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Effect of Desulfovibrio Desulfuricans on Stress Corrosion Cracking of Ductile Iron in Slow Strain Rate Testing Mode

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***Abstract*— Stress corrosion cracking of ductile iron was investigated in presence of sulphate reducing bacteria (SRB). Notched tensile specimens were subjected to slow strain rate testing in air, Postgate's medium with and without SRB cultures. Susceptibility to SCC as was determined by average crack velocity may be indexed as: SRB + Postgate's medium > Postgate's medium > Air. Flat fracture surface of specimens signifies loss of ductility and an increasing amount of brittleness in SRB containing medium compared to Postgate's medium and air. Micro cracks on the fracture surface tested in SRB signify hydrogen induced crack propagation.**

Keywords*— Ductile iron; stress corrosion cracking; SRB; hydrogen embrittlement; brittleness

I. INTRODUCTION

Environment sensitive failure (ESF) through stress corrosion cracking (SCC) and associated hydrogen induced cracking (HIC) occur unpredictably in metallic materials [1]. These cracking may propagate inside the structure undetected until a sudden catastrophic failure takes place. Being acted by the synergic action of environment and under stress condition it is seemingly the most dangerous form of corrosion-assisted failure [2]. ESF in ferrous metal are very frequently observed throughout the world. During the last four decades
numerous research works [3-12] have been carried out in the development and standardization of laboratory methods of quantitative prediction methodologies for the determination of ESF effects in metallic material in liquid and gas environments
at different experimental conditions. These studies help to understand environmental reactions that result in understanding of crack nucleation and propagation, effect of metallurgical structure on the dynamics of ESF, effect of mechanical aspiration, stress-strain on cracking dynamics, etc.

Stress corrosion cracking is an aggravated spontaneous damage or loss of mechanical properties of the materials that occurs because of the simultaneous presence of tensile stress, a critical environment, and a susceptible material [1]. The widely accepted SCC mechanisms suggest that the cracking susceptibility may be governed by metal dissolution and oxide film growth at potentials to the active-passive transition or may involve hydrogen embrittlement [1, 2]. The progress of embrittlement is influenced by various factors such as adsorption, absorption and diffusion of hydrogen atom, concentration of corrosive ions and also influenced by bacteria.

Microbiologically influenced SCC of iron and its alloys has received increased attention in recent years [13-17]. The importance of microbial synergy on SCC has recently been reconfirmed in studies of low carbon steel and high carbon ferrous metal [4, 7]. Some specific bacteria induce in pitting, crevice corrosion, stress corrosion cracking and under-deposit corrosion on ferrous metal [13-15].

Sulphate reducing bacteria (SRB) is considered as the major troublesome group among all microorganisms involved in microbiologically influenced SCC. This group of bacteria is one of the most important causes for SCC. Considerable scientific attention has been devoted to investigate the microbial pitting and SCC process of steel and iron in the presence of SRB [18]. SRB are defined as obligate anaerobes which obtain energy for growth from the oxidation of organic substances, using sulphate as the external electron acceptor and reducing sulphate to sulphide [19]. SRB are also non-fermentative anaerobes that obtain their energy for growth from the oxidation of organic substances using inorganic nitrate or sulphur oxy-acids or nitrate as terminal electron acceptors whereby sulphate is reduced to sulphide [20] which in turn reacts with metals to produce metal sulphide as corrosion products.

The presence of microorganism, SRB in the vicinity of the solid surface often leads to highly localized changes in the electrolytes and hydrogen pressure by producing hydrogen sulphide (H_2S), hydrogen ion (H^+), HS^- and S^{2-} [21, 22]. Even low SRB counts (10^4 cell mL^{-1}) in soil or sea water influenced structural failures in pipe lines [23].

Although there have been better understanding of the stress corrosion cracking (SCC) mechanism with the help of environment analysis, metallography, fractography, etc., the diversity in the failure modes in presence of SRB and the associated mechanisms are highly complex, controversial, not completely understood and therefore the phenomenon should be further studied and correlated.

II. MATERIALS AND METHODS

2.1 Test material and specimen

The susceptibility of ductile cast iron to SCC in biotic and abiotic media was investigated by employing slow strain rate testing (SSRT). Specimens were sampled following the NACE TMO 0284-2003 standard test method [24]. Single edge notched (SEN) type flat tensile specimens were machined (milling cutter) to dimensions 84 mm long, 30 mm wide and 2.9 mm thickness with 60º notch situated at the mid-length position. The surface of the specimens were abraded with a grade 1200 emery paper, followed by cleansing in ultrasonic bath in distilled acetone for 10 minutes. The whole assembly was suitably gripped on the testing machine through griping bolts. The chemical composition of the material (as obtained from Electro-Steel Casting Limited, Kharda plant, Kolkata) in mass/mass (%) is given in Table 1.

2.2 Test Method

The method for getting faster information over constant load and constant strain method in laboratory is accelerating the SCC process by SSRT of specimen during exposure to susceptible environment. The application of slow dynamic strain exceeds the elastic limit and thus assists testing of SCC susceptibility. This accelerating technique is consistent with various proposed general mechanisms of SCC. The most significant feature of slow strain rate testing is the magnitude of strain rate. If the strain rate is too high, film formation cannot keep pace with the mechanical plastic strain and test specimen fails by ordinary cup-and-cone ductile fracture. Therefore a critical strain rate must be used as proposed by C. D. Kim and B. E. Wilde [25] where a minimum ductility is observed, characteristic of SCC. The most appropriate strain rate for promoting SCC will vary from system to system. It has been reported [26] that most of testing has done at critical strain rates ranging from 10^{-5} to $10^{-7}s^{-1}$. Here SSRT experiments have been carried out at the strain rate of 1×10^{-5} s^{-1}. Applied load is constantly monitored by load cell impulse feed recording system (Make: Syscon, Model: SPL, SI. No. 4125).

2.3 Test solutions and exposure conditions

All solutions were prepared after a thorough deoxygenated step by passing nitrogen gas for 30 minutes using nutrient-rich medium, consisting of 0.05g KH_2PO_4, 1.0g NH_4Cl, 0.06 g $CaCl_2$,$6H_2O$, 0.06 g $MgSO_4$,$7H_2O$, 4.5 g Na_2SO_4, 10 ml 60% sodium lactate as an electron donor and carbon source yeast extract and 0.39 g $C_6H_5CO_7Na_3$ per litre sterilized water. The solutions were autoclaved at 121°C for 20 minutes then stored at room temperature. 0.004 g $FeSO_4$, which is not autoclaved, is added prior to inoculation and pH adjusted to 7.2-7.4 and the resulting solution is called modified Postgate's medium. The solution with SRB was then poured into the test cell after proper dilution with sterilized water.

2.4 Test Cell

The tests were carried out in cells made of (Pyrex) glass cylinder at one end with rubber stopper through which the test specimen passed. The cell contained the test environment liquids at laboratory temperature.

2.5 Microbial culture

The sulphate reducing bacterium desulfovibrio desulfuricans, NCIM No. 2047 was used in this study. To estimate the number of sessile bacteria, bio-film was removed from the total surface of the one specimen exposed to the medium with a sterile cotton swab that was then immersed in 4 ml modified Postgate's medium ,vortex to disperse the SRB. The resulting bacterial suspension was then serially diluted from 10^{-1} to 10^{-10} using sterilized modified Postgate's medium. Sessile and planktonic SRB counts were determined by plate count method using nutrient agar medium.

2.6 Measurement of crack extension

The chevron groove containing the crack was properly cleaned to remove oxide film from the metal surface with hexamine hydrochloride solution (2 gm hexamine G-R grade in 1L 3N HCl solution) then with distilled water and finally by acetone. The crack extension was monitored optically, using 15X monocular microscope with vernier attachment (resolution 0.01mm) and crack progression was continuously recorded against an electronic time recorder with controls. The recording was discontinued when the test specimen either failed or yawning of the crack profile was observed. The fracture surface of the test close to the edge was examined using similar optical monocular microscope to measure the zone of overload failure and the length of the crack progression under test conditions. This measurement was taken for the fracture surface obtained from each test specimen.

2.7 Scanning electron microscope

Selected fracture surfaces were examined in scanning electron microscope (JEOL, JSM-6360). After cleaning with hexamine hydrochloride solution fracture surfaces were systematically scanned from the root of the notch where cracking had initiated then along crack propagation path, i.e., along the width of the specimen to other extremity of the fracture surface.

III. RESULTS & DISCUSSION

A thick and black colure film of corrosion products and patchy bio-film [27-29] was observed to develop on the ductile iron specimens exposed to the biotic environment of SRB desulfovibrio desulfuricans in modified Postgate's medium. Fig. 1(a) and 1(b), illustrate the presence of desulfovibrio desulfuricans cells on the ductile iron surface. Fig. 1(a) reveals several colonies of SRB. Chronological growth curves for plaktonic and sessile bacteria population and change of hydrogen sulphide concentrations are shown in Fig. 2(a) and 2(b). The cell concentrations of planktonic and sessile bacteria increased to a maximum 7.7×10^9 cells ml^{-1} and 1.94×10^8 cells cm^{-2} after 48 hours incubation respectively and began to decrease slowly thereafter. Fig. 2(a) shows four distinct phases of bacterial growth. The very short 'log phase' during initial adaptation phase followed by a long exponential phase where logarithm of bacterial count is proportional to incubation time. This is then followed by a steady phase where growth rate is equal to the death rate or decline rate. The deaths occur due to depletion of an essential nutrient of medium by wastes or inhibitory product such as organic acid. The last phase is the death phase. Here the time frame of different phases in the growth curves of SRB in planktonic and sessile condition are almost similar except death phase where number counts of SRB, falls more rapidly for planktonic condition than that in sessile condition. This is possibly due to the inhibitory products which were continuously increased in planktonic state but owing to the lack of much sticky power and large solubility in water, these products did not stick to the surface and came out through the porous surface. Fig. 2(b) shows the gradual increase of H_2S concentration with incubation time with sharp increase after 48 hours. The time period of such change occurs in the stationary and death phase of the bacterium. This indicates the enzymes [30, 31] that help in the process of H_2S production remains active at least for sometimes, even after the death of bacteria.

Applied load, and concomitant crack length are related graphically with the time of testing as shown in Fig. 3 and 4 respectively. Fig. 3 indicates changes in load with testing time as gradual crack extension had occurred in above three environments. Fig. 4 represents changes in crack

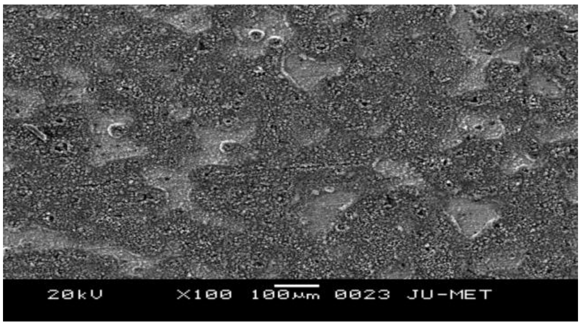

Fig. 1(a) Surface of notched tensile specimen after 48 hrs immersion in SRB culture

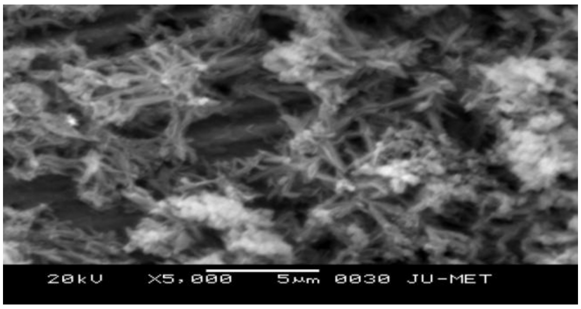

Fig. 1(b) Colony of SRB on notched tensile specimen after 72 h immersion in SRB culture

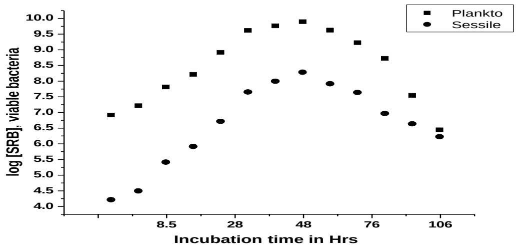

Fig. 2(a) Growth curves of planktonic and sessile desulfovibrio desulfuricans bacteria.

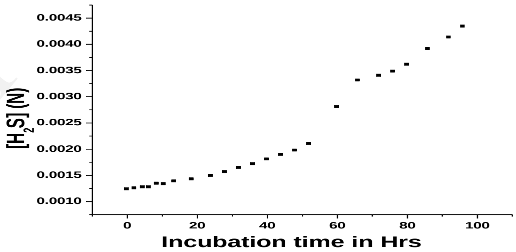

Fig. 2(b) Change of hydrogen sulphide with growth of SRB bacteria in Postgate's medium containing desulfovibrio desulfuricans bacteria

length occurring as test progressed in same environment. These two figures are comparable to the well documented classical theories for SCC in general [26, 32]. Distinct differences are apparent when increased load and increasing crack length
with testing time are compared in the tests carried out in air and biotic medium. For the tests in air and abiotic media load increased monotonously with concomitant crack extension in test specimen with time till the failure of the specimen had occurred. Effect of SRB culture media, on the other hand, both on advancing crack length and increasing load with time indicates crack progression till the failure had occurred. Duplicate tests were performed under the same experimental conditions and nearly same SRB concentration to test reproducibility of the results.

During slow stain rate tests the crack tip experiences stress which increase continuously with increasing load. Failure through cracking in air and Postgate's environment initiate and progresses, in general, by microvoid coalescence (MVC, Fig. 5 and 6) mechanism. Therefore dimples on the fracture surface represent ductile mode of failure as revealed extensively in the case of failure in air on the ductile iron. Fine bands of MVC arranged in a semicircular pattern were observed (Fig. 5 and 6) on the fracture surface as a discontinuous and irregular event in the direction of fracture progression. This type of semicircular pattern is more intensively found in air indicates more ductility. During SSRT in air it is inferred that as the internal stresses were generated on tensile loading, activated Frank Reed sources [33] generating dislocations moving towards the point of greater stress intensity at the crack tip. The repetition of this process under stress results in the cubic lattice deformation. This deformation is the transition from an un-slipped state to slipped state.

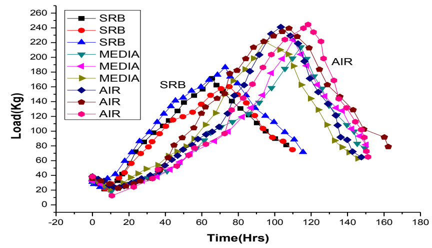

Fig. 3 Plot of load against time curves produced by SSRT tests (C.H.S=10^{-5} mm/sec) of ductile iron in air, Postgate's medium and SRB culture. D = discontinued.

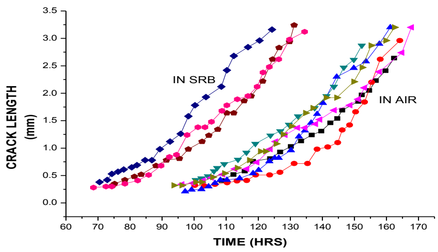

Fig. 4 Crack lengths against time curves produced by SSRT tests of ductile iron in air, Postgate's medium and SRB culture.

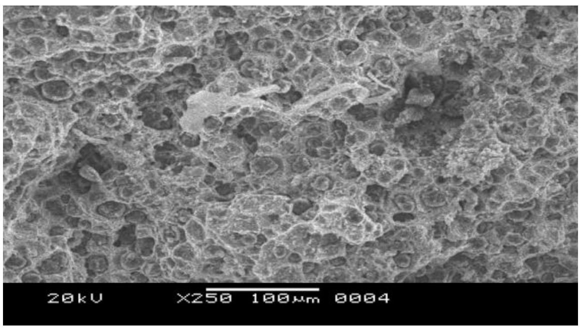

Fig. 5 Ductile fracture for ductile iron sample tested by slow strain rate test in air as reference test. Crack propagating from left (L) to right (R).

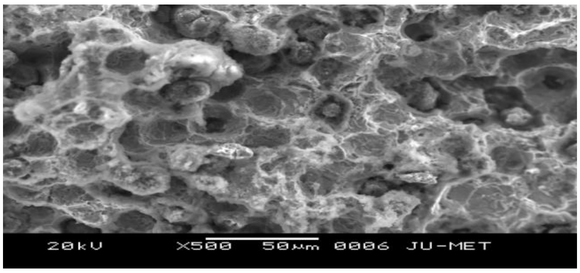

Fig. 6 Fracture surface tested (C.H. 1.0×10^{-5} mm s^{-1}) in Postgate's medium, Crack propagating from L to R.

With increasing deformation (in air), the number of lattice defects increases. The dislocations mutually interfere with each other and their movement is hindered by e.g. precipitates, grain boundary, and crack slip. The possibilities for slip are therefore continuously reduced and finally exhausted, so that no further deformation can occur. Further application of stress brings about only ductile overload failure.

In culture medium, SRB reduces sulphate to sulphide [27, 28] combined with hydrogen to form H_2S. The blackening of the medium and odour of rotten egg is presumably an indication of iron sulphide as corrosion product. Discontinuous bio-film and corrosion product are formed on the surface of the sample exposed to pure SRB during SSRT tests. Such discontinuities could be the locations of pitting. Failed sections of the specimens tested in both the abiotic and SRB containing biotic environments suggested pit formation at the edges. A comparison of the two cross-sections also suggests that biotic samples have suffered more pitting than abiotic ones.

As this experiment has been carried out in SRB containing aqueous environment electrochemical reactions also occurred with SCC. The process of corrosion of ferrous metal due to SRB by the cathodic depolarization theory [34, 35] is

Oxidation/Anodic/metal dissolution reaction:

$$4Fe \longrightarrow 4Fe^{2+} + 8e \quad (1)$$

Electrolytic dissociation of water:

$$8H_2O \longrightarrow 8H^+ + 8OH^- \quad (2)$$

Cathodic reaction: $8H^+ + 8e = 8H_{ad}$ (3)
Mono atomic hydrogen adsorption

Cathodic depolarization by SRB:
(Enzyme hydrogenase)

$$SO_4^{2-} + 8H_{ads} = S^{2-} + 4H_2O \quad (4)$$

Overall reaction
$$4Fe + SO_4^{2-} + 4H_2O = 3Fe(OH)_2 + FeS + 2OH^- \quad (5)$$

Corrosion product:
$$Fe^{2+} + S^{2-} = FeS \quad (6)$$

Another corrosion products:

$3Fe^{2+} + 6OH^{-} = 3Fe(OH)_2$ (7)

$FeS + 2H_2O = Fe(OH)_2 + H_2S\uparrow$ (8)

$H_2S + Fe = FeS + [2H]$ (9)

The above reactions justify the corrosive attack by SRB on ferrous metal. The produced sulphide would react with available proton to form hydrogen sulphide, H_2S, which is known as a very corrosive species related to the corrosion of ferrous materials [16]. In aqueous solution of SRB the nature of corrosive environment is drastically changed in the same service condition of Postgate's media, temperature and loads.

Therefore in air and Postgate's solution of SRB, anodic reaction would be the formation iron oxide/hydroxide and cathodic reaction would be the reduction of H_2S and hydrogen evolution reaction since H_3O^+ generates at the crack tip region.

$H_2S + e = HS^- + ½H_2$ (H_2 generation)

$Fe + HS^- = FeS + H^+$ (acid generation)

$H^+ + e = ½ H_2$

Therefore the probable reaction at the crack tip would be

$Fe \longrightarrow Fe^{2+} + 2e$ (anodic)

$H^+ + e \longrightarrow ½ H_2$ (cathodic)

The conduction of ion at removal of corrosion products from surface being easier in aqueous solution than moist air so crack velocity is greater and ESF is earlier for SRB containing Postgate's medium compared to air

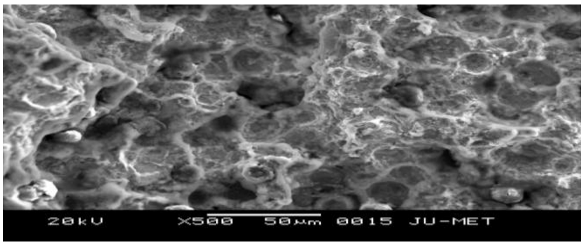

Fig. 7 Fracture surface tested in SRB culture, crack front part, small cracks observed, mixed area ductile quiser brittle in stage I.

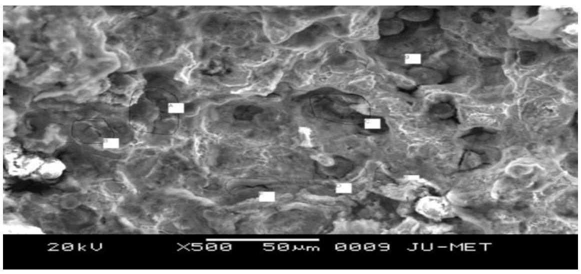

Fig. 8 In SRB culture, crack propagating from L to R, IG mode of cracking, flat land indicates more brittleness. A, B, C, D, E, F and G are small cracks on the fracture surface. Ductile and brittle areas are present.

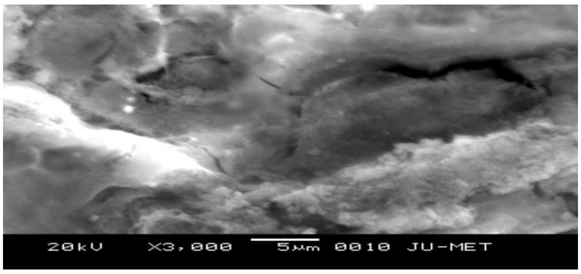

Fig. 9 In SRB culture, crack front part, crack propagating from left to right with anodic dissolution.

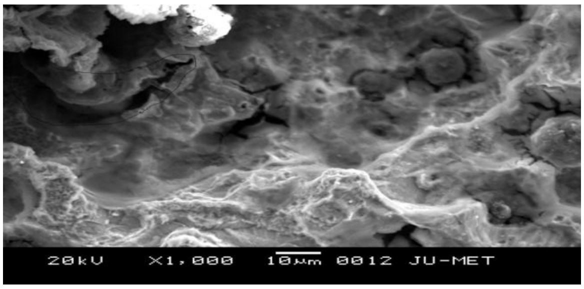

Fig. 10 In SRB culture, front part within 1.5mm crack length, quiser brittle area, crack progresses from left to right and changing the plane (marked).

During SSRT, SRB containing Postgate's medium, continuous increasing stress would initiate mechanical breakdown of surface film and when

the ratio of the concentration of the aggressive to passivating species become greater than unity , enhanced anodic dissolution occurring selectively at the ductile iron crack tip. The crack tip region would also experience stress deferential across the crack tip radius. Consequently , crack tip would become relatively more active with respect to crack sides as the crack sides do not experience similar stress assisted electrochemical activities to the extent crack tip does.

During testing in the SSRT mode, load increases with time. The deformation process would maintain the crack tip in a condition that may only be intermittently film-free on a local scale corresponding to discontinuous surface yielding. The steps formed by individual grains protruding may form the necessary discontinuity if the grain boundaries have been weakened due to precipitations. For a critical rate of deformation, dissolution interactively promotes stress corrosion cracking.

Therefore the conditions for stress corrosion cracking were met only when deformation processes at the crack tip region under continuously increasing load cracks the passive film and enhance anodic activity and prevent repassivation process at the crack tip region. At the commencement of cracking of the specimen, above the threshold load had evidently marked the transition from no cracking situation to commencement of crack progression stage. For tests carried out in a SRB containing corrosive environment this transition indicates commencement of SCC by the effect of SRB.

After the nucleation in the early stage of crack propagation there would be the probability of ingression of hydrogen produced at cathodic sites as a result of anodic dissolution at crack tip into the metal lattice. Therefore in stage I, a situation would emerge when film rupture and repassivation kinetics are in a state of balance to sustain stress corrosion cracking. The crack tip region experiences the critical stress level and the critical anodic reaction rate for stress corrosion crack to progress [29, 32]. Preferential dissolution at the crack tip occurs presumably due to pre existing galvanic potential difference between the grain boundaries and lattice vacancies at the grain boundaries. The former being anodic to grains resulting inter-granular (IG) mode of failure.

Initially the crack growth rate was relatively slower than in the later stage of crack propagation. Initial crack is slower due to low concentration of H_{ads} with the formation of molecular H_2. Subsequently it is fast due to generation of H^+, HS^- , H_2S in the local holes and generation of H_2 which brittles the surface. After a critical time sufficient number of H atoms of $M_{H\ abs}$ make a permanent defect of the metal lattice and is included in the bulk metal and brittles internal metal structure thus crack velocity increases after the critical point.

Scanning electron microscopic fractography obtained from this region revealed discrete IG (intergranular) mode of failure together with general ductile cusps interspersed with crack progression marking resembles hydrogen inspired failure Fig. 7. Flat brittle facets and crack arrest markings [Fig. 8] indicate discontinuous nature of crack progression under conjoint action of stresses and anodic activities at the crack tip in presence of SRB culture. In Fig. 9 corrosion products on crack morphology at the edge of the specimen depict dissolution characteristics on fracture facets. Small cracks at the crack front [Fig. 10] also highlight trans-granular (TG) crack progression. These fracture morphological details are better revealed in Fig. 8.

Critical role of absorbed hydrogen could be seen in the Fig.10 causing discontinuous quasi cleavage type failure in the ductile iron material. The existence of hydrogen induced cracking (HIC) can affect the material’s cracking susceptibility in culture media. Residual or applied stresses can increase the stress field surrounding the cracks. This generates localized yielding which can produce a small crack perpendicular to the tensile stress direction. This mechanism suggests that hydrogen weakens inter atomic bonds but the crack development occurring by localized slip. This mechanism proposes that hydrogen on the surface and among the first few atomic layers of atoms weakens inter atomic bonds, facilitating the emission of dislocation from the crack tip. Crack growth occurs primarily by alternate slip from the crack tip with contributions from plasticity and void formation ahead of the crack.

The favourable postulation of HIC in SCC envisages ingress of hydrogen atoms at the crack tip by adsorption on the stretch zone formation markings. Crack advancement ensues to a finite distance at critical concentration of atomic hydrogen at the triaxial stress concentration sites ahead of the crack tip. The atomic hydrogen produced by eqn. 3 may also enter the crack tip region by normal lattice diffusion or by dislocation transport. Dislocation transport may occur with the hydrogen either located in Cottrell atmospheres around the dislocations or in the dislocation core. This hydrogen transported to recombine at suitable nucleation sites and create atmosphere of high pressure hydrogen gas which provides additional crack tip stress sustaining discontinuous nature of brittle failure modes. It follows that the extent of embrittled material that the growing stress corrosion crack can pass through is limited by the diffusion distance of hydrogen atoms. This diffusion distance will be dependent upon the hydrogen concentration at the crack tip surface and the form of the concentration profile and hence will vary the degree of environmental aggression. While microspores on the tear rides, and TG cleavage facets mark hydrogen entrapments, crack arrest markings indicated hydrogen concentration gradient related discontinuous crack propagation.

4. CONCLUSIONS

In the study the results obtained from SCC in slow rate testing of ductile iron in air and aqueous medium in presence and absence of sulphate reducing bacteria, desulfovibrio desulfuricans (SRB) are highlighted and probable influence of SRB in the mechanisms for stress corrosion effects are postulated.

1. The experiment reveals that the initiation of corrosion and subcritical crack growth fastened in the order of medium: air < Postgate’s medium < Postgate’s medium +SRB, indicating strong influence of SRB to fasten SCC.
2. Assessed average crack velocity (CV) of ductile iron was higher in SRB containing medium than in air and follows the above mentioned order
3. Variation in average CV outlining different stages of indication the effect of conjoint action of applied tensile load and environmental effects.
4. SSRT modes of testing in air had revealed MVC mode of failure in the materials tested whereas more flattened fracture surface is observed for Postgate’s medium in presence and absence of SRB culture.
5. Stress corrosion cracking (SCC) is postulated to have occurred by intergranular cracking (IG) and transgranular cracking (TG). Increasing susceptibility to SCC dominated failure was indicated increasing brittle fracture mode (IG+ TG).

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TABLE I. Chemical composition of ductile iron pipe material in mass /mass percentage (%) of constituting elements

C	**Si**	**Mn**	**S**	**P**	**Mg**	**Ti**	**Cr**	**Ni**	**Mo**
3.66	2.23	0.374	0.0085	0.0664	0.0602	0.0415	0.0090	0.0353	0.0025
Al	**Cu**	**V**	**Pb**	**Sb**	**Sn**	**As**	**Ce**	**Fe**	
<0.0003	0.0102	0.0043	0.0017	0.0010	0.0033	0.0041	0.0007	Balance	

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