using consumables of matching composition. For high Cr steels like, modified 9Cr-1Mo steel, hardenability is high and hence, in the as-welded condition, microstructure of the heat affected zone (HAZ) and weld metal is martensitic. This makes both HAZ and the weld metal susceptible to hydrogen assisted cracking (HAC).

HAC susceptibility of different Cr-Mo steels have been studied employing

Table I : Chemical Composition of Mod. 9Cr-1Mo Steel Plate

Element wt.%	C Mn	Si	S	P	Cr	Ni	Mo	V	Nb	Al	N	
Mod. 9Cr-1Mo steel	0.097	0.37	0.31	0.0047	0.018	9.29	0.38	0.92	0.26	0.08	0.006	0.057
9Cr-1Mo steel	0.072	0.36	0.265	0.0008	0.021	8.24	–	0.95	—	—	—	—

different cracking tests [1-5]. It has been found that preheat temperature is a function of alloy chemistry and hydrogen content in these steels [6]. Preheating generally recommended for these steels to avoid cracking during welding is in the range of 200-300°C. In actual fabrication, in addition to preheating, post-heating is also employed to ensure removal of hydrogen and to prevent cracking.

In the present investigation, HAC of modified 9Cr-1Mo steel is studied using UT-Modified Hydrogen Sensitivity Test (UT-Modified HST). In this test, specimens with different levels of hydrogen are prepared after preheating to different temperatures. These specimens were subsequently strained to a known strain level to facilitate cracking. The critical preheat temperature above which no cracking occurs is determined for a given hydrogen level and strain level. Results obtained from this study are discussed and compared with those obtained from normal 9Cr-1Mo steel using the same test.

EXPERIMENTAL PROCEDURE

Modified 9Cr-1Mo steel plates (thickness 12 mm) available in normalised (1060°C/ 25min) and tempered (750°C/1h) condition was used in this study. Chemical composition of the plate is given in Table I. Also shown in the table is the chemical composition of the normal 9Cr-1Mo steel with which a comparison is made later in the text. Plate was sliced into two (~5mm thick) which were subsequently shaped to reduce the thickness to 3mm. Specimen blanks of 45x15x3mm were prepared from these plates.

UT-Modified Hydrogen Sensitivity Test (UT-Modified HST) : This test is a variation of the RPI augmented strain test [7-9]. Specimen for testing is prepared by making an autogenous bead-on-plate weld along the length of the specimen using GTAW torch. Welding parameters employed are given in Table II. Hydrogen is introduced into the weld pool by mixing it with the shielding gas. Hydrogen content in the shielding gas is varied from 0.25 to 1.5 vol.% to obtain different levels of hydrogen content in the weld metal. Specimen is held in a copper fixture that can be preheated to the desired temperature. After welding, the specimen is allowed to cool to room temperature and then strained in a fixture, as shown in Fig.1. The nominal augmented strain on the surface is given approximately as

$$\varepsilon \sim t/2R$$

Table II : Welding Parameters Used in UT Modified HST

Current	90-100 A
Voltage	11-12 V
Welding speed	125 mm/min
Gas (Argon) flow rate	10 l/min
Electrode	Thoriated Tungsten

where e = nominal augmented strain, t = specimen thickness and R = bending radius. Die blocks of different radius are used to produce different strain levels varying from 0.5 to 4%. The susceptibility to cracking is determined by observing cracks formed on the specimen face

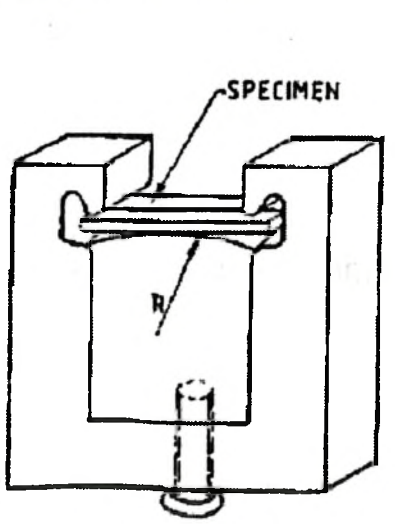

Fig.1: Loading Jig for UT-Modified Hydrogen Sensitivity Test.

strained in tension after straining for 24 h. Preheat temperature above which no specimen cracked for a given hydrogen level and strain level is chosen as the critical preheat temperature for that condition. Critical preheat temperature for modified 9Cr-1Mo steel was determined for different levels of hydrogen in the shielding gas and strain.

Macrographic Examination and Metallography : A stereomicroscope was used to examine the cracks and the fracture surface. In order to estimate the area that cracked during testing, specimens were immersed in liquid nitrogen and then broke open by hitting at one end of the specimen. For differentiating the area cracked during testing from that fractured during breaking, specimens were oxidised for 15 min at 600° C before immersing in liquid nitrogen. Area of the specimen cracked during testing was observed using the microscope.

Top surface of the specimens cracked during testing were prepared for metallography by grinding and polishing.. Etching was done using Vilella's reagent (1g picric acid,5 ml concentrated HCl and 95 ml of methanol). After etching, specimens were observed in an optical microscope to study propagation of cracks in the weld metal and heat affected zone (HAZ).

Hardness Measurements : Hardness of the weld metal, HAZ and base metal was measured using Vickers hardness tester at 10kg load. Microhardness profile on the surface of the specimen across the weldment was also taken at 200 g load using Shimadzu HMV 2000 machine.

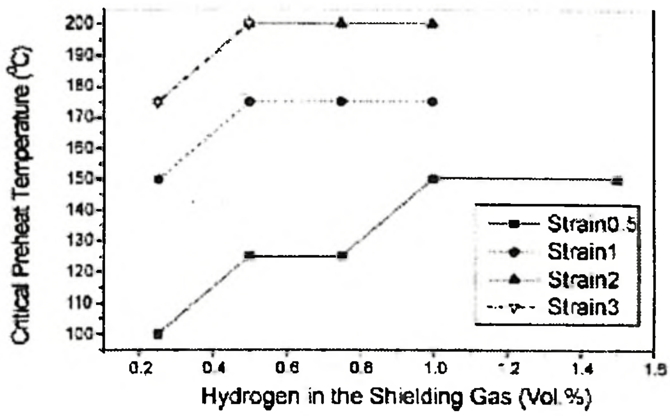

Fig.2: Variation of Critical Preheat Temperature with vol.% of H2 in the Shielding Gas and Loading Strain.

RESULTS

Hydrogen Assisted Cracking : None of the specimens tested without addition of hydrogen in the shielding gas cracked during testing. However, cracking observed in specimens tested at room temperature, irrespective of the strain level, even with 0.25vol.%hydrogen in the shielding gas. This confirmed that cracking observed during testing is solely due to hydrogen. Further, in many specimens, especially those tested without preheating, more than one crack was observed.

Critical preheat temperature was determined for a given vol.% of hydrogen in the shielding gas for different levels of strain. The preheat temperature at which no specimen cracked after three repeated tests is chosen as the critical preheat temperature. Variation in critical preheat temperature as function of hydrogen content in the shielding gas for different strains is shown in Fig. 2. For 0.5% strain, preheat temperature increased from 100 to 150°C only for a six-fold increase in hydrogen content from 0.25 to 1.5 vol.%. For other strain levels too this increase in critical preheat temperature with hydrogen levels similar. Further, critical preheat temperature seems to saturate at higher hydrogen levels. There was no change in this temperature for testing carried out at 1 and 2% strains as the hydrogen levels increased from 0.5 to 1.5%. Variation in critical preheat temperature with strain is more pronounced than that with hydrogen content in the shielding gas. With 0.25vol.% of hydrogen in the shielding gas, cracking could not be avoided even with a preheat temperature of 200°C when strained to 4% strain. Further, as the strain increased from 0.5 to 3%, critical temperature increased from 100 to 175°C.

In general, cracks observed on the specimen surface were continuous and extended from HAZ on one side of the weld to the other as shown in Fig 3. Shape of the cracks are convex in the direction of welding as shown in Fig.3. Some of the cracks were very fine and in certain cases cracks also exhibited extensive branching. Cracks that did not progress to the HAZ were also observed, mainly in those specimens that had more than one crack. Very rarely, cracks that were confined to HAZ on one side were also seen in specimens that had more than one crack.

Fig.4 shows fracture surface of a typical crack after oxide tinting. Crack has extended to whole of weld metal and part of the HAZ on either side. However, in the specimens with multiple cracks, some of the cracks were confined only to weld metal.

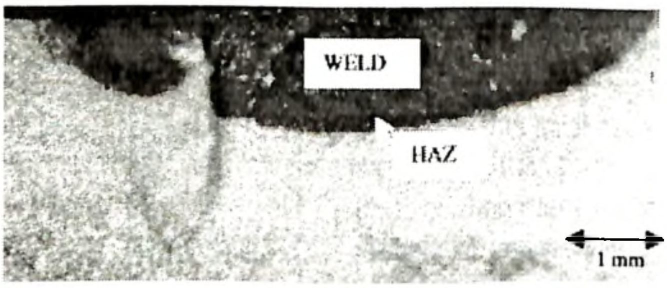

Fig.4 Fracture Surface after Heat Tinting revealing cracking in both weld metal and part of HAZ- (0.25 vol.% of H2, No preheat, 4% strain)

Microstructure : Fig.5 shows microstructure near the fusion boundary of the sample revealing both the weld metal and HAZ along with a crack. Crack has stopped in the fine grained HAZ and does not propagate further. Microstructure of both weld metal and HAZ is fully martensitic. Microstructure near the crack tip for another specimen is shown in Fig.6. In this zone, crack propagation is not straight and it appears to have taken place along prior austenite boundaries. Microstructure of the weld metal along with a branched crack is shown in Fig.7. Width of the crack is more in the weld metal and its propagation was not confined to prior austenite boundaries as in the HAZ. Thus it appears cracking is more severe in weld metal than in HAZ. Neither weld metal nor HAZ seem to contain any d-ferrite that is often reported to be present in this steel.

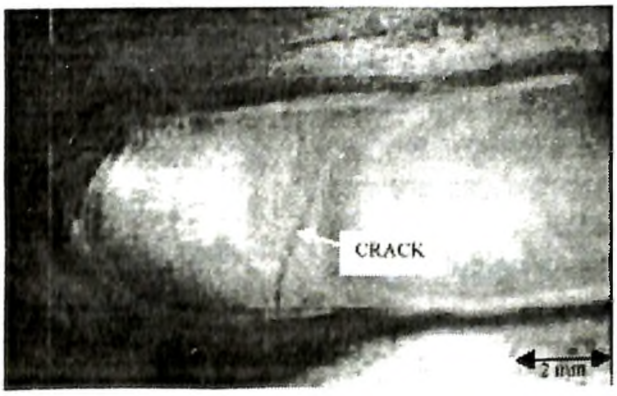

Fig.3: A Typical Crack in the Specimen after Testing (0.25 vol.% of H2, No preheat, 4% strain)

Hardness : Average hardness of base metal was around 260 VHNwhile that of HAZ and weld metal was around 500 VHN. Base metal hardness is typical of the normalised and tempered steel while that of HAZ and weld metal is typical of an alloy steel with ~0.1wt.% carbon and fully martensitic structure. Microhardness profile across the weldment for a typical specimen is given in Fig.8. In the weld metal hardness was uniform (~500 VHN) and the value is in agreement with the macro-hardness measurements. In the HAZ, hardness showed some variation with distance from fusion zone. Hardness very near to the fusion line is significantly lower than the rest of the region and this is attributed to alloy partitioning during initial stages of solidification. In the region close to that of the base metal that is heated up to inter-critical temperature range and not beyond, hardness is below 500 VHN. Extension of the crack into either side of the HAZ was measured for some typical cracks. These measurements confirmed that crack did not extend beyond fine grained HAZ.

DISCUSSION

The above results clearly show that Modified 9Cr-1Mo steel is highly susceptible to HAC. Even with 0.25 vol.% of hydrogen in the shielding gas cracking occurred irrespective of the strain level for specimens prepared without preheating. This level of hydrogen corresponds to hydrogen levels as low as 0.5ml/100g of weld metal [10]. In addition, for this hydrogen level, cracking could not be prevented when tested at 4% strain level, even after preheating the specimen up to 200°C. Very high susceptibility of this steel to HAC becomes clear if the present results are compared with those obtained for normal 9Cr-1Mo steel. For this steel, specimens prepared with 0.25vol.% of hydrogen and without preheating did not crack even with 4% strain. Further, critical preheat temperature of this steel with 1vol% hydrogen was only around 175°C for 4% strain [6]. However, for modified 9Cr-1Mo steel, even with 0.25 vol.%, .

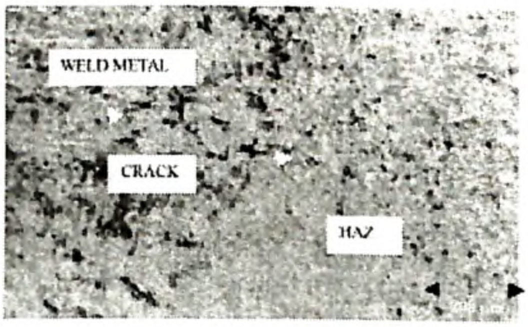

Fig. 5: Microstructure near the fusion boundary showing extension of the crack from weld metal to HAZ

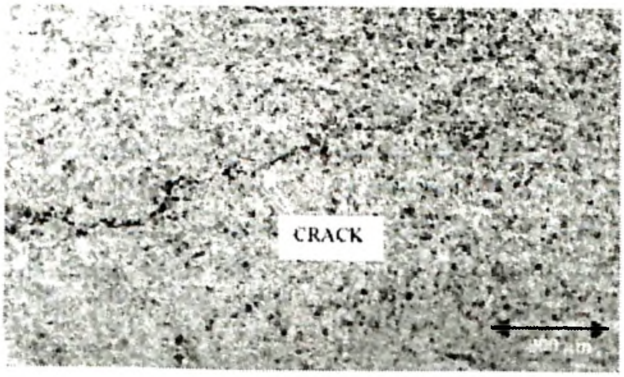

Fig. 6 Microstructure near the crack tip at HAZ showing propagation of crack along prior austenite boundaries

Fig. 7 Microstructure of the weld metal with a branched crack

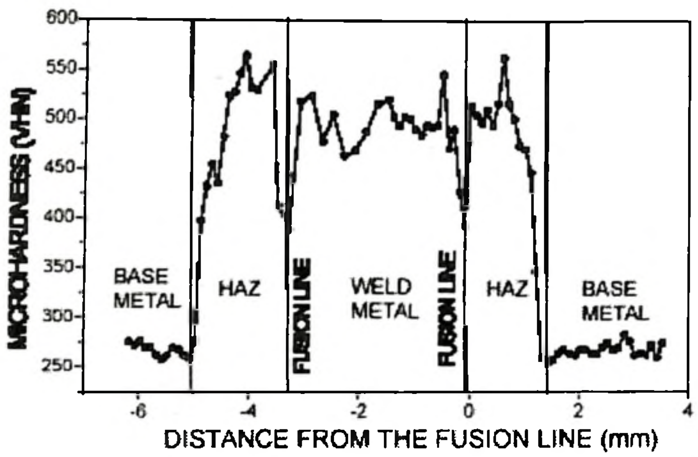

Fig. 8 Hardness profile across the weld for the UT-HST Specimen

of hydrogen, this temperature seems to be above 200°C. Differences are also seen in the cracking behaviour. For normal 9Cr-1Mo steel, cracking was predominantly confined to weld metal while in this steel cracking is observed both in the weld metal and HAZ.

Reasons for significant difference in the cracking behaviour of two steels are not very clear. The major difference in the specification of these two classes of steels is the presence of Nb,V and N in the modified steel. They are all present in <1wt.% and in the as welded condition, these elements would be mostly in solid solution. Microstructure of both weld metal and HAZ for both the steels is martensitic. Further, it has also been reported that for Cr-Mo steels with 2-3wt.%Cr, presence of elements like Nb and V did not significantly influence the cracking behaviour [4]. However, in a recent study wide variation in the critical preheat temperature for 9Cr-1Mo steels showing minor variation in composition has been reported [5]. In this study, which also employed UT-modified HST with 5 vol.% of hydrogen in the shielding gas, critical preheat temperature obtained for 1%strain varied from 25 to 400°C for eight different compositions of both modified and normal 9Cr-1Mo steels. Steel with a minimum carbon content of 0.055 wt.% had the minimum value while the one with maximum carbon content (0.11wt.%) had the highest critical preheat temperature. Results also indicated that Si increases the susceptibility of the steel to HAC. On the basis of these reported results, higher HAC susceptibility of the steel than that of the normal 9Cr-1Mo steel with which it is compared, can be explained. A comparison of the chemical composition of these steels (Table I) shows that both carbon and Si are higher in the former than the latter. High carbon content also results in high hardness in the weld metal and HAZ (500 VHN compared to 400 VHN in the normal steel). It is well known that susceptibility to HAC increases with hardness.

As HAC is essentially due to interaction of hydrogen atoms with the steel microstructure, significant difference in HAC susceptibility should also imply differences in hydrogen solubility and diffusivity of the steels. This has also been confirmed from the hydrogen permeability studies carried out in these steels in water quenched condition which produces a fully martensitic microstructure similar to that obtained in the weld metal and HAZ [11]. Apparent solubility of hydrogen in the modified steel was only around 1.18×10^{-5} mol/cm^3 against 1.78×10^{-4} mol/cm^3 for the normal steel. Similarly, hydrogen diffusivity of the modified steel was much higher (6.82×10^{-8} cm^2 /s) than that of the normal steel (1.13×10^{-2} cm^2 /s). However, further studies are required to find out how these differences eventually leads to differences in HAC susceptibility of the steels.

CONCLUSIONS

Results show that modified 9Cr-1Mo steel, employed in the present study, is highly susceptible for HAC. For specimens prepared without preheating, even hydrogen as low as

0.25 vol.% in the welding arc is sufficient to cause cracking. A comparison of the results with that of the similar tests conducted on normal 9Cr-1Mo steel and with the results reported in literature indicates the reason for high susceptibility is the high carbon and Si content in the steel. It appears, minor variations in the chemical composition are sufficient to cause significant changes in the susceptibility of this class of steels to HAC. This is also supported by variation in the hardness, hydrogen diffusivity and solubility with minor variation in alloy content.

ACKNOWLEDGMENT

Authors acknowledge the support and guidance provided by Dr.S.K.Ray, Head, Materials Technology Division, IGCAR during course of this work. Assistance by Mr. P.Rajeesh and Mr.P.Faizal. during the course of the work is also gratefully acknowledged.

REFERENCES

1. Colombe, G., Dibiez, S., Donati, J.R, Petreguin, P. and Zacharie, G: Metallurgical Aspects of Weldability of Cr-Mo Stabilised Ferritic Steels in Ferritic steels for Fast Breeder Reactor Steam Generators", ed. S.A.Pugh and E.A. LIttle, BNES, London, PP 447-458 (1978).
2. Hart, P.H.M and Watkinson, E : Weld Metal Implant Test Ranks Cr-Mo Steels Hydrogen Cracking Resistance: Weld. J. 65(1), 14s (1986).
3. Terai, K., Shimazu, S. Nakai, Y, Murakami, T., Ota, S. and Fujiwara, M. : Weldability and Joint Properties of Advanced Carbon Stabilised 2.25Cr-1Mo Steels for LMFBR in Ferritic Steels for Fast Breeder Reactor Steam Generators Ed. S.F. Pugh and E.A. Little, BNES, London, PP442-446 (1978).
4. Lindin, C.D. Henning, J.A., Menon, R. and Khan, K.K : Transformation, Metallurgical Response and Behaviourof the Weld Fusion Zone and HAZ in Cr-Mo Steels for Fossil Energy Applications, Report ORNL/Sub/81-07685/02&77 (1987).
5. Lundin, C.D., Milton, R., Henning, T.A. and Richey M.W. : Hydrogen-Assisted Cracking Sensitivity of Cr-Mo Steels During Welding: Advances in Welding Science and Technology Ed. S.A. David, ASM International, Ohio, PP 585-596. (1986).
6. Albert, S.K., Gill, T.P.S., Ramsubbu V and Kulkarni, S.D. : Variation in Diffusible Hydrogen Content and Hydrogen Assisted Cracking Susceptibility of Cr-Mo Steels' Proc. of International Welding Conference, The Indian Institute of Welding, Mumbai, 1996, PP. (A54-1)- (A54-8).
7. Savage, W.F., Nippes, E.F and Szekeres, E.S. : Hydrogen Induced Cold Cracking in a Low Alloy Steel: Welding Journal 55(9), 276s (1976).
8. Savage, W.F., Nippes, E.F and Homma, T. : Hydrogen Induced Cracking in HY-80 Steel Weldments: ibid 55(11), 368s (1976).
9. Savage, W.F., Nippes, E.F and Husa, E.S. : Hydrogen Assisted Cracking in HY-130 Weld Metal: ibid 61(8), 233 (1982).
10. Albert S.K., Vinoy T.V., Hasan Shaik and Gill T.P.S. : Use of Acoustic Emission to Monitor Hydrogen Assisted Cracking in Weldments" Proc. of International Welding Conference, Feb.15-17 1999, Delhi PP 1031-1038.
11. Parvathavardhinit, N. Personal communication.

■