COILED TUBING ACID CORROSION
LABORATORY VS. FIELD

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ABSTRACT

Use of Coiled Tubing in underbalanced acid washing is becoming more prevalent in carbonate wells with H_2S production. Corrosion testing under pressures and temperatures representing downhole conditions is used to qualify a corrosion inhibitor loading. Sour conditions warrant testing with differing amounts of H_2S in the gas phase. Safety factors, weight loss and pitting guidelines are employed in the inhibitor design to ensure continued integrity of Coiled Tubing. The thin wall of these tubulars makes corrosion control of the utmost importance.

Evaluations of Coiled Tubing after acid treatments examines the surfaces for defects and uses the perlite layer at the concentric center of the tubing wall to determine material losses. Comparisons of laboratory tests to effects on Coiled Tubing, used to treat wells producing from 0 to 60% H_2S at temperatures of 75° to 110°C (167" to 230°F) are presented. Specifically material deterioration and surface evaluations are compared.

INTRODUCTION

Many areas around the world are using coiled tubing to work on wells that are not only deep with high bottomhole temperatures and pressures but also produce corrosive fluids (H_2S and CO_2). The well temperatures range from 275" to 415°F (135" to 212°C).[1-5] H_2S concentrations can vary from <100 ppm to 60%. There is an array oftreatments typically performed on oil and gas wells, including fracturing, bullhead matrix, underbalanced washing and wellbore cleanout operations.

Washing treatments are typically utilized to clean out scales or debris produced from the reservoir.[1,3,4] Table 1 lists the relative importance of reactions that may occur in the acid treatment of sour wells. Since washing treatments are being performed under a less than overbalanced pressure condition and could be performed to remove iron sulfide, both coiled tubing and the production tubulars are highly vulnerable to increased corrosion potential. Another occasion of concern is during the recovery of spent or partially spent acids.[6]

Some evaluation of the impact of H_2S on the usage of coiled tubing under sour downhole conditions has been performed.[7-9] These evaluations have shown that in a sour environment, a preferred tensile strength of coiled tubing for downhole usage is 80 Kpsi or less. In addition, there is a potential for some damaging precipitation products as the result of acidizing operations, from metallic inhibitor intensifiers and some hydrogen sulfide scavengers. Of paramount importance was the need for as close to true downhole conditions as possible for laboratory testing.

To more realistically evaluate the ability to protect coiled tubing given the increased severity of corrosive conditions during washing treatments, it was necessary to perform some comparisons of laboratory test results to actual effects of the downhole conditions on coiled tubing strings. Corrosion aspects of this paper focus on the loss of weight and, therefore, wall thickness and tubing surface conditions (pitting).

BACKGROUND

A review of the test methods and test environments of NACE (National Association of Corrosion Engineers) and EFC (European Federation of Corrosion) by Hay[10] has recently been published. The fit-for-purpose testing of metals for sour service should be done in an environment reflecting the worst case, using methods and samples appropriate to an application. It was found that more than one method of testing should be employed to avoid missing a point of failure. Several evaluations of oilfield materials for use in H_2S environments have also been conducted.[11-12] Of particular interest is the work by Kane and Wilhelm[11] in which aspects of general corrosion; SSC (Sulfide Stress Cracking); SCC (Stress Corrosion Cracking); and HE (Hydrogen Embrittlement) in H_2S and CO_2, and H_2S and chloride environments are examined. Corrosion and cracking that can occur, as a result of acidizing operations, on high alloy steels, and newer materials, used in the completion of wells with a sour environment, are examined.

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The test methods employed by laboratories in the industry to determine whether a particular loading of corrosion inhibitor and/or intensifier are adequate at actually protecting metals during an acid treatment are not standardized and vary in procedures and conditions. The criteria for passing an inhibitor package are based on weight loss and surface characteristics (pitting). For coiled tubing, these criteria are a weight loss below 0.02 lb/ft2 (0.098 kg/m2) and only minor microscopic pitting.

EXPERIMENTAL

Laboratory Corrosion Evaluation. Detailed procedures currently employed in the evaluation of acid corrosion on coiled tubing samples are outlined in Delorey et al.9 The apparatus currently in use (since October 2000) is shown pictorially in Figure 1 along with the lower pressure apparatus used from 1995 to 2000. The current corrosion cell has a working pressure of 10,000 psi although the associated plumbing and available nitrogen pressure is restricted to a cold starting pressure of 2,000 psi. For tests above 212°F (100°C), the pressure rises to approximately 3,000 psi due to vapor pressure and gas expansion with temperature. Also, the maximum H2S concentration in the vapor space that we can currently use is 200 psi, the maximum vapor pressure of the liquid H2S used to prepare the gas mixtures. This results in a 10% H2S concentration at 3000 psi at test temperature. The current vapor space volume is 300 ml with an acid volume of 150mL. The test coupons are sized to provide a liquid volume to surface area ratio of 33mL/in2. This is in line with the testing guidelines in the API-NACE subcommittee on acid corrosion testing during the late 1970's. Previous apparatus had a maximum pressure rating of 1,800 psi so that the cold starting pressures were restricted to 1,000 psi and an H2S concentration of 20%. The expansion chamber on the previous apparatus was used to provide a vapor space of 450 mL. The change to the current apparatus represents an ongoing attempt to improve the evaluation of materials under more realistic downhole conditions. It is planned in the future to build a gas mixing chamber and a transfer pump that will permit full simulation of downhole conditions.

Coiled Tubing String Monitoring. Samples of coiled tubing were cut from the end of a string before and after one to several treatments. These samples are visually inspected for obvious surface defects (pits and cracks) and calipered around the circumference to check wall thickness. The samples are then split into two halves longitudinally so that both the inside and the outside surfaces can be examined. The samples are then cleaned of any rust or scale by pickling in 15% hydrochloric acid inhibited with 0.2% of conventional corrosion inhibitor. If a crack exists, the existence of thinning or necking is used to determine whether simple overtensioning of the tubing was responsible for the crack or whether SCC, SSC, hydrogen induced cracking (HIC) or stress orientated hydrogen induced cracking (SOHIC) may be a factor. Typically, a strategically chosen cross section is prepared to complete diagnosis. In this procedure, the sample is potted in epoxy and ground down to the point of interest and polished with gradually finer mesh abrasive until a final polishing with 0.02 micron grit alumina.

The preparation of a cross section sample will also be necessary if corrosion or mechanically formed pits are present. The cross section sample can reveal cracking associated with the pits. A micro hardness test can also be used to evaluate the quality of the welded seam or tubing joints. Poor welding techniques can result in a large heat affected zone, HAZ. To evaluate the grain structure of the tubing and the HAZ, the polished surface is etched with a 2% nitric acid and 98% methanol solution. The presence of pearlite and ferrite grains can be examined with polarized light. The existence of a pearlite band at the center of the tubing cross section can be used to determine whether significant corrosion has occurred on the outside or inside of the tubing, **Figure 2.**

RESULTS

Coiled Tubing String 14139. This is an 80,000 tensile strength tapered coiled tubing string with an 1.5-inch outside diameter and originally 13,164 feet in length. It was put into service August, 2000. This coil and the information gained is a good example of the two major concerns in working in sour environments, namely uphole SSC under nonacidic conditions and accelerated acid corrosion under downhole conditions. Samples of the tubing were cut off prior to and after several treatments to evaluate wear and corrosion. The jobs performed were a milling job on a well with 23% H2S and two acid treatments on two zones in the same well with 55 to 60% H2S in the zones. The details of each treatment are listed in **Table 2.**

Since milling in a sour environment can lead to uphole SSC, a corrosion and stress cracking inhibitor was pumped periodically throughout the operation. This inhibitor was composed of a filming amine and a keto-acid. Samples of the coil showed black scale on the outside representing incorporation by the inhibitor of iron sulfide to protect the coil from cracking. Evaluation showed no cracking or pitting evident after this milling operation, **Table 3.**

The first acid treatment was two stages (2 m3 per stage), and the acid was designed for an exposure time of 4 hours

(Table 4) at 212°F (100°C). The corrosion rate criteria used was less than 0.02 lb/ft2 for the exposure time at the bottomhole temperature. The zone produces 55% H2S with a bottomhole pressure of 35.0 MPa (5076 psi). The treatment consisted of spotting the acid across the perforations and then squeeze as pulling the coiled tubing up through the perforations. Once above the perforations, the remaining acid was squeezed into the formation with diesel being used as the flush. Then the well was to be unloaded with nitrogen. The squeeze treatment was performed twice as outlined above per each stage.

The second acid treatment **(Table 2)** using this coil consisted of a wash with 2 m3 of acid **(Table 4)** followed by a squeeze of the remaining acid (10 m3) into the formation. This zone, as the zone previously treated downhole, produces H2S *(55%).* As before a 4-hour exposure time was used in the design of the acid corrosion inhibitor package.

After the acid treatments, the inside of tubing samples showed no corrosion. All wall thickness losses **(Table 3)** were on the outside of the coil. The outside surface of the samples exhibited some pitting, Figure 3. The sample after the second acid treatment had some pits with a depth of penetration of 0.5 mm or 20% of the wall thickness, Figure 4. This large pit incubated on the plastically deformed surface. The plastic deformation was indicated by a sinking profile of the subsurface layers (pearlite and ferrite bands) on the outside of the tubing, **Figure 5.** This type of problem is impossible to predict or to test for in the laboratory. The other pitted areas showed spherical morphology and did not indicate any crack incubation.

Corrosion weight loss calculated based on the wall thickness losses, **Table 5,** were three to 10 times the 0.02-lb/ft2 criteria used in laboratory evaluations. Actual laboratory corrosion test data, **Table 5,** were obtained under sweet conditions prior to the treatments. This particular area has restrictions on the use of certain chemicals in well treating and as such the inhibitor used was at its maximum range of effective protection. However, this coil had a higher than normal carbon content (36% more). **Figure 6,** shows the high carbon content in the microstructure. This higher carbon content should have offered more inherent resistance to acid corrosion. The acid systems were not tested under sour conditions because the inhibitor was already being used at its maximum effective concentration. Based on all the information, it was deemed acceptable to sacrifice some of the coil in order to perform the treatment.

Coiled Tubing String 12940. This is also an 80,000 tensile strength tapered coiled tubing string but with a 2.0-inch outside diameter and originally 15,537 feet in length. It was put into service September, 2000. Samples of the tubing were cut off prior to and after several treatments to evaluate wear and corrosion. An acid job performed was on a well with 24% H2S and a bottomhole pressure of 40.7 MPa (5903 psi) and temperature of 94°C (201 °F). The details of this treatment are listed in **Table 6.**

The acid treatment consisted of a wash with 15 m3 (3,963 gals.) of acid **(Table 4, Acids C and D)** followed by a squeeze with 103 m3 (27,213 gals.) into the formation. A 4-hour exposure time was used in the design of the acid corrosion inhibitor package. At the time, the systems were designed only a squeeze treatment was to be conducted. Therefore, the system was not tested with H2S. Testing of the treating fluids in 2002 with H2S at 10% showed a weight loss of 0.047 lbi ft2, which is over the acceptable 0.02 lb/ft2 criteria, **(Table 7).**

Table 8 compares physical evaluations of the coil samples from before and after the acid treatment. As with the sample above (String 14139) after the milling treatment, the after coil sample outside surface was covered with a black scale, with some oil contamination. No variance in wall thickness was measured. Therefore, no calculation of weight loss could be determined. **Table 7** gives **a** comparison of laboratory corrosion test data from 2000 to 2002. Pickling of the sample revealed random external pitting **(Figure 7),** large pits (1.5 mm by 0.4 mm) at a mechanically made grooved area **(Figure 8)** and transverse small superficial cracks (less than 0.1 mm deep) agglomerated in one area **(Figure 9).** Closer evaluation of the pits, using metallographic techniques of the grooved area, showed the pits to be the result of plastic deformation, **(Figure 10).** It was also determined that no cracks were found to be incubating at this site. The superficial cracks that were found were not incubated from the pits, **(Figure 11).** They were determined to be the result of sulfide stress. There was not enough exposure time during the treatment for the cracks to propagate. The pitting indicated above, again, is a result of conditions that were not anticipated.

Coiled Tubing String 9978. This is a 70,000 tensile strength coiled tubing string with a 1.5-inch outside diameter. Samples of the tubing were cut off prior to and after an acid treatment consisting of a wash followed by a squeeze on a well producing 40% H2S with a bottomhole pressure of 37 Mpa (5367 psi). The details of the treatment are listed in **Table 9.** The acid treatment wash (*6.5* m3) and squeeze (35 m3) were designed with acids having a corrosion protection based on an exposure time of 4 hours **(Table 4, Acids E and F)** at 225°F (107°C).

After the acid treatment, the inside and outside of tubing samples showed no corrosion, **Figures 12 and 13.** Also, no wall thickness losses **(Table 10)** were found. **Table 11** lists the corrosion test results from the laboratory compared to the accepted criteria.

CONCLUSIONS

1. Examination of coiled tubing samples before and after underbalanced acid treatments show greatly accelerated corrosion on the outside of the coil due to the commingling of the inhibited acid and H_2S.
2. The increased corrosion observed can be predicted with good correlation by laboratory acid corrosion tests under simulated downhole pressure, temperature and H_2S content.
3. Increased inhibitor and intensifier concentrations are required to adequately inhibit against these adverse conditions.
4. In non-acid treatments, the use of anticracking inhibitors is effective in minimizing the tendency towards sulfide stress cracking and preventing the propagation of cracks in small pits and localized surface defects

FUTURE WORK

1. Extend testing to higher pressures.
2. Investigate effect of gas to fluid volume ratio on acid corrosion in H2S environments.
3. Acid corrosion under H2S environment studies should be expanded to other metals

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Table 1
Relative Importance of H_2S reactions

	CONVENTIONAL OVER BALANCED TREATMENTS	UNDER BALANCED COIL TREATMENTS
Iron sulfide precipitation	Formation damage important in small wellbore treatments. Much less important on large high rate acid squeezes	Important on small jobs but the use of coil reduces the amount of iron contamination
Sulfur Precipitation	Very important on large acid treatments because of the effect of thermal and pressure shock on the H_2S/H_2S_n equilibrium. Less serious sulfur precipitation can also result from ferric iron contamination of acid	Less important on coil jobs because of the limited flow rates and volumes.
H_2S /Additive Precipitation	Formation damage possible	Formation damage possible
Corrosion in Acid / H_2S	Generally there is little contamination of the acid by H_2S so the inhibitor requirements are the same as in sweet wells. Can be a problem in low rate acid removal of iron sulfide scales	High concentrations of inhibitor required to prevent damage to the outside of the coil due to the flow of H_2S and partially spent acid
Sulfide Stress Cracking	Generally not a problem provided tubulars selected for sour service. Avoid shut-in of early flowback	Anti-cracking inhibitor should be circulated around outside of the coil so that the metal near the surface will not be embrittled
H_2S/Elastomer Reactions	"0-Rings" and Valve seats need to be chosen for sour service. Ball sealers should be checked for compatibility with H_2S	Similar requirements apply. Also the 'blow-out preventers' for the coil need to be H_2S resistant

FLUID	VOLUME	RATES	PRESSURES	TEMP., °F (°C)	H_2S	CONTACT TIME, HRS
Water/N_2	N/A	Water 100 to 160 Limin N_2 15 m^3/min	N/A	187 (86)	23%	77
			irst Acid Treatment			
Acid A	2 m^3	1^{st} Stage 50 to 200 Limin Avg. 80 Limin	20.9 to 32.7 MPa	230 (1^{10})	60%	4:00 Inside 4:00 Outside A Diluted Acid Blend was on the outside of the coil over 12 hours
Acid A	2 m^3	2^{nd} Stage 20 to 150 L/min Avg. 70 L/min	14.9 to 35.1 MPa	230 (110)	60%	0:43 Inside 1:00 Outside A Diluted Acid Blend was on the outside of the coil over 18 hours
			cond Acid Treatment			
Acid B/N_2	2 m^3	Wash70 to 100 Limin Avg. 80 L/min N_2 Rate	23.8 to 35 MPa	212 (100)	55%	Total Washing Time Acid on Inside and Outside 0:27
Acid B/N_2	10 m^3	Squeeze 100 to 177 L/min Avg. 115.6 Limin	25 to 35.1 MPa	212 (100)	55%	Total Acid Contact Through Both Wash and Squeeze Inside 2:37 and Outside 2:00

Table 3
Coiled Tubing String 14139 Samples Physical comparison Before and After Usage

Treatment	Wall Thickness, millimeters		Comments
	Before	**After**	
Milling	2.68 – 2.72	2.68 – 2.72	Black scale on all surfaces but no abnormal corrosion.
First Acid Treatment	2.68 – 2.72	2.58 – 2.6	All wall losses on OD.
Second Acid Treatment	2.58 – 2.6	2.55	Wall losses on OD, some large pits were 0.5 mm deep.

Additive	Concentration	Purpose
Acid A – 15% HCI		
Alcohol Free Ethoxylated Amine Inhibitor	6010 L/m^3	Acid corrosion Inhibitor
45% Solution Potassium Iodide	2.2 L/m^3	Acid Corrosion Inhibitor Intensifier
Cuprous Chloride Reduction Catalyst	*0.5* Kg/m^3	Iron Control
Sodium Salt of NTA	*5.0* Kg/m^3	Iron Control
Acid B – 15% HCI		
Alcohol Free Ethoxylated Amine Inhibitor	60.0 L/m^3	Acid corrosion Inhibitor
45% Solution Potassium Iodide	2.2 L/m^3	Acid Corrosion Inhibitor Intensifier
Cuprous Chloride Reduction Catalyst	0 *5* Kg/m^3	Iron Control
Sodium Salt of NTA	10.0 Kg/m^3	Iron Control
A Blend of Oxyalkylated Alcohols and Alkylphenol Formaldehyde Resins	1.0 L/m^3	Nonionic Surfactant
Mannich Reaction Base Inhibitor	15 L/m^3	Acid Corrosion Inhibitor
Calcium Chloride	*50* Kg/m^3	Anhydrite Dissolution Control
Formic Acid	*50* L/m^3	Acid Corrosion Inhibitor Intensifier
Acid D – 28% HCI		
Mannich Reaction Base Inhibitor	15 L/m^3	Acid Corrosion Inhibitor
Calcium Chloride	*50* Kg/m^3	Anhydrite Dissolution Control
85% Formic Acid	*50* L/m^3	Corrosion Inhibitor Intensifier
50% Amphoteric Surfactant in Alcohol and water	30 L/m^3	Non-emulsifying Foamer
Blend of Oxyalkylated Alcohols Oxyalkylated Alkylphenol Formaldehyde Resins in an Isopropanol Solution	2 L/m^3	Nonemulsifier
Quaternary Amine and Propargyl Alcohol	30 L/m^3	Corrosion Inhibitor
85% Formic Acid	50 L/m^3	Corrosion Inhibitor Intensifier
Sodium Salt of NTA	*5* Kg/m^3	Iron Sequestering Agent
Phosphonic Acid Derivative	1 L/m^3	Scale Inhibitor
Methyl Siloxane Emulsion	0.1 L/m^3	Antifoam Agent
Acid F- 28% HCI		
Blend of Ethoxylated Alcohols, Glycols Aromatic Solvents	2 L/m^3	Nonemulsifier
Quaternary Amine and Propargyl Alcohol	30 L/m^3	Corrosion Inhibitor
Formic Acid	30 L/m^3	Corrosion Inhibitor Intensifier
Sodium Salt of NTA	*5* Kg/m^3	Iron Sequestering Agent
Phosphonic Acid Derivative	1 L/m^3	Scale Inhibitor
Methyl Siloxane Emulsion	0 1 L/m^3	Antifoam Agent

Table 5

Treatment	Acid Blend	Exposure Time, Hours		Corrosion Rate, lb/ft^2		
		Design	Actual	2000 Laboratorv Tests	2002 Laboratorv Tests	Actual
Milling	None	N/A	N/A	N/A	N/A	0
First Acid Treatment	A	4	4	0.018 (Sweet)	0.24 (10% H_2S)	0.1771
Second Acid Treatment	B	4	<4	0.018 (Sweet)	0.24 (10% H_2S)	0.0644

FLUID	VOLUME	RATES	PRESSURES	TEMP., °F (°C)	H_2S	CONTACT TIME, HRS
Acid C/N_2	15 m^3	Wash 10 to 220 L/min N_2 Rate 30 to 45 m^3/min	14.3 to 37.3 MPa	201 (94)	24%	Total Washing Time Acid on Inside and Outside 8 hours
Acid D/N_2	103 m^3	Squeeze 100 to 177 L/min Avg. 115.6 L/min	9.5 to 43.6 MPa	201 (94)	24%	Total Acid Contact on Inside and Outside 11 hours

	2000	2002
C	0.01 lb/ft^2 (Sweet, 16 hours)	0.047 lb/ft^2 (10% H_2S, 3 hours)
D	0.01 lb/ft^2 (Sweet, 16 hours)	0.056 lb/ft^2 (10% H_2S, 3 hours)

Table 8
Coiled Tubing String 12940 Samples Physical comparison Before and After Usage

Treatment	Wall Thickness, mm		Comments
	Before	After	
Acid reatment	3.38 – 3.45	3.38 – 3.45	Outside covered with black scale contaminated with oil. There was no apparent abnormal corrosion or pitting. Cutting open the sample to examine the inside revealed no corrosion, even on the weld flash. Pickling of the sample confirmed no corrosion on the inside surface but revealed pitting and cracks on the outside surface.

FLUID	VOLUME	RATES	PRESSURES	TEMP., °F (°C)	H_2S	CONTACT TIME, HRS
			Acid Treatment			
Acid E	6.5 m^3	Wash - 100 L/min N_2 – 10 m^3/min	10 to 23 MPa	225 (107)	40%	Washing Time Acid on Inside – 1:20 and Outside 2:50
Acid F	35 m^3	Squeeze – 110 L/min N_2 – 15 m^3/min	10 to 34.9 MPa	225 (107)	40%	Acid Contact Through Squeeze Inside 6:56

Treatment	Wall Thickness, millimeters		Comments
	Before	After	
Acid Treatment	2.84 – 2.92	2.84 – 2.92	No change in surface quality of tubing as a result of the acid treatment in an H_2S environment

Treatment	Acid Blend	Exposure Time, Hours		Laboratory Corrosion Rate, lb/ft^2	
		Design	Actual	Design	Actual
Acid Wash	E	4	–3	0.0165 (10% H_2S)	<0.02
Acid Squeeze	F	4	–7	0.01 (Sweet)	<0.02

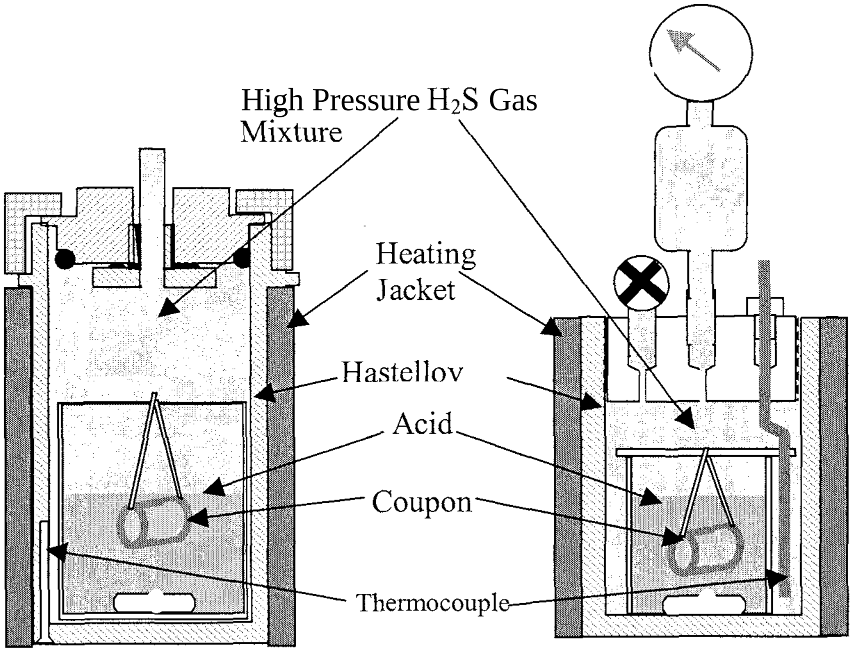

Figure 1 - Diagram of Acid Corrosion Test Cells Used in Pre-2000 (right) and Current (left).

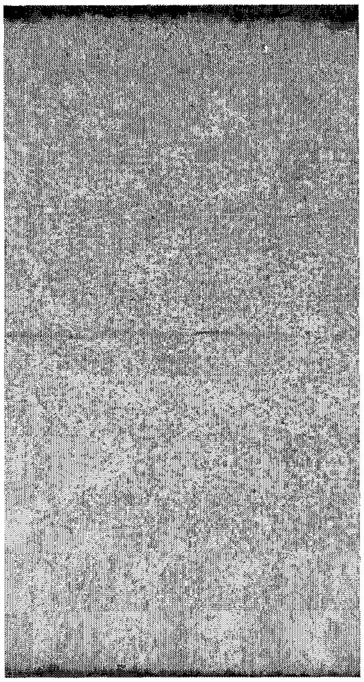

Figure 2 - Photograph of Microscopic View of Cross Section of Pearlite Band at the Center of Coil Sample from String 14139

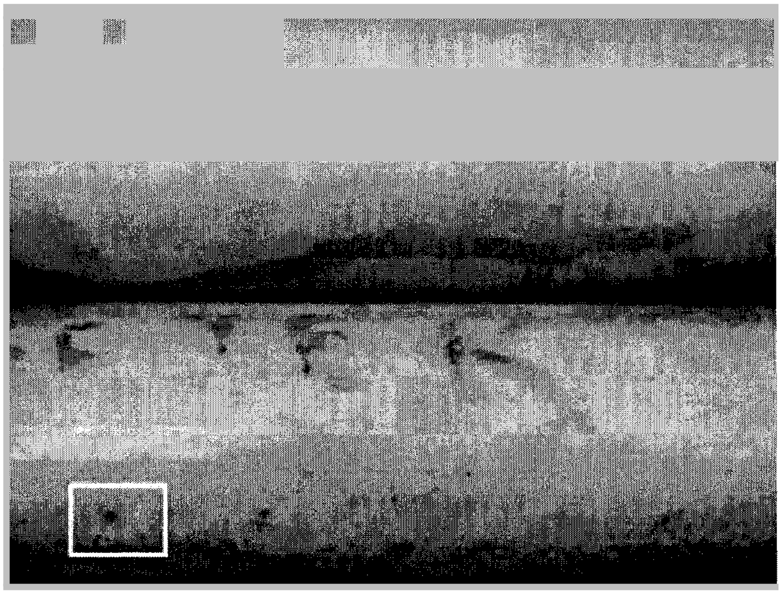

Figure 3 - Photograph of the Outside of the Coil Sample from String 14139 After Acid Treatments[13]

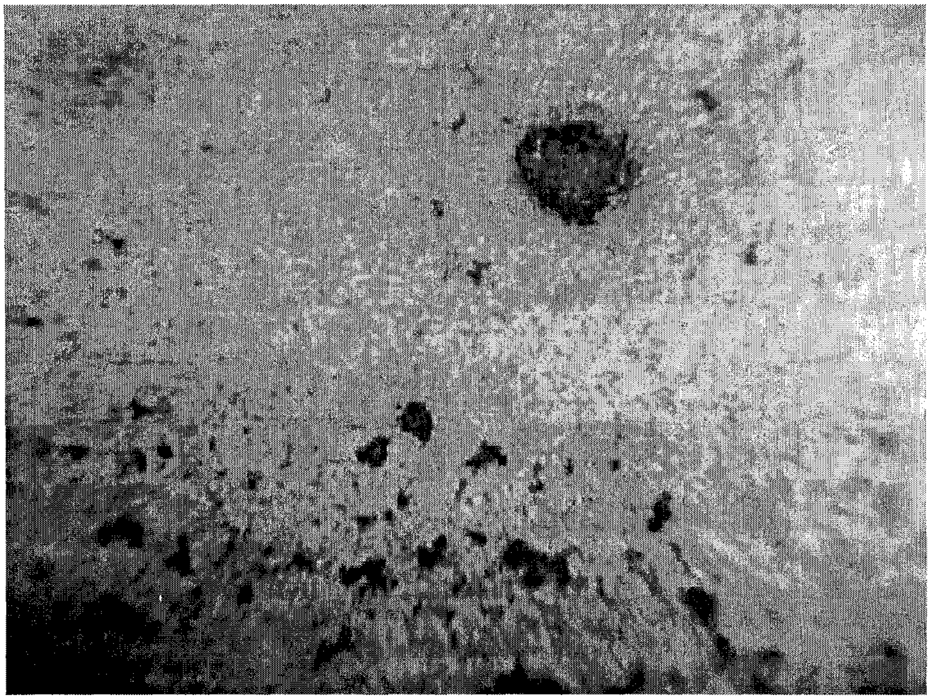

Figure 4 - Photograph of Microscopic View of Cross Section of Large Pit in Outside Surface of Coil Sample from String 14139

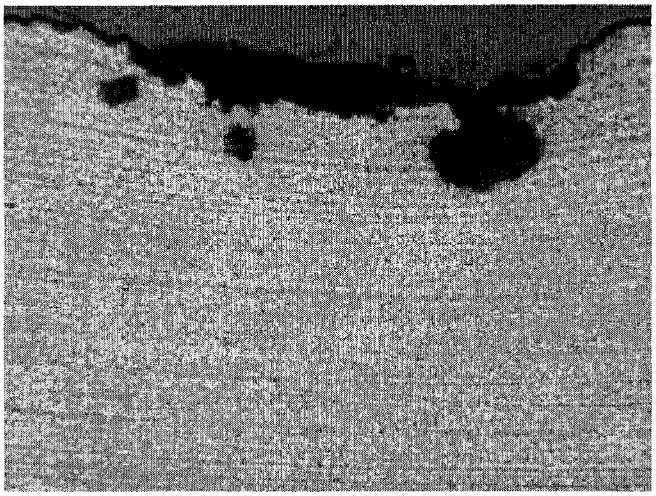

Figure 5 - Photograph of Microscopic View of Plastic Deformation of Coil Sample from String 14139

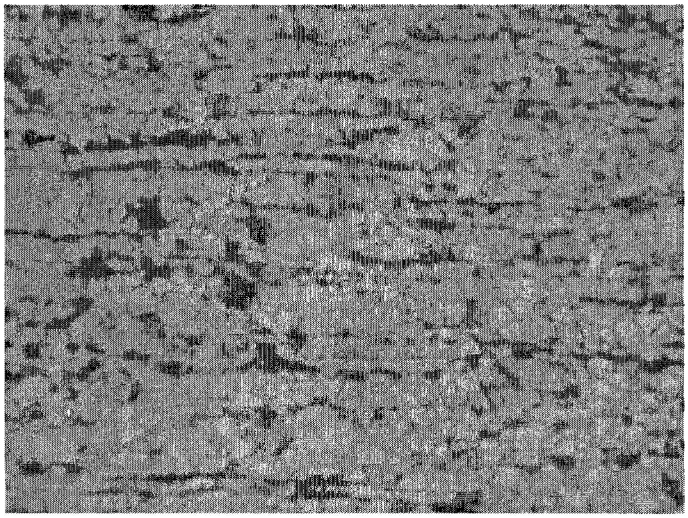

Figure 6 - Photograph of Microscopic (X800) View of the Microstructure of Coiled Tubing String 14139

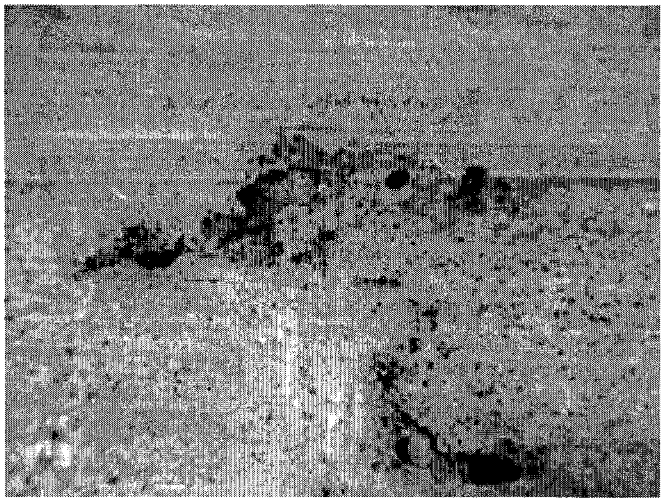

Figure 7 - Photograph of Pitted Area on the Outside of Coil Sample from String 12940[14]

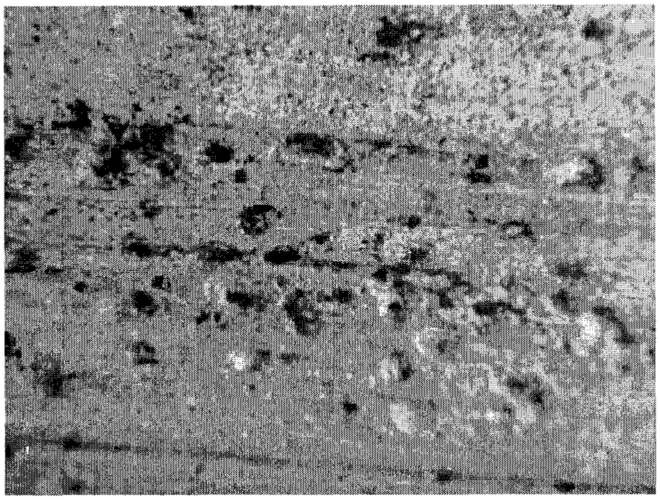

Figure 8 - Photograph of Grooved Area on the Outside of Coil Sample From String 12940

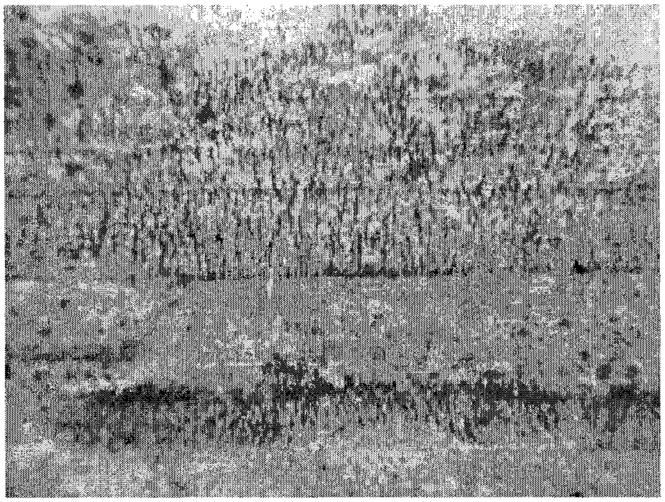

Figure 9 - Photograph of Area of Superficial Cracks on the Outside of Coil Sample from String 12940

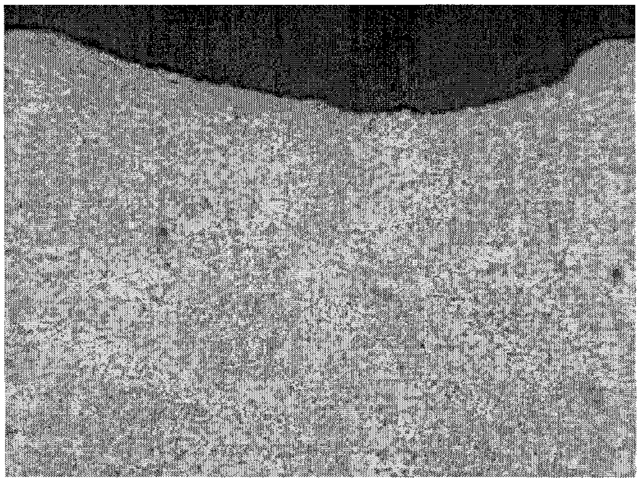

Figure 10 - Photograph of Microscopic View of a Longitudinal Section of the Grooved Area Found on the Outside of the Coil Sample from String 12940

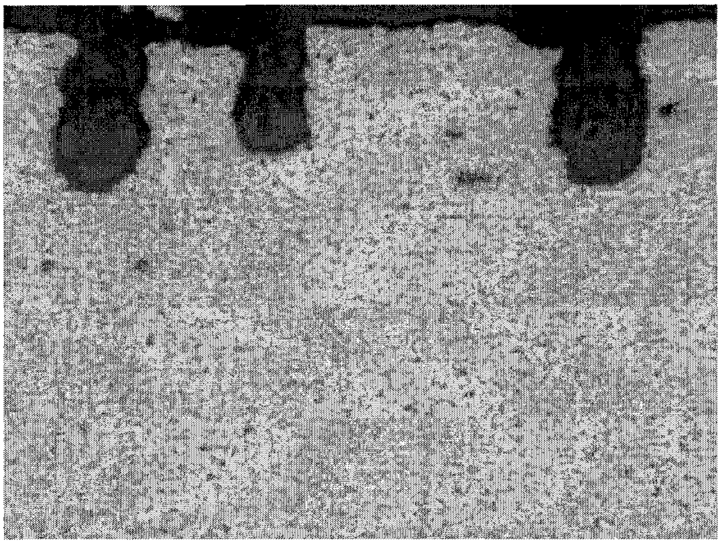

Figure 11 - Photograph of Microscopic View of a Longitudinal Section of the Superficial Cracked Area of the Coil Sample from String 12940

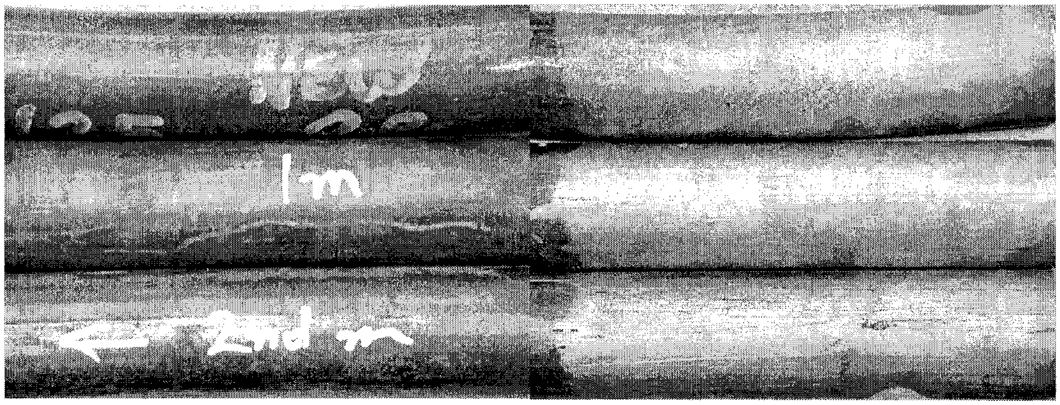

Figure 12 - Photograph of the Outside of the Coil Samples from String 9978 Before and After Acid Treatment[15] - Left picture is the samples as received and the right after a pickling in the laboratory.

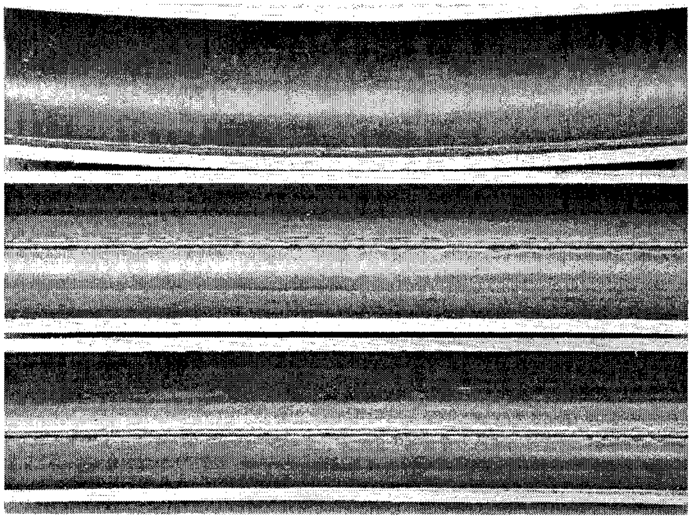

Figure 13 Photograph of Interior Including the Weld on Coil Samples from String 9978 after Before and Acid Treatment