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Measurement of Diffusible Hydrogen Content and Hydrogen Effects on the Cracking Potential of Duplex Stainless Steel Weldments (Part I)

Yasushi KIKUCHI*, Carl D. LUNDIN**, K. K. KHAN***

Abstract

The variation in the hydrogen content was achived by employing a controlled electrode baking schedule, which varied the moisture content of the electrode coating and thus the hydrogen avaiable to the weld pool. Three different SMAW electrodes were employed with a variation in ferrite content achieved by coating control (depositing welds of three different chemistries). Cracking susceptibility of welds with different ferrite contents at four hydrogen levels were carried out with "Y-Groove" cracking test. Hydrogen content was measured using the AWS standard method (45 °C, 72 hours) and with an elevated extraction scheme.

- (1) The diffusible hydrogen in duplex stainless steel weldments determined at 45 °C (weld metals of FN 73, 96.5 and 103) is negligibly small.
- (2) The diffusible hydrogen content increases with an increase in extraction temperature up to $600\,^{\circ}$ C and remains essentially constant at 25ml/100gm to $950\,^{\circ}$ C for an extraction time of 1 hour.
- (3) The diffusible hydrogen content for extraction at 200 °C for 72 hours (>20 ml/100 gm) approaches that measured for 600 ° and 950 °C for 1 hour.
- (4) "Y-Groove" test results show that cracking occurs in weld metals of FN 73.5, 95 and 103 made with "green" unbaked electorodes (25ml/100gm diffusible hydrogen). Cracking does not occur in weld metals of FN 73.5 and 95 when "green" electrodes baked above 315 °C are employed. For weld metal of FN 103, a "green" electrode baking temperature of 370 °C is needed to eliminate craking in the "Y-Groove" test.
- (5) The critical diffusible hydrogen content for weld metal cracking is estimated to be 15 ml/100 gm for FN 73.5 and 95 weld metals and 10 ml/100 gm for FN 103 weld metal.

KEY WORDS: (Stainless Steel) (Cold Cracking) (Hydrogen Content) (Ferrite Content)

Introduction

Duplex stainless steels have been developed to combine the advantages of both the austenitic and ferritic phases, with austenite imparting good toughness and corrosion resistance and ferrite providing strength and resistance to stress corrosion cracking and sensitization. Hydrogen assisted cracking is not of serious concern in austenitic stainless steel weldments containing deltaferrite. However, as the ferrite content in the stainless steel weld metal is increased, as in a duplex steel it is considered that the weld metal may become sensitive to hydrogen assisted cracking. However, it is not clear whether the potential for hydrogen cracking in duplex stainless steel is of significant importance. Thus, to determine the behavior of hydrogen in duplex stainless steel is of considerable interest and since little research has been conducted in this area the current study was instituted.

Current Status

There is only limited information concerning hydrogen assisted cracking in duplex stainless steel weld metal. GTAW with $Ar-H_2$ mixtures in the shielding gas and SMAW are often employed as a common practice in welding of both duplex stainless steel and austenitic stainless steels. However, a recent study has established a deleterious ductility effect and cracking related to hydrogen in duplex stainless steel weldments (1-2). These papers have shown weld metal cold cracking in the 60-100 % ferrite range.

In the current study the level of diffusible hydrogen which can be extracted at different temperatures and times with three ferrite levels in the weld metal has been determined. The "Y-Groove" test and UT-modified hydrogen sensitivity test have been employed to evaluate the hydrogen cracking susceptibility and the level of diffusible hydrogen necessary to potentially influence mechanical properties and cracking has been estimated.

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Continued work on the determination of mechanical properties at different diffusible hydrogen levels is needed to recognize the full influence of hydrogen in the duplex stainless steels. Research toward this end is to be conducted in the next phase of the work together with the evaluation of different SMAW duplex consumables.

Materials and Experimental Procedures

a) Materials

The base metal used was Ferralium Alloy 255 duplex stainless steel (thickness 1" and 1/2"). Three different SMAW electrodes were employed with a variation in ferrite content achieved by coating control (depositing

welds of three different chemistries). The chemical composition of the base materials and the undiluted weld pad deposits together with the ferrite number of the weld pad is presented in **Table I**.

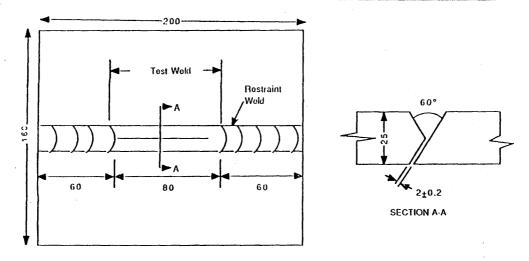
"Y-groove" test samples were extracted from the 1" thick plate and were machined to the required shape as per the Japanese Industrial Standard (JIS Z 3158) [3]. The shape and dimensions of the "Y-groove" test samples conform to Fig. 1.

For hydrogen measurement two different sample configurations were used as shown in Fig. 2, $(13 \times 12.5 \times 110 \text{ mm})$ and $25.4 \times 12.5 \times 140 \text{ mm}$ samples compatible with Oerlikon-Yanaco diffusible hydrogen analyzer). The smaller sized specimen was used for hydrogen extraction at high temperatures after encapsulation in a glass or

Table I Chemical Analysis of Base Metals and Undiluted Weld Pads and Ferrite Number of Weld Pads.

Base Metal	C	Cr	Cu	Мn	Мo	Ni	P	S	Si	N	H ₂ (ml/100g)
1"Thick	0.02	24.7	1.9	1.0	3.1	5.9	0.020	0.003	0.4	0.17	•
1/2"Thick	0.01	25.0	1.8	1.0	3.1	5.8	0.023	0.003	0.5	0.18	0.37

	Electrode Lot No.	Teledyne Weld Pad FN	С	M n	P	s	Si	Cr	Νi	Мо	Cu	N
Г	9726-082-1	92.3	0.035	0.72	0.029	0.003	0.38	24.76	6.11	3.24	1.83	0.183
١	9726-083-1	74.7	0.034	0.74	0.028	0.003	0.37	24.79	7.43	3.23	1.83	0.178
L	9726-084-1	48.4	0.032	0.69	0.027	0.004	0.33	24.45	8.7	3.24	1.85	0.187



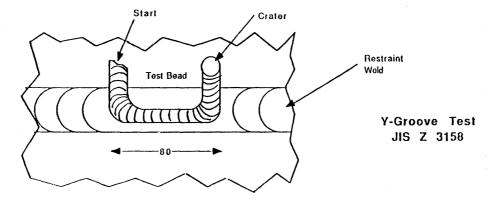


Fig. 1 "Y-Groove" Test Specimen.

quartz tube (The tube is later broken in the hydrogen diffusion analyzer sampler for hydrogen measurement).

b) Measurement of Diffusible Hydrogen

The variation in the hydrogen content was achieved by employing a controlled electrode baking schedule, starting with the electrodes in the "green" condition, which varied the moisture content of the electrode and thus the hydrogen available to the weld pool. Figure 3 shows the relationship between moisture content of the coated electrode and baking temperature (1hour) for the green starting condition. With increased baking temperature the moisture content of electrode markedly decreases. In this study four baking temperatures were selected to provide different coating moisture and therefore diffusible hydrogen in the weld deposit.

The hydrogen content was measured using the AWS standard method (45 °C, 72 hours) and with an elevated temperature extraction scheme.

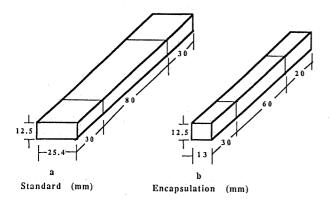


Fig. 2 Samples for Diffusible Hydrogen Analysis. a) Standard, b) Encapsulation.

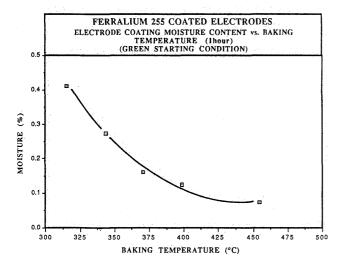


Fig. 3 Electrode Coating Moisture Content Versus Electrode Baking Temperature (Green Starting Condition) One Hour for Ferralium 255 SMAW Electrodes.

Standard method:

Weld metal specimens were prepared according to the method recommended by AWS. For SMA electrodes of 4.0 mm (5/32") dia., the 3-section test coupons are shown in Fig. 2. The welds were clamped in a copper jig, and after welding, the test piece was quenched in iced water 20 sec.) and then transferred into a liquid nitrogen bath where it was held for 2 minutes. The 3-part test piece was broken to obtain the center section which is the actual specimen for hydrogen analysis. The diffusible hydrogen content was measured by gas chromatography using the Oerlikon-Yanaco diffusible hydrogen analyzer.

Elevated Temperature Extraction Scheme:

Encapsulated weld coupons (using a quartz or glass tube) were thermally treated to evolve hydrogen. After thermal treatment, the sealed tube was placed in the sample chamber of the gas analyzer. The sealed tube was broken by a shock to the chamber. Thus, hydrogen in the capsule was released and measured. A. schematic illustration of the glass tube used is shown in **Fig. 4**. The sample size varied from the standard so that the largest size capsule could be used in the diffusible hydrogen analyzer (see Fig. 2).

The temperature for hydrogen evolution was varied to determine the temperature dependence of hydrogen evolution. A series of times from 1 to 72 hours depending on the temperature was chosen to evaluate the time dependence of hydrogen evolution.

c) Cracking Susceptibility Tests

Cracking susceptibility of welds with different ferrite contents at four hydrogen levels was carried out with the "Y-groove" cracking test and UT-Modified Hydrogen Sensitivity Test (UT-Mod.HST). The "Y-groove" test was conducted using SMA slectrodes and the hydrogen content was varied by the electrode baking schedule. Cracks on the weld metal surface and in the weld cross section were used for cracking susceptibility characterization. The UT-mod.HST specimens were extracted from all-weld-metal pads. In this method an autogenous GTA weld is made on an extracted weld metal sample. Hydrogen is directly added to the argon shielding gas. After welding the sample is allowed to cool

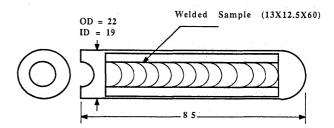


Fig. 4 Schematic Illustration of Encapsulation.

to 90 °F and then strained. Cracking is evaluated as a function of strain level and hydrogen content.

d) Microstructural Investigation

The ferrite content of the test samples was determined by the modified Magne-Gage method. Microstructural features such as the morphology of the austenite and ferrite were evaluated and the fracture surface was characterized for the cracked "Y-Groove" samples.

Experimental Results and Discussion

a) Measurement of diffusible hydrogen

The results of the diffusible hydrogen analyses using standard AWS method for diffusible hydrogen are shown in **Fig. 5**. The electrodes used were in the unbaked, "green", condition and contained greater than 0.4% coating moisture. As can be seen from Fig. 5. the diffusible hydrogen levels (45 °C-72 hours) for EFN 73, EFN 95 and EFN 103 deposits are less than 1 ml/100 g.

The modified hydrogen analysis sample shown in Fig. 2 was encapsulated in quartz tubes which were evacuated to 5-6 microns before sealing. The capsules were exposed for 1, 2 and 4 hours at 950, 600, and 400 °C to extract the hydrogen. In addition, tests were conducted at 100 and 200 °C for 72 hours. For temperatures less than 600 °C, a Pyrex tube of the same size was used.

The relationship between the extracted diffusible hydrogen content for green, unbaked electrodes at 950 °C and FN is shown in **Fig. 6**. The 950 °C temperature was chosen because other researchers had indicated that this was an optimum temperature to remove all diffusible

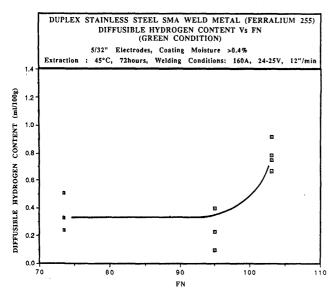


Fig. 5 Diffusible Hydrogen Content Using AWS Standard Method (45 °C, 72 Hours) Versus FN for SMA Ferralium 255 Weld Metal.

hydrogen from duplex weld metals (1). From **Fig. 6** it appears that the diffusible hydrogen content is relatively independent of FN for the unbaked electrodes (moisture > 0.4%) at a level of 24-25 ml/100 gm (The hydrogen content of the base plate is 0.37 ml/100 gm by extraction at 950 °C).

The diffusible hydrogen content of the weld deposits was varied by baking the green electrodes. The effect of baking temperature on the diffusible hydrogen content of the weld metals is shown in Figs. 7 and 8 for FN 73 and 102 weld metal ferrite. The diffusible hydrogen content of the weld deposit decreases with an increase in baking temperature as anticipated. The effect is apparently independent of ferrite content as shown by the combined data presented in Fig. 9. Using a baking temperature of

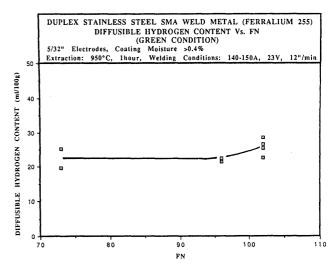


Fig. 6 Diffusible Hydrogen Content (Extraction at 950 °C-1 Hour) Versus FN for SMA Ferralium 255 Weld Metal.

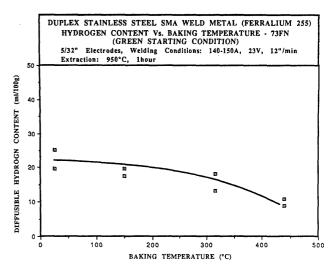


Fig. 7 Diffusible Hydrogen Content (Extraction at 950 °C-1 Hour) Versus Electrode Baking Temperature (Green Starting Condition) for 73 FN Ferralium 255 Weld Metal.

> 300 °C reduces the diffusible hydrogen content to < 10 ml/100 gm. (It is to be noted that the "Y-Groove" test results reveal that weld metal of EFN 73 deposited by electrodes baked at 315 °C did not crack. Thus, ferrite level is also to be considered critical when examining the effects of diffusible hydrogen content.)

The effect of extraction temperature, for a time of one hour, on the diffusible hydrogen content was studied. The relationship between extraction temperature and diffusible hydrogen content is shown in **Fig. 10**. The diffusible hydrogen extracted at 950 °C and 600 °C is actually identical. However, the diffusible hydrogen content

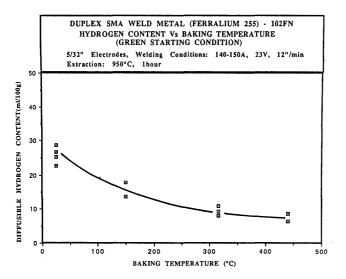


Fig. 8 Diffusible Hydrogen Content (Extraction at 950 °C-1 Hour) Versus Electrode Baking Temperature (Green Starting Condition) for 102 FN Ferralium 255 Weld Metal.

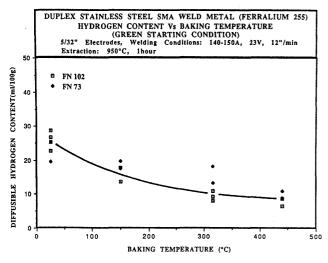


Fig. 9 Diffusible Hydrogen Content (Extraction at 950 °C-1 Hour) Versus Electrode Baking Temperature (Green Starting Condition) for 73 and 102 FN Ferralium 255 Weld Metals.

decreases with extraction temperature when the temperature is less than 500 $^{\circ}$ C. For example, at a 400 $^{\circ}$ C extraction temperature, the diffusible hydrogen content is approximately 19 ml/100 gm whereas at 950 $^{\circ}$ C the level is approximately 25 ml/100 gm.

Extraction of diffusible hydrogen was undertaken at 100 and 200 °C for a time of 72 hours to determine if a lower temperature for a longer time could be effective in removing hydrogen since the procedure would be significantly simplified if this was viable. The results of the 45, 100 and 200 °C extractions for 72 hours are shown in **Fig. 11** and the effect of time for a 200 °C extraction temperature is shown in **Fig. 12**. It is clear that an extraction temperature of 200 °C will remove most of the diffusible hydrogen (20 ml/100 gm) when employed for a time of 72 hours. Times greater than 72 hours need to be

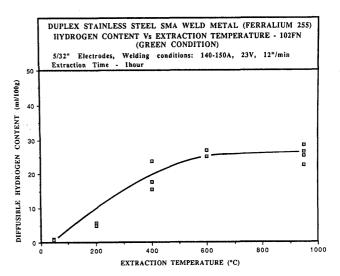


Fig. 10 Diffusible Hydrogen Content Versus Extraction
Temperature for 102 FN Ferralium 255 Weld Metal Deposited with Green Condition Unbaked Electrodes.

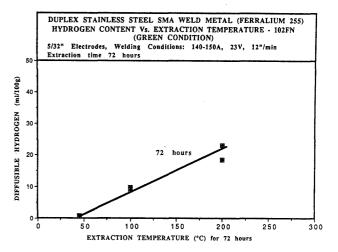


Fig. 11 Diffusible Hydrogen Versus Extraction Temperature for 72 Hour Extraction Time (Green Electrodes).

explored at $200\,^{\circ}\text{C}$ and below for developing a postweld heat treatment schedule that can be readily employed in the field.

The effect of extraction times greater than 1 hour for the higher temperature extraction temperatures was studied further determine time temperature relationships. The full spectrum of diffusible obtained in this study is presented in Fig. 13 as a function of extraction time with extraction temperature as a parameter. The effect of times up to four hours for the 400, 600 and 950 °C extraction temperature reveals some interesting relationships. It is noted that after 4 hours at 400 °C the extracted hydrogen is equivalent to that extracted at 950 °C for 1 hour. Further, it is evident that the measured diffusible hydrogen decreases for extraction at 600 and 950 °C after 1 hour. The reason for this decrease is not known at this time but further work is apparently dictated by these results.

(b) "Y-Groove" Testing

"Y-groove" testing was carried out using coated

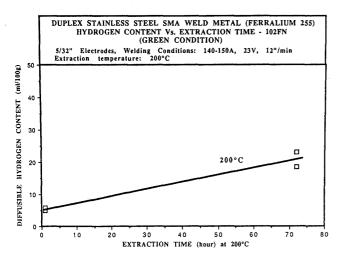


Fig. 12 Diffusible Hydrogen Versus Extraction Time at 200 °C (Green Electrodes).

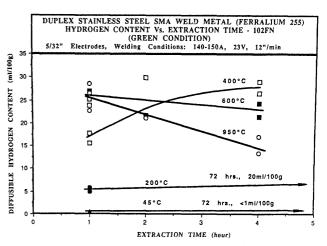


Fig. 13 Diffusible Hydrogen Versus Extraction Time for Various Temperatures (Green Electrodes).

electrodes by manual SMAW. Weld metal diffusible hydrogen content was varied by controlled electrode baking as previously described. The welding conditions were 140-150 Amp., 23V and 4.5-4.7 inch/min. The test results are summarized in Table II. This test is a selfrestraint test and cracking takes place in the deposited weld metal. In our studies, cracks were observed immediately after welding on the weld metal surface of the FN 73, 96 and 103 welds made using unbaked (green) electrodes. (It is to be noted here that the ferrite number of the "Y-Groove" welds is different in the undiluted weld pads for the same electrodes due to a dilution effect. Both ferrite levels are given in Table II for comparison). Cracking was not observed when welds were made using FN 73 and 95 weld metals after baking for all baking conditions (baking temperatures > 315 °C). However, the weld metal at FN 103 using electrodes baked at 315 °C (600 °F), exhibited cracking. Upon increasing the baking temperature to 370 °C (698 °F) and further to 440 °C (824 °F), none of the welds exhibited cracking. The dye penetrant appearance of a cracked Y-groove sample is

Table II "Y-Groove" Test Results.

Baking Temperature	Teledyne Weld Pad FN	Y-Groove Weld Metal FN	No. of samples	No. of cracked samples
	48	73.5	2	2
Unbaked	74	9 5	2	1
(Green Condition)	9 2	103	1	1
	4 8	73.5	1	0
315°C(600°F)	7.4	9.5	1	0
	9 2	103	5	2
	4 8	73.5	-	-
370°C(698°F)	74	95	-	
	9 2	103	1	0
	48	73.5		-
440°C(824°F)	7 2	9 5	-	-
and the second of the second	9 2	103	1	0

shown in Fig. 14.

From the "Y Groove" test results, it is considered that a measured diffusible hydrogen content of approximately 15 ml/100 gm is required to cause cracking at low FN (73 and 96) whereas 10 ml/100 gm is the approximate critical level for 103 FN deposits. From the results of this study, the approximate relationship of diffusible hydrogen, the FN of the weld metal and the crack sensitivity is shown in Fig. 15 based on "Y-Groove" testing. The critical level of diffusible hydrogen content for weld metal cracking decreases with an increase in FN. To prevent hydrogen induced cracking in weld metals of FN 73.5 and 95, the diffusible hydrogen content must be less than approximately 15 ml/100 gm. In the case of a ferrite level of FN 103, it must be less than approximately 10 ml/100 g. (The diffusible hydrogen contents are based on extraction at 950 °C for 1 hour).

c) UT-Modified Hydrogen Sensitivity Testing

The results of hydrogen assisted cracking susceptibility using the UT-Modified hydrogen sensitivity tests were inconclusive because the level of hydrogen introduced through the arc was only 2-3ppm. Further tests are suggested with a higher level of hydrogen in the welding arc.

d) FN determination

In this study, the Magne-Gage was used to determine FN. Calibration for duplex stainless steel weld metal was accomplished according to the standard procedure recommended by the AWS committee on filler metal. The sample surface was ground smooth before measurement.

The measured FN on the UTK weld pads, BOP (bead on plate) standard hydrogen analysis samples, BOP glass capsule hydrogen analysis samples, "Y-Groove" weld metal and the base plates are summarized in **Table III** (the Teledyne weld pad was made by using the same electrodes). The FN of the UTK pad is virtually identical to FN of the Teledyne weld pad. The FN of the BOP-standard hydrogen sample. BOP-glass capsule hydrogen sample and "Y-Groove" weld metal show almost the same FN. These single pass welds show a slightly higher FN than the pad welds (dilution effect).

e) Microstructural investigation

The microstructure of the base plate, weld metal and HAZ are shown in **Figs. 16** to **19**. Figure. 16 shows that the microstructure of the base plate is typical of that of duplex stainless. An elongated austenite phase is evident in the ferrite matrix. Ferrite appears dark in these micrographs. Weld metal microstructures of FN 73.5, 95 and 103 are shown in Fig. 17 through 19 respectively. In

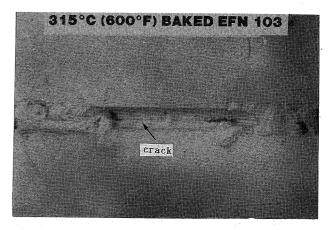


Fig. 14 Dye Penetrant Appearance of a Cracked "Y-Groove" Sample, FN 103, Electrode Baking Temperature 315 °C.

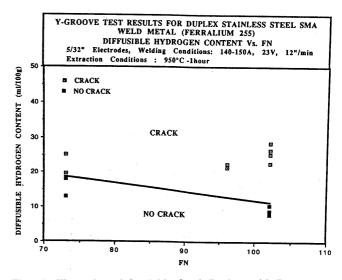


Fig. 15 Illustration of Crack-No Crack Regions with Respect to Diffusible Hydrogen for Different FN SMAW Ferralium 255 Weld Metals.

Table III Ferrite Number (FN) Measured on Various SMAW Ferralium 255 Welds Using Magne-Gage.

Teledyne Weld Pad	UTK Weld Pad	BOP-H ₂ Analysis Standard Sample	BOP-H2 Analysis Glass Capsule Sample	"Y-Groove" Weld Metal	Base Metal 1/2"Plate	Base Metal 1 "Plate
48 (36)	56(41)	70 (49)	73 (50)	73.5 (51)		
74 (51)	79.5(54)	96.5 (64)	96 (64)	95 (63)	85 (57)	88 (59)
92 (61)	91.5(61)	103 (68)	102 (67)	103 (68)		100

(): Ferrite Volume % by TWI equation

the higher FN weld metal the austenite content is decreased and its morphology changes to granular and acicular and a fine precipitate appears in the ferrite. The morphology of "Y-Groove" cracks in weld metal is shown in Fig. 20. The cracks propagate in the ferrite in a zigzag

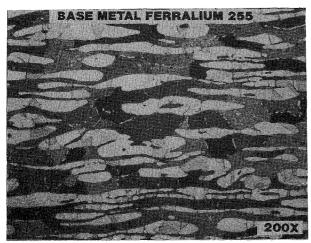


Fig. 16 Microstructure of Ferralium 255 Base Plate, 200 X, Kallings Reagent.



Fig. 17 Microstructure of Ferralium 255 SMA Weld Metal, FN 73.5, 200 X, Kallings Reagent.



Fig. 18 Microstructure of Ferralium 255 SMA Weld Metal, FN 95, 200X, Kallings Reagent.

mode. It is considered that the austenite phase has higher ductillity and toughness than the ferrite phase and thus the austenite phase is resistant to crack propagation. The HAZ structure is shown in Fig. 21. Coarsened grains are observed near the fusion boundary. A granular and particulate second phase appears to be present in the ferritic regions near the fusion boundary. The granular

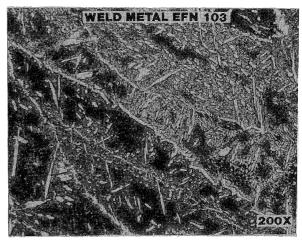
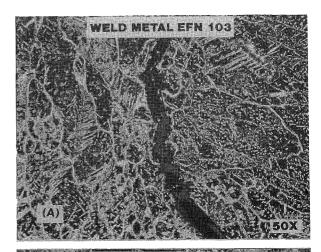


Fig. 19 Microstructure of Ferralium 255 SMA Weld Metal, FN 103, 200 X, Kallings Reagent.



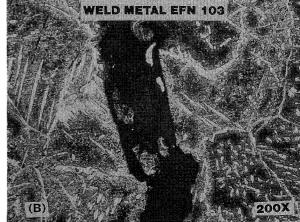


Fig. 20 Morphology of Crack in Ferralium 255 Weld Metal, FN 103, (A) 50 X, (B) 200 X, Kallings Reagent.

regions within the large ferrite grains are austenite. However, the particulate precipitates appear to be carbides, nitrides or carbonitrides. These particulate precipitates are also present in the ferrite grains in the HAZ away from the fusion line.

The fracture surface morphology from cracked "Y-Groove" samples extracted from FN 73 and 103 weld metal tests are shown in the SEM micrographs in Figs. 22 and 23. The cracking at the two ferrite levels appears to be quite similar in morphology with the grain size and orientation in the weld metal evident. The higher

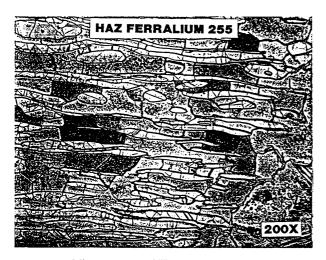
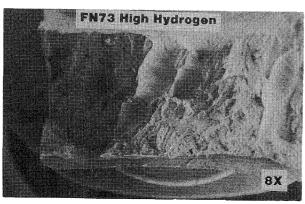


Fig. 21 Microstructure of HAZ, 200X, Kallings Reagent.



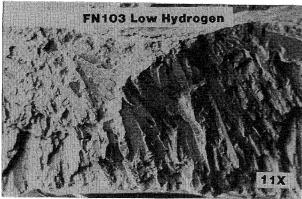


Fig. 22 SEM Fractographs of "Y-Groove" Sample Test Cracks with Different FN and Hydrogen Contents.

magnification fractographs in Fig. 23 clearly show that the fracture is cleavage in the ferrite phase and ductile dimple in the austenite which envelopes the ferrite grains

Summary

- (1) The diffusible hydrogen in duplex stainless steel weldments determined at 45 °C (weld metals of FN 73, 96.5 and 103) is negligibly small.
- (2) The diffusible hydrogen content increases with an increase in extraction temperature up to 600 °C and remains essentially constant at 25 ml/100 gm to 950 °C for an extraction time of 1 hour.
- (3) The diffusible hydrogen content decreases as a function of time for times greater than 1 hour at extraction temperatures of 600 and 950 °C.
- (4) The diffusible hydrogen content for extraction at $200\,^{\circ}\text{C}$ for 72 hours ($> 20\,\text{ml/}100\,\text{gm}$) approaches that measured for $600\,\text{and}\,950\,^{\circ}\text{C}$ for 1 hour.
- (5) Baking of "green" electrodes at temperatures > 315 °C reduces the diffusible hydrogen content to < 10 ml/100 g.
- (6) "Y-Groove" test results show that cracking occurs in weld metals of FN 73.5, 95, and 103 made with "green" unbaked electrodes (25 ml/100 gm

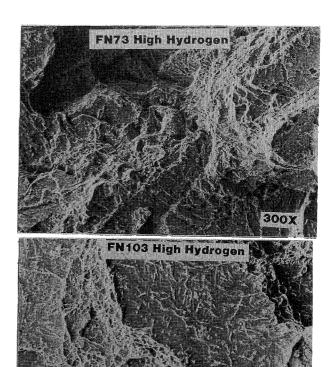


Fig. 23 SEM Fractographs of the Fracture Morphology for the Cracked 73 and 103 FN "Y-Groove" Tests.

- diffusible hydrogen). Cracking does not occur in weld metals of FN 73.5 and 95 when "green" electrodes baked above 315 °C are employed. For weld metal of FN 103, a "green" electrode baking temperature of 370 °C is needed to eliminate cracking in the "Y-Groove" test.
- (7) The critical diffusible hydrogen content for weld metal cracking is estimated to be 15 ml/100 gm for FN 73.5 and 95 weld metals and 10 ml/100 gm for FN 103 weld metal.
- (8) Post heat treatment at 200 °C for 72 hours may be effective in preventing delayed hydrogen induced cracking of duplex stainless steel weld metals since the hydrogen content appears to be reduced to < 10 ml/100 gm by this treatment.

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