CHAPTER-II

PREAMBLE OF STUDIES ON CORROSION OF CARBON STEEL AND ITS CONTROL IN CARBON DIOXIDE REMOVAL SYSTEMS

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PREAMBLE OF STUDIES ON CORROSION OF CARBON STEEL AND ITS CONTROL IN CARBON DIOXIDE REMOVAL SYSTEMS

SECTION - I : PROBLEMS IN CARBON DIOXIDE REMOVAL SYSTEMS USING HOT POTASH/ GV SOLUTIONS

A survey of corrosion problems faced by the absorbers, regenerators and other equipment connected to the carbon dioxide removal system is carried out. The section is divided into two parts, viz :

a) Corrosion problems in hot potash/ GV systems and
b) Corrosion problems in MEA/ DEA systems.

a) Corrosion Problems in hot potash/ GV System :

The most serious operating problem, encountered with carbon dioxide removal systems, using potassium carbonate either alone or with arsenic trioxide, glycine, MEA or DEA is general thinning. Various theories have been presented through patents and technical articles to explain the mechanism of this type of corrosion (1-6).

In short, the acid gases themselves are main culprit in such phenomena. It is observed by many workers (7-9) that the increase in corrosion is directly propotional to increased acid-gas concentration. It was further noticed that free carbon dioxide causes severe corrosion, particularly at elevated temperatures and in the presence of water.

As explained by Evans (10), the metallic iron from the carbon steel material reacts with carbonic acid to form iron bicarbonate, but rise in temperature causes the carbon dioxide to be released and precipitates the iron as iron carbonate. The iron may also be removed from solution by hydrolysis to basic carbonates or hydrated oxides or further oxidation to the less soluble ferric compounds. The saturated carbon dioxide solution and subsequent heating, dissolve more iron which aggravate the corrosion rate.

Potash solutions, thus has a high corrosive property and, if, interlocked stresses are present in the fabricated vessel, stress corrosion cracking also may occur (11-16).

SCC as defined by Scully (17) and others (18) is a non ductile fracture, resulting from the simultaneous application of a tensile stress and a specific corrodent.

According to Mervyn Turner (19), SCC failure, especially in Gv solutions used for carbon dioxide removal systems, the following points are of paramount importance.

1. The corrodent is specific to individual metals and alloys, e.g. hydroxyl ions, nitrate ions and carbonate-bicarbonate ions are specific to carbon steel material.

2. Presence of stress and specific corrodent is necessary.

3. The stress does not have to be the result of an applied load. Residual stresses in the material can cause SCC failure.

4. Concentration of a specific ion in the layer of liquid in contact with the metal commonly known as 'Helmhotz layer'. (A typical example is the 'splash zone' in a vessel).

5. Increase in temperature promotes SCC.

6. SCC occurs in a series of steps rather than continuous and the time to failure may be prolonged up to years.

7. SCC is frequently characterised by multiple cracking due to microstresses imposed by inhomogenities in the metal.

Froning and Jones (7) showed that corrosion by carbon dioxide is most severe at concentrations of carbon dioxide (in the synthesis gas) of 20 to 30%. Corrosion rates upto 0.03 inches /year were measured in these conditions.

R. Erskine (20) in his paper on vessel inspection procedure pertaining to pressurized equipment of ammonia plants had found out that considerable corrosion was taking place in carbon dioxide absorbers using GV solutions. Most of these defects were found to be below the semi lean level and consisted of fine, shallow cracking in the welds, 'tram line' edges and general corrosion up to 5 mm deep in the 37 mm wall thickness of the shell. Cracking, he observed, was most concentrated at regions of high stress like nozzles, manholes and longituidinal welds. In the same way, carbon dioxide regenerators, after many years of service was found to be prone to cracking, especially near the welds between the rich solution intake and the semi lean take off. Corrosion and cracking was so concentrated in some heat affected zone (HAZ) of welds that external strapping of the longituidinal welds was carried out. General corrosion of regenerator plate was also observed.

According to Ing V. Alemanno (21) of Amic Agricultura spa, Italy, the carbon dioxide absorber faced a very serious problem due to an explosion in 1976, when the 4.5 meters long upper section of the absorption column splitted open and thrown to a distance more than hundred meters. The absorption column was using GV-arsenic trioxide system for carbon dioxide removal from synthesis gas. The cause of the failure, culminating into a disaster was a large crack (due to SCC) on the weld connecting the upper truncated cone to the lower column.

Padh et al (22) has given an excellent review of corrosion problems faced by carbon dioxide removal systems pertaining to ammonia plants in India. Fertilizer corporation of India Ltd. Gorakpur uses two stream carbon dioxide removal system, hot potassium carbonate followed by MEA wash. In the carbon dioxide regenerator, leakages were observed from the welding seam. Transverse cracks were observed on the material and the areas attacked were in the form of circular grooves. In the absorber and regenerator the inside wall revealed deep cuts and grooves all around the periphery. Corrosion was most severe in the bottom beds progressively decreasing towards the top most bed.

High local concentration of iron in the form of ferrous ions at the contact surface of the potash solution and the carbon steel material was one of the possibilities envisaged for this causing localised attack.

Rashtriya Chemical and Fertilizers Ltd. Trombay, uses GV process followed by MEA and caustic wash. In the absorber pits of 0.25 to 0.4" deep in the shell above packing space on each bed was observed. AISI 304 SS lining of the entire shell was carried out to prevent this problem. Same type of pits were observed in the regenerator.

Fertilizers and Chemicals Travancore Ltd. Cochin using GV system for carbon dioxide removal, faced problems of sludge formation in the absorber column. General corrosion may be one of the factor contributing to this type of problem.

In Gujarat State Fertilizers Co. Ltd., Baroda, one of the three ammonia plants which uses two stream method of carbon dioxide removal (GV followed by MEA wash) had faced serious corrosion problems. Fouling of heat exchangers/ coolers and corrosion of the regenerator top bed portion had encountered serious problem. Cracks in the regenerator on the circumference at two different levels in top bed was found. Preferential dissolution was observed. Probable reason was stress corrosion cracking.

b) Corrosion Problems in MEA/ DEA Systems :

Corrosion is also of concern in plants having carbon dioxide systems of MEA/ DEA solutions as reported by Polderman et al (23), Moore (24), Fitzerald et al (25), Smith et al (26) and Hall et al (27).

Another factor contributing to corrosion in MEA/ DEA systems is the formation of ethanol amine-iron compounds as identified by Dixon and Williams (28). The decomposition and degradation products of these solvents is a matter of considerable controversy but of practical importance (1, 2, 29).

Moore (24) established that corrosion rate increases with increasing concentration of degradation products. 3-(Hydroxyethyl)-2 Oxazolidone, was found to be a major degradation product of DEA by Chakma et al (30).

Intergranular stress corrosion cracking (IGSCC), in the vicinity of non stress releived welds in carbon steel equipments in MEA solution was studies by D G Hughes (31) and Gutzeit et al (32).

Verduijn (33) had shown that corrosion is bigger problem in carbon dioxide removal units using potassium carbonate - DEA systems even with inhibitors like vanadium. A comprehensive account is available along with remedial measures from Verduijn. Kolff and Kemira (34) in ontinuation of the study by Verduijn had observed extensive cracking in the repair welds in the same absorber tower after only 3 years of repairing.

Stress Corrosion Cracking of mild steel in MEA solutions is extensively studied by Parkins et al (35) and Richert et al (36).

Padh (22) in his studies had pointed out various corrosion problems faced by carbon dioxide removal systems using MEA/ DEA systems in Indian environments. Corrosion of pumps and impellers and casing in lean/ rich MEA exchanger at Coromandel Fertilizers Ltd., Visakhapatnam; Fertilizers and Chemicals Tranvancore Ltd., Udyoga Mandal which uses 15% MEA for carbon dioxide removal had severe corrosion, erosion of let down valves with carbon steel internals ; Modified Benfield process at Shriram Chemicals industries, Kota also was facing corrosion problems at the top bed of absorber due to deposits, are some of the problems studied by him.

In Gujarat State Fertilizers Co. Ltd., Baroda, the GV system where subsequently GV-glycine was introduced, within four months of which serious cracking and corrosion problems were noticed in places where stress concentration was envisaged. The problem was extensively studied by Thanki et *al*(37) and found out that where non-stress relieved welds in the absorber column, regenerator column and the branch connections of the GV transfer line were the sensitive areas of localised attack. Moreover, it was observed by them that vanadium pentoxide do not give adherant film on the carbon steel surface and hence high solution velocity area suffered serious attack due to removal of vanadium pentoxide bound film.

SECTION - II : CORROSION CONTROL IN CARBON DIOXIDE REMOVAL SYSTEMS :

This Section is divided into three sub-sections viz :

a) General methods for corrosion control
b) Corrosion control in hot potash/ GV systems by inhibitors
c) Corrosion control in MEA/ DEA systems by inhibitors

a) General methods for corrosion control :

Corrosion in the carbon dioxide removal systems can be reduced by various means, like certain operational practices and design features as recommended by Kohl et al (38).

a.1) It is observed that corrosion is most prevalent in places where the highest concentration of acid gases is accompanied by highest temperature. Therefore, the temperature of the solution should be kept as low as possible.

a.2) Pump glands are the source of oil contamination in the circulating fluid and thereby decrease the heat transfer capacity of the system which in turn, aggravates the corrosion phenomena. Good maintenance of pump may help us to control this effect.

a.3) Pressure regeneration with accompanying high temperatures causes severe corrosion, hence lowest possible pressure must be maintained.

a.4) To prevent any unwanted air, positive pressure should be maintained on the suction side of the pumps.

a.5) Continuous removal of suspended particles by filtration and decomposition products by distillation also helps in reducing corrosion.

a.6) Proper practices in cleaning of plant equipment is also critical.

a.7) It is desirable to circulate a considerable excess of solution to maintain low acid-gas concentration in the rich solution.

a.8) Maintaining of low velocities in the heat exchangers is desirable.

a.9) The rich solution should be circulated through heat exchanger tubes, rather than in the shell.

a.10) In order to reduce corrosive attack by acidic gas flashes heat exchangers should be at positive pressure.

a.11) Use of more expensive corrosion resistant alloys in place of carbon steel, wherever possible.

The concentration of the scrubbing solution is also an important factor which aggravates/ reduces corrosive effects (39).

Many case studies and laboratory tests are reported, which indicate disintegration of porcelain packing rings, used in the absorber and regenerator, which adds to the sludge formation and consequently erosive/ corrosive effects take place (37, 40).

For limiting SCC type of phenomena, lining of the problematic section with stainless steel material and/ or synthetic resins is recommended (33).

Removing the residual stresses by shot peening or other stress releiving methods is also recommended (41).

The use of inhibitors/ passivators in the solution for reducing the effect of corrosion is also pointed out and is the most widely used method.

b) Corrosion control in hot potash/ GV systems by inhibitors :

U.S. Bureau of Mines (38), in the tests carried out, had revealed that severe corrosion of carbon steel encountered, especially where the conversion of bicarbonate was high, can be effectively controlled by the addition of 0.2 percent potassium dichromate. It is recommended only for plants handling sulfur free gas, since the presence of appreciable quantities of sulfur compounds reduces chromate ion resulting in the formation of insoluble precipitates leading to operational difficulties and erosion of equipment.

1.5 to 5% sodium nitrite was tried as a corrosion inhibitor in 40% potassium carbonate solution by Forolius et al (42) at 99 Deg. C. An optional additional injection of air was also suggested by them. After carbon steel strips exposed for six days in the solution, the inhibitor effeciency was found to be highest at 5% sodium nitrite.

Eickmeyer (43) had studied the effect of continuous or intermittent introduction of air into the system (22% potassium carbonate and 0.2 - 0.3% sodium or potassium metavanadate) for corrosion prevention. In place of air, the study was also conducted with oxygen, hydrogen peroxide and potassium dichromate. These oxidizing agents prevents reduction of meta or peroxyvanadate, thus, indirectly helping in the prevention of corrosion. Blake (44) had used a combination of vanadium compound and an antimony compound for inhibition of corrosion of carbon steel in 20 - 40 weight % potassium carbonate. 1 - 9 parts by weight of a pentavalent vanadium compound and a 1 - 9 parts by weight of trivalent antimony compound was used. 0.2% sodium metavanadate, 0.33% vanadyl sulfate dihydrate, 0.2% potassium antimonyl tartrate with 0.02 weight % tartaric acid and a mixture of sodium metavanadate and potassium antimonyl tartrate with 0.01% tartaric acid was tried. In the last combination mentioned, the corrosion rate was found to be reduced to 0.012 mm/ yr from 8.46 mm/yr and had the highest inhibitor effeciency of all inhibitor combination tried.

Turner Edward (45), in his study of inhibitor in GV-arsenic oxide systems, had used trivalent antimony ions and ferric ions, to increase the redox potential so that it is far away from the redox potential at which the carbon steel surface is susceptible to intergranular corrosion. A 30% w/w solution of potassium carbonate and 10% w/w arsenic oxide solution was used as stock solution. Antimony ions

(0.05 - 0.20%) and ferric ions (100 - 300 ppm) which was produced by bubbling of O_2 containing gas, was added in the stock solution and metal samples were exposed to this solution. Electrode potential measurements and microscopic investigations were carried out.

Stress Corrosion Cracking phenomena and its prevention was also studied by Fyfe (46) in potassium carbonate - arsenic oxide systems. He used ferric ions, antimony and/ or vanadium ions. Electrochemical potentials were measured. With an inhibitor concentration of ferric ions 100 ppm and antimony trioxide 0.10% the potential was found to have increased to -590 mV (vs SCE) from - 790 mV in 30% potassium carbonate - 10% arsenic oxide solution.

Potassium nitrite (0.01 to 0.5% w/w) alone and its combination with 0.01 to 2.0% w/w of various vanadates like sodium vanadate, potassium vanadate, sodium metavanadate and potassium metavanadate was studied as inhibitors in potassium carbonate solutions by Field et al (47). The concentration of the nitrite in the solution should be 0.1 - 0.5 times of the vanadate solution, in which case maximum effeciency was observed.

Forolius (48) found out that corrosion of metals in alkali metal salts (or amine compounds) could be inhibited by addition of an ammonium or alkali metal sulfide or polysulfide and elemental sulfur. Thus a solution containing potassium carbonate 28%, N-cyclohexyl - 1,3 - propanediamine 7%, pipecolonic acid 7%, sodium sulfide 7.4%, sulfur 0.5%, water 50.1% w/w, carbon steel material had no corrosion, compared to 18.8-19.8 mm/ yr in a solution with no sodium sulfide and sulfur.

Inhibitors like sodium perborate ($Na_2B_4O_7$), potassium dichromate ($K_2Cr_2O_7$), vanadium pentoxide (V_2O_5), bismuth oxide (Bi_2O_3) and sodium silicate (Na_2SiO_3) was tested with various grades of carbon steel material (St 3SP, St 20, 16 GS) in 23% potassium carbonate solution at 60 Deg. C. by Nikitina et al (49). The most effective protection was obtained with addition of 0.1% V_2O_5 and 0.7% $K_2Cr_2O_7$, when the steel surface was transformed into the stable passive state.

JGC Corp. Japan (50), suggests for protection of the strippers used in regeneration of potassium carbonate solution, addition of cerium nitrate ($CeNO_3.6H_2O$) at a concentration of 0.5% (0.16% cerium). According to them, this inhibitor can also be used in alkanolamine solutions or a mixture of both at 100 Deg. C. Loss in weight after 300 hours of exposure in 25% aqueous potassium carbonate solution with inhibitor was < 0.001 grams versus 0.8 - 1.1 grams without inhibitor.

Vanadates used in potassium carbonate solutions as inhibitors have been studied extensively by Bensheng (51).

c) Corrosion Control in MEA/ DEA Systems by inhibitors :

Blanc et al (52) had given the use of degradation products of DEA/ MDEA as corrosion inhibitors for carbon steel material in carbon dioxide removal systems.

Inhibition of corrosion of steel in alkanolamine solutions was achieved by Edward (53), by addition of soluble salts of vanadium (tetra and penta valent) and cobalt (divalent). In a 50% MEA solution, corrosion rate was found to be < 1 mils per year with 300 ppm cobalt and 200 - 400 ppm vanadium.

Kosseim et al (54) had developed a corrosion inhibiting package, amine guard ST, which is a mixture of two corrosion inhibitors and is used in ethanolamine systems and the inhibitors are not disclosed for proprietory reasons.

Pearce (55) cited the use of divalent copper (50 ppm). nickel and/ or bismuth oxide, dihydroxy ethyl glycine (50 ppm) and ammonium thiocyanate in ethanolamine solutions for corrosion protection.

Henson et al (56) and others (57) conducted detailed studies on the use of $(C_2H_4OH)_2NCH_2COOH$ in combination with cuprous ions, permanganate ions and thiocyanates as inhibitors in alkanolamine systems. Thiourea - amine - formaldehyde copolymers were also studied in combination with nickel compounds.

Mitina (58) reveals the use of tertiary amino nitrile (0.003 -0.09% w/w) as corrosion inhibitor in 5.0 - 35% w/w alkanolamine systems.

SECTION-III - CORROSION PROBLEMS IN GV (POTASSIUM CARBONATE-ARSENIC TRIOXIDE) SYSTEM OF AMMONIA PLANT AT GSFC LTD.

At Gujarat State Fertilizers Co. Ltd., the ammonia plant with a capacity of 500 MT/ D using GV split stream method for carbon dioxide removal from synthesis gas containing > 20% carbon dioxide, faced serious corrosion problems after about 15 years of service.

A thorough inspection of the absorber was taken up in view of the melting of polypropelene packing rings in the 3rd and 4th bed. It is to be noted here, that at the 3rd bed of the column is the splash zone where semi lean solution from regenerator enters into the absorber.

Some salient features of the inspection of the absorber is given, which is divided into three parts, namely :

a) Visual

b) Fluorescent Magnetic Particle Inspection (FMPI)

c) Thickness measurements.

a) Visual Inspection :

At the 3rd and 4th bed vessel wall, extensive erosion/ corrosion marks were revealed. Grooving and channeling effects were visible on internal surface of shell wall. The circumferential welds and fillet welds of support ring brackets had erosion/ corrosion marks (plates 1 to 5).

b) FMPI :

This was carried out on all weld joints of 3rd and 4th bed. In the 3rd bed 3 numbers of fine cracks, ranging from 55 mm to 175 mm in length were observed. 4 numbers of cracks ranging from 40 mm to 90 mm length were observed on the lower fillet bed of 2nd bed support.

In the 4th bed, 9 numbers of crack marks were seen on the toe of the weld joints of cleats supporting the 3rd bed ring. The cracks had initially appeared shallow, so two of the cracks were ground up to 1 mm for elimination purpose and remaining cracks were not ground.

One crack on the fillet weld on the beam support appeared to be deep and propagating inside. The crack was ground up to a depth of about 4 mm in the welding, however, the crack profile indicated to be going towards beam support and not towards shell wall. The dimensions of the crack after grinding is shown in (Figure-1).

Circumferential crack marks were observed on the edges of weld seams. These were shallow in nature and easily removable by grinding.

c) Thickness measurements :

Thickness was carried out in 3rd and 4th bed. A general thickness reduction of 5 to 6 mm from the original 40 mm was observed. In the last 8 months the thickness reduction was up to 0.2 to 0.3 mm.

Considerable reduction in thickness in 3rd and 4th bed shell wall, deep grooving and channeling of irregular pattern scattered around 70% of the area was observed. In most of the areas corrosion allowance (3 mm) had been consumed and more and more cracks of varying depth and morphology were observed as was the case 8 months before.

SECTION - IV : BASIS OF PRESENT WORK

a) Outlines of the studies on corrosion problems and its control in CO_2 removal systems.

It is observed from the above survey that carbon steel material used in the absorbers regenerators and other equipment in carbon dioxide removal system is prone to general thinning and Stress Corrosion Cracking.

MEA/DEA units solutions, when used with controlled lower percentage can minimize corrosion problems to some extent, but hot potash and GV systems are prone to such type of problems. It is also seen that the carbonate solutions, upon saturation with more carbon dioxide, is capable of dissolving more iron and increasing the corrosion rate. Carbon steel material is sensitive to Stress Corrosion Cracking (SCC) in potash solution and for this material, the specificity of the corrodent like hydroxyl ions, carbonate/ bicarbonate ions is well known.

Especially with GV split stream method, the concentration of these SCC causing ions are more at the splash zone of the absorber, where semi lean solution from the regenerator half way is taken out and splashed at the middle portion of the absorber, which results in fine, shallow cracks and general thinning. The cracking in high pressure GV systems had lead to explosions, culminating into major industrial disasters. The welding seams and the heat affected zones are particularly prone to SCC due to interlocked stresses. Transverse cracks, leading to circular grooves are formed near attacked areas.

The packing material, using Intallox saddles made of stainless steel or porcelain material causes pitting along the contour of saddles.

General corrosion also leads to excessive sludge formation leading to concentration pockets.

In MEA/ DEA systems, the fact that degradation products cause more corrosion problems and intergranular SCC, especially in MEA units, is proved beyond doubt.

DEA units, in which corrosion problems are encountered at lesser intervals up to now, are also facing corrosion problems with extensive cracking at the repair welds.

General methods for corrosion control, by certain operational practices and design features are always followed world wide, does not eliminate the problem altogether, but certainly reduces the chances and frequency of problem considerably.

Removing the residual stresses, in the carbon steel material by stress relieving methods is very costly and cumbersome. To spot the personnel for carrying out such techniques is also very difficult and it is also extremely difficult to get 100% reproducible result in towers of the height of more than 30 meters.

The use of inhibitors to passivate the carbon steel material is the most widely used corrosion prevention method in these systems.

The inhibitor used, has to act as a passivator, by reducing the corrosion current and also shifting the potential of the carbon steel material to the passive state far away from the potential range at which SCC occurs, thereby preventing both general corrosion and SCC.

Many inhibitors and its combinations are studied and recommended for such use in hot potash and GV system. Potassium dichromate, vanadium pentoxide and sodium nitrite are some example of single inhibitor system.

Many combinations are also extensively studied. Compounds of vanadium and antimony, antimony and iron, vanadium and iron, potassium nitrite with various combinations of vanadates, alkali metal sulfide and sulfur, bismuth oxide and sodium silicate are some examples of this combinations.

For MEA/ DEA units, inhibition is achieved by addition of salts of vanadium and cobalt. Amine guard ST also works as a inhibitor package. Nowadays copolymers are studied in combination with nickel compounds in these systems.

With special reference to Gujarat State Fertilizers Co. Ltd., it is experienced that after 15 years of service period CO_2 absorber walls had been affected by severe corrosion leading to cracks initiation.

B) SCOPE OF WORK

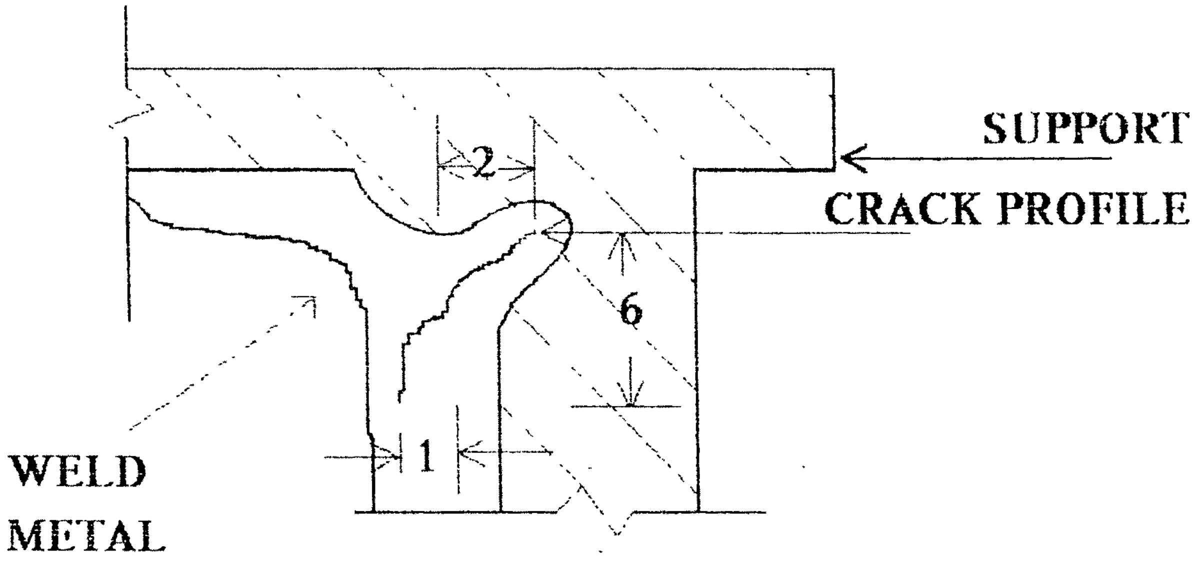
1. A Search of an inhibitor or a combination of inhibitors which when added to the GV solution in small concentration will effectively control the corrosion problems.

2. Apply electrochemical evaluation technique to understand the mechanistic aspects of corrosion phenomena and its inhibition.

3. Detailed studies of selected inhibitor for its compatibility and effeciency at the lowest possible concentration to economize the large volume handling system.

4. Narrow down the compatibility and effeciency contributing factors by diversified views like impedance, pearlite leaching for surface characterization.

5. To substantiate the performance of selected inhibitor on its availability, handling, safety, economy and operational viability.



All dimensions are in mm

Figure 1: Crack profile depicting the dimensions of the crack after grinding

1

2

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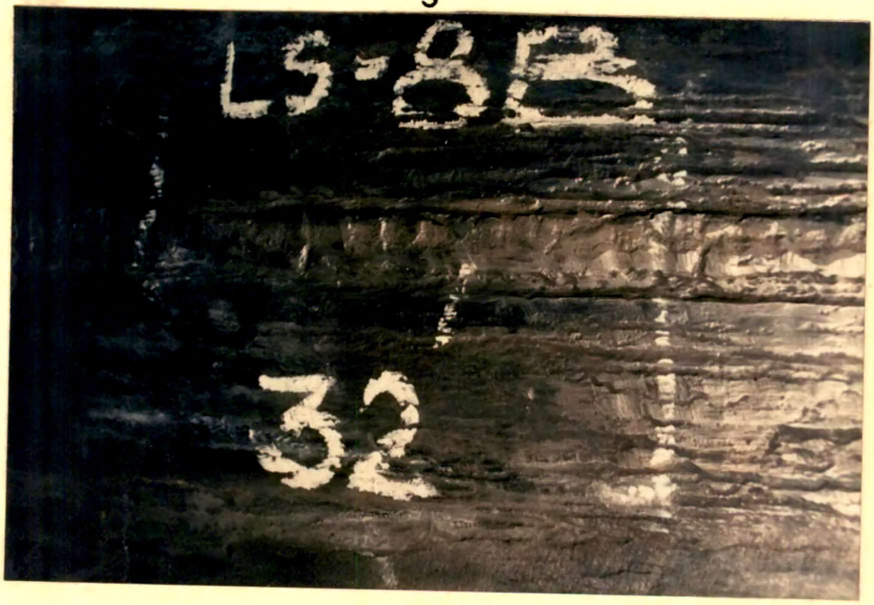

PLATES 1-3 : PHOTOGRAPHS SHOWING EXTENSIVE GROOVING / CHANNELING EFFECTS AND EROSION/CORROSION MARKS ON THE INTERNAL SURFACES OF THE CARBON DIOXIDE ABSORBER WALL.

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PLATES 4-5 : PHOTOGRAPHS SHOWING CORROSION AT THE CIRCUMFERENTIAL WELDS AND FILLET WELDS OF SUPPORT RING BRACKETS IN THE CARBON DIOXIDE ABSORBER

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