CHARACTERISATION OF WELD METAL FOR 3.5% NICKEL STEEL IN MANUAL METAL ARC WELDING AND ITS STRESS CORROSION AND LOW CYCLE CORROSION FATIGUE PROPENSITIES

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INTRODUCTION

3.5% nickel steel possesses a combination of high strength and resistance to brittle fracture which makes it suitable to be widely used for the fabrication of storage tanks designed to be used at between 223K and 172K (pressure vessels to produce ethylene or propylene from naptha in chemical plant)[1-4]. These structures are fabricated, typically, by manual metal arc welding with coated electrodes. Though many are marginally electrodes satisfactory at 172K, the weld metal toughness is the limiting criteria for such grade of steel. Globally, failure of 3.5% nickel steel all weld containers are known. During 60's the failure of LPG tankers led to R & D works in USA and Japan [5]. To enhance the safety of these structures, under cryogenic service conditions. adequate fracture properties, particularly low temperature toughness, need to be assessed

It is now well established that the presence of acicular ferrite in the primary microstructure of low alloy steel weld deposits leads to improved toughness [6, 7].

In this project work, assessment of cryogenic toughness properties of weld metal produced by microstructural development through controlled heat input and thereby inducing the effect of acicular ferrite has been attempted. Subsequently, environment sensitive failure effects were characterised as stress corrosion (SC) and low cycle corrosion fatigue (LCCF) failure.

EXPERIMENTAL PROCEDURE

Experimental steel and joint preparation

3.5% nickel steel plates of sizes 450mm x 160mm x 12mm were procured in the normalized and tempered condition. Normalizing was carried out at a temperature of 1173K and tempering at 873K followed by cooling in air. The chemical composition of 3.5% nickel steel is given in Table - 1.

The plates in as-received condition were cut and shaped to make

experimental steel blocks of size 200mm x 70mm x 12mm. The edges of the blocks were grooved with single-vee shapes having included angle of 70°. The groove design is shown in Fig. 1a.

Welding Parameters

All the joints for the present study were made by manual metal arc welding process with D.C. power source using two types of coated electrodes, namely Type - I (indigeneously available) and Type -II (imported). Before welding the coated electrodes were dried at a temperature of 573K for one hour and all the welding were made in flat position using identical welding parameters. (Table - II).

All completed welds were inspected by X-ray Radiographic Technique. The sound weld blocks were then sectioned into slices to make samples for mechanical, corrosion and microstructural studies.

Table - I								
	С	Si	Mn	Ni	S	Р		
Base Metal	0.12	0.20	0.70	3.60	0.02	0.03		



Mechanical Testing

Both transverse and all-weld tensile testing were carried out at room temperature (298K) in an Instron Universal Testing Machine of 10,000 Kg (10 ton) capacity and with a cross head speed of 3.3 x 10⁻⁵m/sec.

Charpy Impact Testing of the base and weld metals, prepared with two different electrodes, was carried out by Avery Charpy Impact machine at 172K. For weld joint, the V-notch was made perpendicular to the weld axis.

Microhardness Testing was carried out in Wolpert Testing Machine using 0.025Kg load and knoop indentor on polished and flat samples. Hardness measurement was taken at different positions across the weld metal (Fig.2).

Specimens for optical metallography were obtained from the transverse direction of the weld. (The quantitative assessment of the microstructure was made using a point counter. A minimum of 500 points were measured on each The microstructural specimen).

Table - II : Welding Parameters

Parameter	Value
Current (amp)	110-130
Voltage (Volts)	32
speed (m/sec)	2.08 x 10 ⁻³
Polarity	DC Electropositive

constituents were identified according to the classification of IIW, [8]

Environment sensitive failure tests

In order to assess stress corrosion cracking (SCC) propensities, tests were carried out in slow strain rate testing machine (SSRT) and corrosion fatigue (CF) tests at ambient

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and cryogenic temperatures at low cycle fatigue frequency of 0.2 Hz were carried out in specially designed fatigue machine. Single edge notch type test specimens (Fig. 3) were prepared from (i) the base and (ii) the weld metals (W1 & W2).

SSRT mode of testing was carried out at cross head speed (CHS) of 10⁻⁵mm. s⁻¹ (ferrous alloys testing at similar CHS, in sensitive environments, generates recordable environment sensitive failure effects on crack velocity and fracture appearance).

Low cycle fatigue (LCF) mode of testing at a frequency of 0.2 Hz (facilitates corrosion reaction time), at σ max = 0.1 of the breaking load of the test specimen and at "R" value of 0.1, was carried out at ambient and 193K (±2K) temperatures for W2 material.

Table - III Chemical Composition of Weld Metals with different Electrodes

	1	C	Si	Mn	Ni	S	P	
Weld Metal (W1) (Type-I electrode)		0.045	0.20	0.65	4.13	0.007	0.01	
Weld Metal (W2) (Type-II Electrode)	-	0.038	0.266	0.47	3.93	0.0076	0.009	

Cryogenic test cell for the LCF mode of testing was designed to contain specimen in test environment-methanol in a chamber surrounded by liquid nitrogen vapour chamber and vacuum chamber (vacuum in excess of 10⁻² torr was maintained during test).

Crack extension was monitored (a) optically, using monocular telescope with vernier scale attachment and (b) by direct current potential drop (DCPD) technique adopted for these series of tests. Scanning electron microscopy (S.E.M.) of charpy impact and corrosion test failure specimens was carried out to study fracture micromechanism.

RESULTS AND DISCUSSION

Characterisation of weld metal properties

The chemical composition of the weld metals is given in Table - III. There are small differences in the amount of C, Mn and Ni contents in the base and the two types of weld metals.

Table -	IV	: Quantitative	Metallographic	Data of V	Neld Metal u	using differer	t Electrodes
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Material	Different Fe	errite Morphologie	s (Vol %)
	G.B.F.	P.F.	A.F.
W1	26.2	17.60	56.6
W2	28.4	20.46	51.4

A.F. = Acicular Ferrite G.B.F. = Grain Boundary Ferrite P.F. = Polygonal Ferrite

Table - V											
Transverse	Tensile	Testing	Data	of	base	metal	(3.5%	Nickel	Steel)	and	Weld
Metals using different Electrodes											

Material	0.2% Y.S. (MPa)	U.T.S. (MPa)	% Elongation	% Reduction In area	Remarks
Base Metal	385	509	34	75	
W1	391	519	28	72	broke at base metal
W2	384	525	24	73	broke at base metal



The microstructure of the base metal consists of polygonal ferrite (light area) as shown in Fig. 4. Typical microstructure of the weld prepared by using Type-II electrode (W2) is presented in Fig. 5. The results of the quantitative assessment of the microstructure are given in Table -IV. This comparison shows that the welds consist of relatively high proportion of acicular ferrite with various amounts of grain boundary and polygonal ferrites.

Transverse and all-weld-metal tensile properties of different weld metals using two different types of electrodes are listed in Tables-V & VI respectively. All transverse tensile specimens broke at the base metals indicating strengthened weld metals compared to the base metal.

The charpy V-notch impact toughness values at 172K are shown in Table-VII. The better impact toughness at 172K associated with the weld metal (W1) produced by Type I electrode was probably due to higher amount of acicular ferrite (Table-IV) and inherently associated with lower strength of the weld metal (W1).

All-Weld	Tensile Testing	Data of Weld m	etals using diffe	rent Electrodes
Material	0.2% Y.S. (MPa)	U.T.S. (MPa)	% Elongation	% Reduction in area
W1	411	532	30	71
W2	457	551	30	- 72

Table VI All-Weld Tensile Testing Data of Weld metals using different Electrodes

 Table - VII

 Charpy Impact Testing Values of Base Metals and Weld Metals using different electrodes at 172K

Material	Charpy Impact Value at 172K (J)
Base Metal	153.9
W1	30.4
W2	24.5

Table - VIII Crack Velocity (C.V.) of Base Metal and Weld Metal Tested in SSRT mode at Laboratory Temperature (297K)

Material	Environment	Average Crack Velocity (mm/sec)					
		Stage I	Stage II	Stage III			
Base Metal	Air Methanol	# (2 x 10 ⁻⁶) 6 x 10 ⁻⁶	(1 x 10 ⁻⁶) 1.4 x 10 ⁻⁴	Tearing and Yawning			
W1	Air Methanol	# (7 x 10 ⁻⁷) 3 x 10 ⁻⁶	(7.5 x 10 ⁻⁶) 2 x 10 ⁻⁴	Tearing and Yawning			
W2	Methanol	4.5 x 10 ⁻⁶	6 x 10 ⁻⁴	Tearing and Yawning			

Values given in bracket indicates coincidential approach to relate with environmental effect values.

Similar improvement of toughness with lowering of strength for similar microstructural constituents was previously reported [9].

The micromechanism of failure at 172K was studied by SEM. Evidently, failure (charpy) in the base metal was by transgranular cleavage (TGC) interspersed with discontinuous areas of micro void (MVC) coalescence mode. Qualitative assessment of fracture mode of weld metals produced by the different types of electrodes general, crack revealed, in propagation by TGC mode with

discreet and varying amount of MVC. Fig. 6 represents the fracture mode of the base metal and Fig. 7 for W2.

Environment sensitive failure tests

a) SSRT in (i) Air and (ii) Methanol: Data obtained from tests carried out in "slow strain rate testing" mode, at a CHS of 1 x 10⁵mm s⁻¹ in methanol environment at ambient laboratory temperature of 297K (approx) are related as in Fig. 8 and Table - VIII. Data obtained from these tests in methanol environment for the base and W2 material indicate effects of the test environments. Distinct differences were apparent when

increasing load and increasing crack length with testing time were compared in the tests carried out in air and methanol environments in that i) While tests in air monotonously increased stress and crack length in specimen with time till failure of the specimen had occurred, effect of methanol environment on the other hand, both on advancing crack length and increasing load with time, when related as shown schematically in Fig. 9, indicates crack progression in stages I, II & III till failure of the specimen had occurred. ii) Calculated crack advancement, i.e., crack velocity (CV), detailed in Table -VIII, indicates faster rate of crack growth in tests carried out in methanol environment. iii) Stage III crack progression in all specimens were discarded as discreet vawning of the crack was observed.

Fracture Analysis : In general fracture mode observed for tests in air was typically by microvoid coalescence (MVC) as in Fig. 10. Fracture appearance in specimen tested in methanol in the base and the weld metal (W2) are depicted in Figs 11 & 12. Initially, crack progression was by MVC at the notch root gave in to quasi brittle zone (Fig.11) outlined with flat ribbed facets indicates faster rate of crack growth in this region. Recorded crack growth data obtained from (base metal) the test indicates this area to have been generated effectively under environmental (methanol) influence.



b) Low cycle fatigue (LCF) testing in (i) Air and (ii) Methanol

Data obtained from selected tests are graphically presented in Fig. 13 and Table - IX.

Irrespective of the test metal composition, i.e., base metal or weld metal (W2), relative comparison of the recorded data highlights the following characteristics : i) In airtests load-time relationship is a continuous slope to failure as expected, under constant displacement type fatigue loading [10, 11, 12]. ii) Crack growth rate (CV) in Air and MeOH remains similar in stage I. In stage II CV has effectively increased in methanol environment (Fig. 13). iii) Tests in acidified methanol did not significantly affect CV in the base

metal vis-a-vis the weld metal tested in methanol. iv) Recorded low CVs in the gradually flattening out stage III region, for all test variation employed here in MeOH & MeOH + HCI environments indicate noncracking conditions, as expected under constant displacement mode of fatigue testing [11,12].

Fracture Analysis : Fracture mode in air was by MVC. Environmental effects on fracture mode are shown in Figs 14(a) & (b) similar to those





Fig.10 : S.E. fractograph of SSRT failed base metal (3.5% Ni steel) in air, x 500



Fig.12 : S.E. fractograph of SSRT failed weld metal (W2) in methanol, x 380



Fig.14a : S.E. fractograph of LCF failed weld metal (W2) in methanol at laboratory temperature, x 500



Fig.11 : S.E. fractograph of SSRT failed base metal (3.5% Ni steel) in methanol, x 380



Fig.13: Low cycle corrosion fatigue testing (f=0.2 Hz) of weld metal (W2) in methanol



Fig.14b : S.E. fractograph of LCF failed weld metal (W2) in methanol at laboratory temperature, x 30

obtained in SSRT tests. Striations (Fig. 14b) observed are due to crack arrest markings as are generally observed in fatigue tests in air denoting mechanical aspiration. Corrosion fatigue effects are depicted in Fig. 14(a) including crack arrest phenomena separating quasi brittle continuous zones.

(c) Low cycle fatigue mode of testing of weld metal specimen (W2) in methanol environment at cryogenic temperature was carried out o characterize the effect of cryogenic temperature (e.g. at 193K) on the weld metal (W2) in a solvent environment.

The Test : LCF tests were carried out using SEN (Fig. 3) specimen contained in a specially designed test cell. Liquid nitrogen vapour was sparingly introduced in the cooling chamber to obtain the test temperature maintained at 193K (\pm 2K). Selected test temperature was 193K, close to the charpy test temperature at 172K which revealed



Table - IX Crack Velocity (C.V.) of Base Metal and Weld Metal Tested in LCF at Laboratory Temperature (297K) and Cryogenic Temperature (193K)

Material	Environment	Average Crack Velocity (mm/sec)				
		Stage I	Stage II	Stage III		
Base Metal	Methanol (294K) (+ 1% HCl)	6 x 10 ⁻⁷	1 x 10⁴	1 x 10 ⁻⁶		
	Air (297K)	# (2 x 10 ⁻⁷)	(4 x 10 ⁻⁷)	1 x 10 ⁻⁶ (close to failure)		
Weld Metal (W2)	Methanol (294K)	4 x 10 ⁻⁷	9 x 10⁵	3 x 10-6		
	Methanol (193K)		1.2 x 10⁵			

Values given in brackets indicates coincidental approach to relate with environmental effect values.

brittle quasi-cleavage mode (>90% of the fractured surface).

Testing Load $(\sigma_{max} \text{ and } \sigma_{min})$: Introduction of liquid nitrogen vapour in the cooling chamber, surrounding the experimental chamber containing the test specimen immersed in the test environment-methanol, had decreased the test temperature (recorded - specimen surface temperature close to the notch) with concomitant increase in σ_{max} (Fig 15), from the maintained σ_{max} value at the ambient test temperature (297K) due to the constraint imposed by (i) the characteristic elastic/plastic domain change in the test material at cryogenic temperature and (ii) the constraint imposed by the constant displacement mode of fatigue loading (tension-tension) of the test specimen. Similarly, any increase in test temperature had decreased σ_{max} along A-B as in Fig. 15. Changes in σ_{mn} with temperature was similar, effectively maintaining the consistency of the 'R' value at 193K. Tests in cryogenic temperature were continued at the achieved higher σ_{max} and σ_{min} values till failure had occurred progressively.

Cryogenic Test Data : Due to technical failure at the time of the experimental work DCPD system could not be engaged to monitor crack extension in the specimen. However, data obtained from the examination of fracture surface



obtained from cryogenic test revealed separate zones developed under the prevailing test conditions were recorded and crack velocity was assessed (Fig.16). In the figure 16,

Area in1 \rightarrow Notch

- Area in 2 \rightarrow Z1 Precracked zone (5.0 mm)
- Area in 3 → Z2 Environment affected area generated by CF (2.61 mm)
- Area in 4 \rightarrow Z3 Instant (Fast) fracture zone (7.92 mm).

[All measurements were carried out (i) using monocular microscope and counter checked by SEM].

Fracture Analysis : In scanning electron microscopic examination precracked zone (Z1 - generated by LCF) revealed guasi-ductile MVC mode typical of Q & T low alloy steel fracture under fatigue loading in air (Fig.10). This zone terminated at zone 2 (Z2). Z2 revealed quasicleavage facets interspersed with MVC. The fracture facets appeared (Fig. 17) blunt as would be expected under conjoint action of the test environment and cyclic loading sequences in the corrosion fatigue crack progression region. Areas covered under brittle facets increased along crack progression towards Z3, the fast fracture zone [the effect of cyclic (tension-tension)

loading in methanol environment]. Z3 revealed quasi-cleavage, large cleaved zone with river line markings [Figs.17(b)&(c)]. Number of cleavaged facets increased with crack progression towards the terminal edges of the specimen. Fracture appearance in Z3 appeared similar to that observed in charpy specimen (W2) tested at 193K (± 2K) (Fig.18).

Consistent with the fracture zone profile, as in Fig. 16, it is assumed that the recorded total time to failure (57.5 hrs of the specimen) was required for the sub-critical crack to progress in environment sensitive failure dominated Z2 only. This zone is identified as due mainly to low temperature CF effect. Assessed crack velocity (CV) in this zone (Z2), 1.2 x 10⁻⁵ mm s⁻¹ is a decade lower than the CV calculated for LCF in methanol at ambient test temperature found experimentally in this work.

Cracking potential at $K_{o}cf$ i.e CF affected zone 2 can be related to :

 $K_{QCF} = \{ [E/(1-v^2)] [\gamma_p - ZF \rho \delta n/M]^{\frac{1}{2}} \}$

Where

- E = Young's Modulus
- v = Poisson's Ratio
- γ_p = Plastic work necessary for crack progression (mechanical work)
- Z = Valency of the solvated ions.

F = Faraday's constant.

- ρ = Density (of the alloy concerned)
- δ = Approximate to COD on height of advancing crack front.
- M = Molecular mass of the metal
- η = Electro chemical potential

[13,14]

A reduction of the γ_p , plastic work term, will result from an increase in the effective yield stress due to lowering of temperature. This would mean an **incresed CV** in a CF environment if η is unaffected. However, value of η will decrease with lowering of temperature or decreasing time required for η potential to be effective [10]. In this work lowering of test temperature by 100K (to 193K) has increased elastic stress level by :

(E X Th. coef.) X Temp. difference where

E = young's modulus Th. coef. = Thermal Coeffeicient (*E* & Th. coef. is in Giga Pascal)

= [(2 x 10") (2 x 10⁻³] 100 = 400 Mpa (approx.)

i.e. increased stresses viz. room temperature would be available for plastic work at 193K.

This increment in stress level at 193K is expected to increase CV in Zone 2. However, in balance effect of lowering the temperature decreased corrosion potential η by



Fig.17a : S.E. fractograph of LCF failed weld metal (W2) in methanol at 193K at - Z2, x 500 Fig.17b : S.E. fractograph of LCF failed weld metal (W2) in methanol at 193K at - Z2-Z3, x 500



a margin which has decreased corrosion fatigue crack velocity (at 193K) compared with CV at room temperature where γ_p term is dominant.

In zone 3 (Z3) crack progression was instantaneous - as in quasi-cleavage brittle failure (known to be at the speed of sound) [14]. The dominating fracture mode observed in this zone is large cleavage facets occasionally terminating at grain boundaries (Fig. 17c). Transgranular crack progression was also evident (Fig. 17b). Crack arrest markings evident in Z2 (Fig. 17b), for instance, were not observed in Z3 (Fig. 17c) at comparable resolution (SEM), which also indicates subcritical crack growth under corrosion fatigue conditions as in Z2 of W2 material was not present in zone 3.

CONCLUSION

The following conclusion with regards to the characterisation of specific properties of the tested weld metals can be drawn : i) In general,

all-weld metals exhibit poor charpy impact toughness at 172K. ii) Scanning electron microfractographs revealed, in general, crack progression by transgranular cleavage mode with discreet and varving amount of micro void Prevalence coalescence. of secondary cracking in fracture surfaces obtained from weld metals and the amount of transgranular guasi cleavage type fracture mode indicates greater brittle failure propensities compared with MVC dominated fracture surface obtained from base metal tested under similar test conditions.

From the series of environment sensitive failure tests (stress corrosion and low cycle corrosion fatigue tests), limited in some respects, following areas, as concluding remarks, have emerged :

In all three modes of testing, namely (i) SSRT, (ii) LCF and (iii) cryogenic (LCF), effects due to the test environment-methanol, a solvent, were apparent in that -

- a) Crack progression in methanol environment was faster by more than a decade vis-a-vis the CV recorded for tests in air,
- b) In SSRT & LCF testing in methanol environment and at the test parameters used in this experimental work stress corrosion and corrosion fatigue effects were identified in stage II zone where CV was at

maximum. Examination of fracture surface in this zone revealed quasi-brittle fracture mode, secondary cracks, crack arrest markings and corrosion effect blunting the fracture facets are typical of fracture modes revealed in SC & CF dominated failure of ferrous material, in general,

- c) In LCF modes of testing brittle to ductile transition at critical load/stress, similar to K_{ocF} , was identified, a parameter indicating safe loading condition in specific environmental use,
- In these cryogenic tests, the effect of methanol environment at cryogenic temperature (193K) revealed CF cracking propensities and observed catastropic failure also indicated unpredictable life performance of these welded material in cryogenic use,
- e) Crack progression in SC and CF continuum, in weld metal, as shown in this work demand critical examination to avoid such occurrences. Solvent such as methanol, under critical loading, monotonous and cyclic, as used in this experimental work, could initiate environment sensitive brittle failure in weld metal continuum in 3½% Ni steel.

In order to avoid unpredictable failure in these material

environment systems it would be prudent to ensure uses :

- When under monotonous loading conditions (e.g. static reserve tank), at ambient temperatures, at strains on the welded section pertaining to Stage I (steady state sub-critical crack growth), enables greater predictability to failure,
- ii) and when under cyclic loading conditions (e.g. durina transportation. continuous loading and unloading) at temperatures higher than 193K (predictably, decrease amount of brittle fracture mode), at strains pertaining either to Stage I (steady sub-critical crack growth) or Stage III (Kocc - no cracking at ambient temperatures) and at loading frequencies higher than 0.17 Hz decrease materialwould environment electrochemical/ chemical reaction time improving life performance.

ACKNOWLEDGEMENTS

Authors are thankful to D.S.T. to provide financial support and M/s ESAB India Ltd., Khardah, 24-Parganas (N), W.B. for their kind cooperation in assisting with testing facilities.

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Pride of Nation, Nobel Laureate Dr. Amartya Sen we felicitate you

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