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XX FIELD - WELL LL-265 Corrosion Behaviour of Stainless Steel Materials in Downhole Conditions

1. Scope of Work

The purpose of this study is to evaluate the corrosion behaviour of the candidate tubular materials in LL-265 downhole conditions. 4 materials have been tested:

- Super 13% Cr (SM13 CRS)
- Duplex Steel 2205 (UNS 31803)
- 22% Cr (SM 2242)
- 25% Cr (SM 2535)

The 2205 duplex steel was tested as part of the RCS downhole corrosion tool (DCMS).

The other materials were tested as part of the ADCOSH assembly. The ADCOSH has been developed jointly for evaluating the risks of pitting corrosion, crevice corrosion and stress corrosions in downhole conditions.

2. Well Chemistry

The well LL-265 is a single oil producer, re-completed in 1996, which produces 23,800 bbl/day from Unit "H". The BSW ranges from 5 to 10%, with 115 to 117 g/l of chloride. The associated gas contains 223 ppm of H_2S and 4.4% of CO_2 . The wellhead pressure is 1100 psi (75 bars). At the producing depth (8400 ft), the pressure is approx. 100 bars and the temperature $120^{\circ}C$.

Based on the available models, the well is defined as very corrosive. The corrosion rate for carbon steel is expected to be in the range 0.5 to 5 mm/year, which justifies the use of higher grade tubulars.

The sketch of the well is shown in Appendix 1.

3. Materials and Tools

The chemical analysis of the tubular materials under test is given hereafter:

	Cr	Ni	Mo	Ti	С	Si	Mn	Fe	YS	EL
SM 13 *	12.1	5.8	0.19	0.08	0.01	0.23	0.42	Bal.	103	27
Duplex 2205	22.0	5.10	3.20		0.01	0.40	0.60			
SM 2242 *	22.0	44.2	3.22	0.079	0.01	0.23	0.66	26.6	119	17
SM 2535 *	25.0	31.3	3.21		0.01	0.20	0.60	Bal.	118	20

YS = Yield strength (in k psi)

EL = Elongation (%)

* = Supplied by Sumitomo Metal Industries

The DCMS and ADCOSH have been mounted in tandem onto a slick line set and a shock absorber by a wireline crew.

The DCMS tool sensor is a cylindrical Corrosometer probe T10 (10 mils thickness). The meter, the data logger and the battery set are enclosed in a cylinder housing, $1-\frac{1}{4}$ " dia,, 1.20 meter long. The data logger records the probe metal loss and temperature versus time.

The ADCOSH housing is a $1-\frac{1}{4}$ " dia, 1.50 meter cylinder containing 3 types of coupons for each material.

- a strip coupon (76 x 4.50 x 1.50 mm) for determining the corrosion rate and pitting tendency
- a cylindrical coupon (slow strain rate test coupon), 3.81 mm dia, for determining the tensile properties of the material after exposure to the downhole environment.
- a bent strip coupon with a stressing fixture, following NACE TM 0177 for determining the susceptibility to SSC.

The configuration of the coupon holders permits also to evaluate the resistance to crevice corrosion.

The photographs of the various samples and fixtures are shown in Appendix 5.

4. <u>Job Description</u>

The complete assembly was installed on 3/10 onto a 2.31" XN nipple at depth 7,960 ft.

The tool battery was activated as per the usual procedure and the probe reading interval was set at 2 hours.

The assembly was retrieved on 1/12/2001. No incident was reported during the installation or retrieval of DCMS, nor during the 60 days exposure of the tools and samples. (See Wireline Reports in Appendix 2)

5. Results

The detailed data report of the DCMS run (metal loss versus time) is given in Appendix 3.

The detailed coupon analysis, tensile tests and SSC tests are given in Appendix 4.

a. Uniform Corrosion Rate

The uniform corrosion rate is negligible for all materials, as shown by the Table hereunder.

	Average Corrosion Rate (over 60 days)
SM 13 CR	0.052 mpy
Duplex 2205	0.073 mpy
SM 2242	0.006 mpy
SM 2535	0.020 mpy

This data confirms the well-known resistance of SS and nickel alloys to the CO₂ corrosion.

The temperature at the probe depth is practically constant at 250 deg F.

b. Pitting Corrosion

No trace of pitting corrosion has been detected during the 60-days exposure.

c. Intergranular Cracking

The test coupons have been submitted to a bend test as per ASTM A282, to determine the resistance to intergranular cracking. No evidence of fissures or cracks was found on the subject materials.

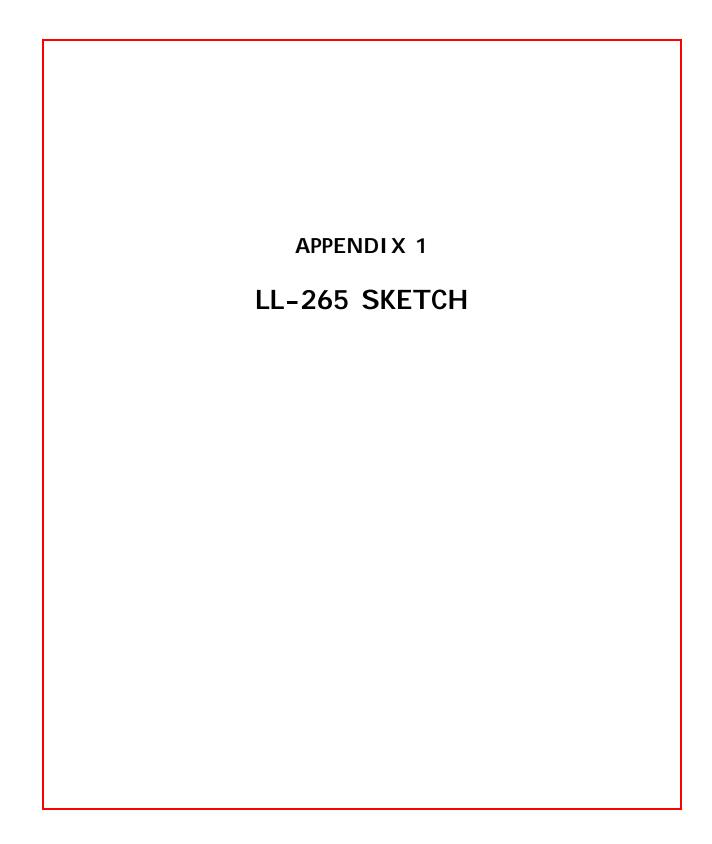
d. Susceptibility to Stress Corrosion Cracking

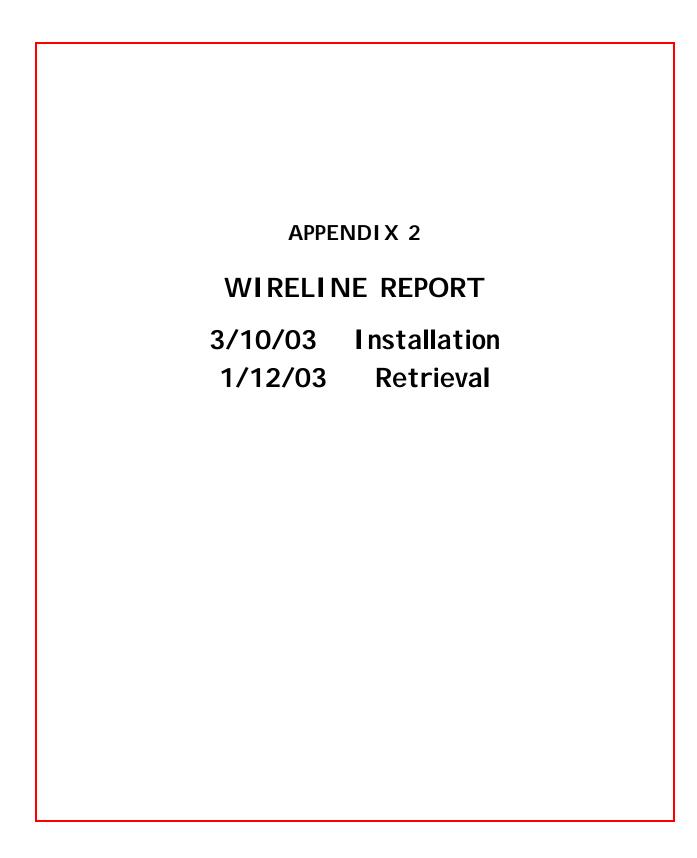
The "bent coupons", stressed at 70% of the actual yield strength prior to exposure, did not show any evidence of fissures or cracks after 60-days exposure.

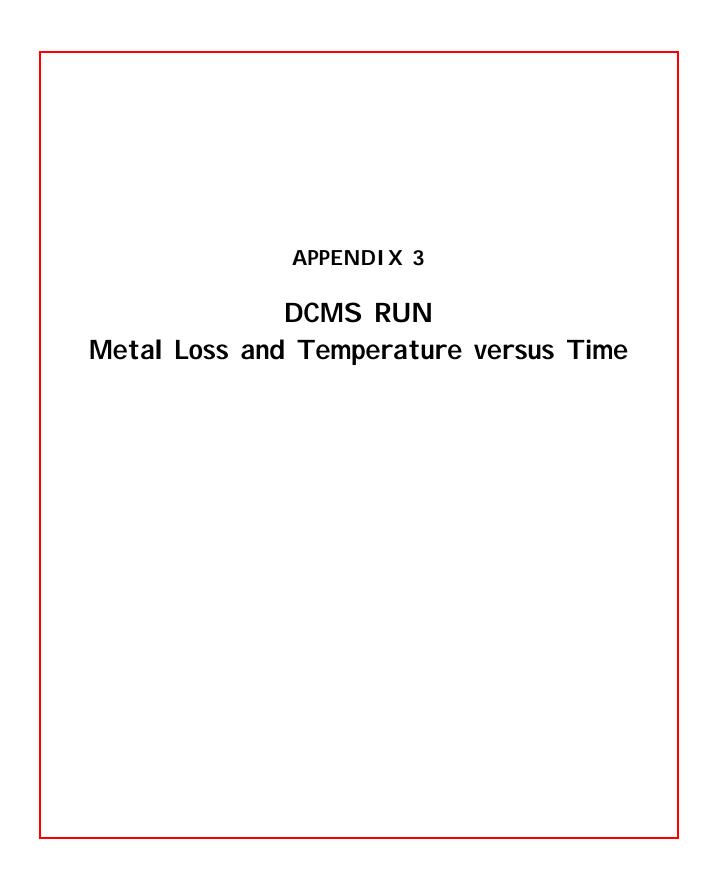
e. Mechanical Properties

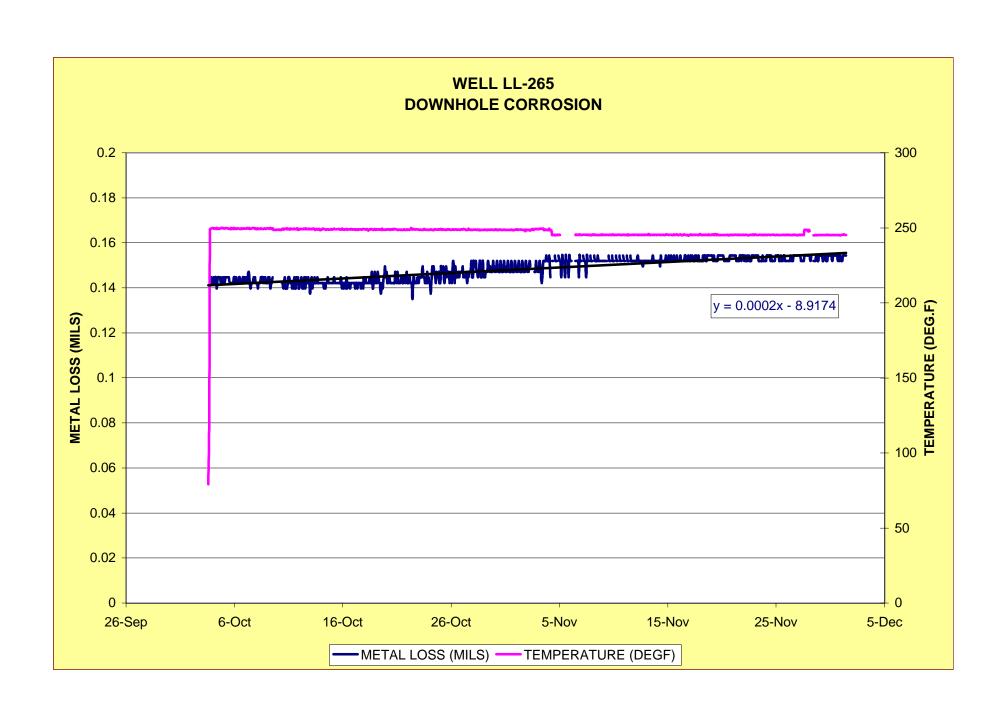
The tensile tests carried out after exposure did not show any alteration of the mechanical properties of the samples after 60-days exposure.

In summary, the materials under test are immune from corrosion in the LL-265 downhole conditions.









APPENDIX 4 COUPON ANALYSIS REPORT WEIGHT LOSS TENSILE TESTS INTERGRANULAR CRACKING STRESS CORROSION CRACKING

SAMPLE IDENTIFICATION	Test coupons supplied by the client for down hole materials testing (detailed descriptions and identifications are included in the body of the report) LTS Sample #500/0533
TEST DESCRIPTION	Included in the body of the report

Background and General Information

The client supplied one set of test coupons reportedly removed from three different materials identified as follows:

- 1. 13% Cr Martensitic Stainless Steel (SM13CRS-95)
- 22% Cr 42% Ni Austenitic Alloy (SM2242-110)
- 25% Cr 35% Ni Austenitic Alloy (SM2535-110)

The set of test coupons from each of the above materials consisted of the following:

1. One normal coupon

The coupon is a strip type test coupon. The test coupon is for determining the corrosion rate and susceptibility to corrosion/cracking in the down hole environment. The test coupon is exposed to the down hole environment in the non-stressed condition.

One bent coupon

The coupon is a strip type test coupon. The test coupon is for determining the susceptibility of the material to stress corrosion cracking in the down hole environment. The test coupon is exposed to the down hole environment in the stressed condition.

One cylindrical coupon

The test coupon is a round tensile test specimen to determine the extent of any deterioration in the tensile properties of the material after exposure to the down hole environment. The specimen is exposed to the down hole environment in the non-stressed condition and tested for the tensile properties after the exposure.

The test coupon identifications supplied by the client are as follows:

	Test Coupon Identification			
Material	Normal Coupon	Bent Coupon	Cylindrical Coupon	
13% Cr - Martensitic Stainless Steel	13A1	13B1	13C1	
22% Cr - 42% Ni – Austenitic Alloy	22A1	22B1	22C1	
25% Cr - 35% Ni – Austenitic Alloy	25A1	25B1	25C1	

The client supplied copies of the manufacturer's inspection certificates for each of the materials from which the specimens/test coupons were reportedly removed. The inspection certificates provided information including material/product type, size, specification number, heat no., heat treatment/processing, chemical composition and mechanical properties.

As requested by the client, Lonestar Technical Services carried out several procedures on the test coupons supplied. The procedures carried out are listed in Tables 1, 2 and 3, for the normal coupons, bent coupons and cylindrical coupons respectively.

A report of the findings for each of the materials is prepared and presented separately in three different sections. The report includes information obtained from the inspection certificates supplied by the client, observations before and after the test, test results and relevant photographs.

Table 1 – Procedures carried out on each 'Normal Coupon'

	Description of Procedure
1.	Identification marking on coupon
2.	Visual examination before inserting the coupon into the coupon holder (ADCOSH unit) – photograph and description of visible condition of coupon
3.	Measurement of initial weight of coupon after cleaning the machined coupon in LR grade acetone and drying
4.	Insertion or the coupon into the coupon holder as instructed by the client
5.	Visual examination after down hole exposure - photograph and description of visi- ble condition of the coupon
6.	Measurement of final weight of coupon after initial cleaning using a nylon brush and dilute solution of detergent and final cleaning in LR grade acetone and drying
7.	Calculation of loss of weight and corrosion rate in mils per year
8.	Bend test following ASTM A262 Practice E as a guideline to determine whether the coupon displays any evidence of intergranular cracking in the environment of exposure
9.	Metallography of the test coupon after the bend test – the section was examined and typical micrographs taken to record the observations at and away from the bend location

Table 2 - Procedures carried out on each 'Bent Coupon'

Description of Procedure

- 1. Identification marking on coupon
- Visual examination before stressing the coupon in the specimen stressing fixture photography and description of visible condition of the test coupon
- Preparation of the specimen stressing fixture and stressing of the test coupon following NACE TM0177 as a guideline specification
- Visual examination after stressing the test coupon in the specimen stressing fixture

 photography and description of visible condition of the test coupon
- Insertion of the coupon into the coupon holder (ADCOSH unit) as instructed by the client
- Visual examination of the test coupon in the specimen stressing fixture after down hole exposure - photography and description of visible condition of the test coupon
- 7. Visual and stereomicroscopic examination of the wide faces of the test coupons after unloading and cleaning the test coupon, to determine whether the coupon displays any evidence of stress corrosion cracking in the environment of exposure following NACE TM177 as a guideline specification

Table 3 - Procedures carried out on each 'Cylindrical Coupon'

Description of Procedure

- 1. Identification marking on coupon
- Visual examination before insertion of the coupon into the coupon holder (ADCOSH unit) – photograph and description of visible condition of coupon
- Insertion the coupon into the coupon holder (ADCOSH unit) as instructed by the client
- Cleaning and visual examination of the coupon after down hole exposure photograph and description of visible condition of coupon
- 5. Tensile test following ASTM E8 as a guideline specification

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13% Cr Martensitic Stainless Steel

Information Obtained from Manufacturer's Inspection Certificate

ltem	Description			
Material (product)	Seamless steel casing test sample SM13CRS-95 plain end			
Standard specification	SM13CRS-95			
Heat No.	F815020			
Size	OD: 165.2mm, WT: 25.6mm			
Heat treat- ment/processing	Quenched and tempered			
Chemical composition	Reported to be within the ranges specified for various elements			
Tensile properties	Reported to meet the specified requirements for various parameters			

Visual Examination and Photographic Documentation of Observations

- All three coupons (13A1, 13B1 and 13C1), normal, bent and cylindrical, display evidence of discoloration after exposure to the down hole environment. The coupons display a dark color when compared with the original bright metallic luster of the coupons before exposure.
- 2. There is no evidence of cracking or pitting in any of the coupons.

Photographs displaying the condition of the coupons before and after exposure are presented at the end of this report.

Determination of Corrosion Rate

The corrosion rate was calculated after determining the weight loss in the 'Normal Coupon' (13A1), and considering down hole exposure for a period of 60 days as reported by the client. The data recorded/calculated are as follows:

Parameter	Value
Initial weight of coupon, grams	4.1410
Final weight of coupon, grams	4.1393
Loss of weight of coupon, grams	0.0017
Corrosion rates, mils per year (mpy)	0.052

Bend Test

A bend test was carried out on the 'Normal Coupon' (13A1) following ASTM A262 Practice E as a guideline to determine whether the coupon displays any evidence of intergranular cracking in the environment of exposure. The nominal test coupon dimensions are as follows:

Length: 76.4mm, Width: 4.6mm, Thickness: 1.5mm

After the down hole exposure, the test coupon was bent through 180° around a mandrel having a diameter equal to 4 times the thickness of the test coupon.

The surfaces of the test coupon were examined on a stereomicroscope. The test coupon displays no evidence of the presence of fissures and/or cracks.

Metallography

Metallography was carried out on the 'Normal Coupon' (13A1) after the bend test.

The test coupon was sectioned parallel to the longitudinal axis. The section was examined in the unetched and etched conditions on stereo and metallurgical microscopes. The section displays no evidence of the presence of cracks. The section displays a quenched and tempered structure, which is normal for the material.

Typical micrographs of the etched microstructure taken near and away from the location of the bend in the section examined are presented at the end of this report.

Test for Susceptibility to Stress Corrosion Cracking

The test was carried out on the 'Bent Coupon' (13B1). The coupon was stressed in a three point bend fixture following NACE TM0177 as a guideline specification. The stress applied on the coupon was 70% of the actual yield strength of the material stated on the inspection certificate.

After down hole exposure in the stressed condition, the test coupon was subjected to visual and stereomicroscopic examination of the wide faces for any indications of stress corrosion cracking.

The test coupon displays no cracks.

General views of the wide faces of the test coupon, after down hole exposure, are presented at the end of this report.

Tensile Test

A tensile test was carried out on the 'Cylindrical Coupon' (13C1). The test results are presented and compared with the specified range and reported values obtained from the manufacturer's inspection certificate, as follows:

Parameter	Specified Range *	Reported Values *	Test Results **
Yield strength, ksi	95 - 110	103	109
Tensile strength, ksi	105 minimum	117.3	122
Elongation, %	13.5 minimum	27.0	22.0

^{*} Obtained from manufacturer's inspection certificate

The result suggests that there is no meaningful degradation of the tensile properties of the material due to exposure to the down hole environment.

^{**} Obtained from the tensile test carried out on the 'Cylindrical Coupon' after down hole exposure

22% Cr - 42% Ni Austenitic Alloy

Information Obtained from Manufacturer's Inspection Certificate

ltem	Description Seamless steel casing test sample SM-2242-110 plain end		
Material (product)			
Standard specification	SM2242-110		
Heat No.	D782709		
Heat treat- ment/processing	Solution (1110°C x 10 minutes W.Q) followed by cold drawing		
Chemical composition	Reported to be within the ranges specified for various elements		
Tensile properties	Reported to meet the specified requirements for various parameters		

Visual Examination and Photographic Documentation of Observations

- All three coupons (22A1, 22B1 and 22C1), normal, bent and cylindrical, display original bright metallic luster of the coupons after exposure to the down hole environment. There is no meaningful change in the surface features due to the exposure.
- 2. There is no evidence of cracking or pitting in any of the coupons.

Photographs displaying the condition of the coupons before and after exposure are presented at the end of the report.

Determination of Corrosion Rate

The corrosion rate was calculated after determining the weight loss in the 'Normal Coupon' (22A1), and considering down hole exposure for a period of 60 days as reported by the client. The data recorded/calculated are as follows:

Parameter	Value	
Initial weight of coupon, grams	4.2340	
Final weight of coupon, grams	4.2338	
Loss of weight of coupon, grams	0.0002	
Corrosion rates, mils per year (mpy)	0.006	

Bend Test

A bend test was carried out on the 'Normal Coupon' (22A1) following ASTM A262 Practice E as a guideline to determine whether the coupon displays any evidence of intergranular cracking in the environment of exposure. The nominal test coupon dimensions are as follows:

Length: 76.2mm, Width: 4.5mm, Thickness: 1.5mm

After the down hole exposure, the test coupon was bent through 180° around a mandrel having a diameter equal to 4 times the thickness of the test coupon.

The surfaces of the test coupon were examined on a stereomicroscope. The test coupon displays no evidence of the presence of fissures and/or cracks.

Metallography

Metallography was carried out on the 'Normal Coupon' (22A1) after the bend test.

The test coupon was sectioned parallel to the longitudinal axis. The section was examined in the unetched and etched conditions on stereo and metallurgical microscopes. The section displays no evidence of the presence of fissures and cracks. The section displays an austenitic structure, which is normal for the material.

Typical micrographs of the etched microstructure taken near and away from the location of the bend in the section examined are presented at the end of this section of the report.

Test for Susceptibility to Stress Corrosion Cracking

The test was carried out on the 'Bent Coupon' (22B1). The coupon was stressed in a three point bend fixture following NACE TM0177 as a guideline specification. The stress applied on the coupon was 70% of the actual yield strength of the material stated on the inspection certificate.

After down hole exposure in the stressed condition, the test coupon was subjected to visual and stereomicroscopic examination of the wide faces for any indications of stress corrosion cracking.

The test coupon displays no cracks.

General views of the wide faces of the test coupon, after down hole exposure, are presented at the end of this section of the report.

Tensile Test

A tensile test was carried out on the 'Cylindrical Coupon' (22C1). The test results are presented and compared with the specified range and reported values obtained from the manufacturer's inspection certificate, as follows:

Parameter	Specified Range *	Reported Values *	Test Results **
Yield strength, ksi	110 - 140	119.3	113
Tensile strength, ksi	115 minimum	123.2	116
Elongation, %	13 minimum	17	21

^{*} Obtained from manufacturer's inspection certificate

The result suggests that there is no meaningful degradation of the tensile properties of the material due to exposure to the down hole environment.

^{**} Obtained from the tensile test carried out on the 'Cylindrical Coupon' after down hole exposure

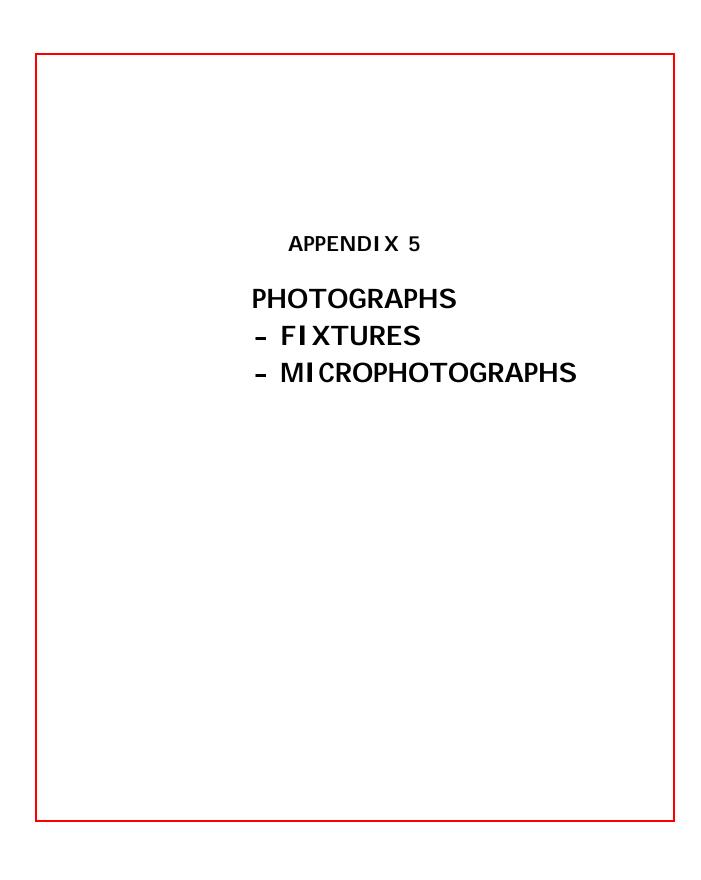




Figure 20 - General view of the bent coupons identified as 13B1, 22B1, 25B1 before loading into the bending fixtures; note all the samples display the normal metallic luster expected in the asmachined condition



Figure 21 – General view of the bent coupons after stressing in the bending fixtures; the view presented is before exposure to the down hole environment; the bending fixtures are identified as 13B1, 22B1, 25B1

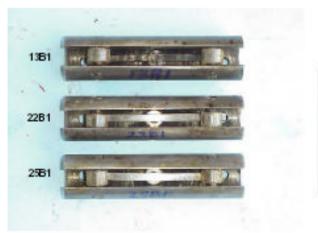


Figure 22 - General view of the bent coupons identified as 13B1, 22B1, 25B1 after exposure to down hole environment in the stressed condition; note the dull/dark color of the coupon 13B1, and bright metallic luster of the coupons 22B1 and 25B1



Figure 23 – General view of the bent coupons identified as 13B1, 22B1, 25B1 after exposure to the down hole environment in the stressed condition; the coupons were removed from the fixtures (see Figure 22); note coupon identified as 13B1 displays discoloration whereas the other two coupons display original bright metallic luster



Figure 17 – Stereomicroscopic view of the etched metallographic section from the bend location of the normal coupon identified as 25A1

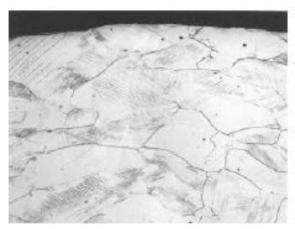


Figure 18 — Micrograph of the normal coupon identified as 25A1 taken near/at the bend location; the micrograph displays a typical austenitic structure, which is normal for the austenitic alloy; note the elongated grains towards the surface and slip bands within the grains resulting from deformation due to bending of the original annealed strip; there is no evidence of cracking or microstructural degradation due to the exposure; Original magnification — 100x



Figure 19 – General microstructure of the normal coupon identified as 25A1 away from the bend location displays a relatively equiaxed austenitic grain structure, which is representative of the original material; Original magnification – 100x



Figure 14 – Stereomicroscopic view of the etched metallographic section from the bend location of the normal coupon identified as 22A1



Figure 15 – Micrograph of the normal coupon identified as 22A1 taken near/at the bend location; the micrograph displays a typical austenitic structure, which is normal for the austenitic alloy; note the elongated grains towards the surface and slip bands within the grains resulting from deformation due to bending of the original annealed strip; there is no evidence of cracking or microstructural degradation due to the exposure; Original magnification – 100x

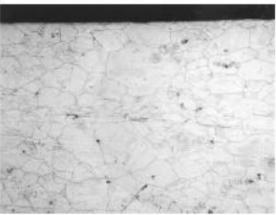


Figure 16 - General microstructure of the normal coupon identified as 22A1 away from the bend location displays a relatively equiaxed austenitic grain structure, which is representative of the original material; Original magnification - 100x

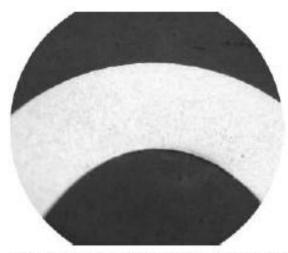


Figure 11 – Stereomicroscopic view of the etched metallographic section from the bend location of the normal coupon identified as 13A1

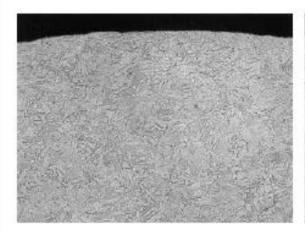


Figure 12 – Micrograph of the normal coupon identified as 13A1 taken near/at the bend location; the micrograph displays a typical quenched and tempered structure, which is normal for martensitic stainless steel; Original magnification – 100x



Figure 13 – Micrograph of the normal coupon identified as 13A1 taken away from the bend location; the micrograph displays a typical quenched and tempered structure, which is normal for martensitic stainless steel; Original magnification – 100x



Figure 7 – Stereomicroscopic view of the polished section of normal coupon identified as 13A1; the section displays no evidence of cracks



Figure 8 – Close view of the polished section of the normal coupon identified as 22A1; the section displays no evidence of cracks

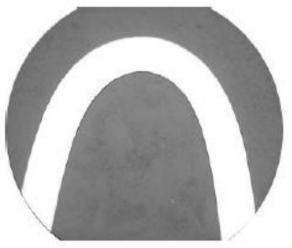


Figure 9 - Close view of the polished section of the normal coupon identified as 25A1; the section display no evidence of cracks

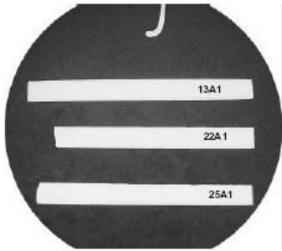


Figure 10 – General view of the polished microsections of the normal coupons identified as 13A1, 22A1, 25A1; these sections were removed away from the bend locations (un-deformed locations of the bend tested normal coupons) and subjected to microstructural examination



Figure 3 – General view of the normal coupons identified as 13A1, 22A1, 25A1 before exposure to the down hole environment; note all the samples display the normal metallic luster expected in the as-machined condition



Figure 4 – General view of the normal coupons identified as 13A1, 22A1, 25A1 after exposure to the down hole environment; note coupon identified as 13A1 displays discoloration whereas other two coupons display the original bright metallic luster



Figure 5 – General view of the normal coupons identified as 13A1, 22A1, 25A1 after the bend test following ASTM A262 Practice E as a guideline; the test coupons displayed no evidence of fissures and cracks on bending

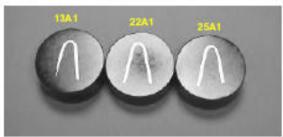


Figure 6 - Polished microsections from the bend/deformed locations of the normal coupons 13A1, 22A1 and 25A1

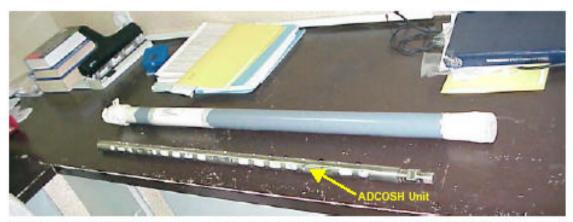


Figure 1 – General view of the coupon holder (ADCOSH Unit) with test coupons inserted and the ends plugged; this view of the coupon holder was taken before delivery of the coupons for exposure to the down hole environment; the test coupons were prepared and supplied by the client; Lonestar Technical Services carried out various procedures before insertion of the test coupon in the coupon holder (see Tables 1, 2 and 3)



Figure 2 — General view of the coupon holder (ADCOSH Unit) and the test coupons separated on eceipt from the client after exposure to the down hole environment; the test coupons are in three respective insertion fixtures; the normal and cylindrical coupons are contained inside their respective 'Teflon' holders (white colored) and the bent coupons are in the bending fixtures (dark colored, constructed from stainless steel with coupon contacting parts in glass as per guidelines of NACE TM0177)