

Element Materials Technology 14805 Yorktown Plaza Dr. Houston, TX 77040 USA P 713 692-9151 F 713 696-6307 T 888 786 7555 info.houston@element.com element.com

Report No. 131195-01

Sulfide Stress Cracking Testing of TDZ Coating Systems P.O. No. 4512835952

Eva Coronado, Ph.D. Corrosion Laboratory Manager

December 15, 2020

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INTRODUCTION

This report presents the results of sulfide stress cracking (SSC) Method A test of TDZ coating systems that you requested, and which was performed by Element Materials Technology (Element). The testing was conducted on 4140 85K yield base metal. Element received 1" OD x 70" LG bar identified as MS-004643-03. Base material MTR documents were provided by the client and they are attached to this report as Appendix A. None of the test results in Appendix A were generated by Element. The coating systems are listed below:

<u>Grade</u>	Coating System Details	Specimen ID
4140	None	545, 552
4140	A / 1.5-2 mil Total TDZ & 1 mil Armorplex / Blue	546 – 548 & 553 – 555
4140	B / 1.5-2 mil Total TDZ & 1 mil Sealer Only / Grey	549 – 551 & 556 – 558

Element was asked to perform SSC tests in accordance with NACE Standard TM0177 Method A using two different test environments. The details are listed below. Test temperature was 75°F, and test duration was 720 hours.

Environment	Specimen ID	Test Solution	Initial pH	Test Gas	Applied Stress
1	545 – 551	Solution A	2.6 - 2.8	100% H ₂ S	66% SMYS
2	552 – 558	Solution B	3.4 - 3.6	35% H ₂ S / CO ₂	66% SMYS

SSC METHOD A TEST SPECIMENS

Fourteen (14) standard Method A test specimens were machined from the sample. The specimens were prepared using a low-stress grinder and an automatic longitudinal polisher for the gauge section preparation. A 1000 grit emery cloth was used as the final polishing step. Twelve (12) of the specimens were sent to Atomic Alloys, LLC for coating with two

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TDZ coating systems. A representative picture of uncoated and coated specimens is shown in Figures 1 through 3.



Figure 1: Uncoated test specimen before exposure.



Figure 2: Test specimen with Coating A (1.5-2 mil total TDZ & 1 mil Armorplex) before exposure.

ARMORGALV® ARMORPLEX BLUE™



Figure 3: Test specimen with Coating B (1.5-2 mil total TDZ & 1 mil Sealer only) before exposure.

ARMORGALV® - AG3000 -

After machining and/or coating, the specimens were degreased with alkaline detergent cleaner, followed by a two-step solvent rinse with ethanol and acetone, and warm-air dried. Adequacy of degreasing was determined in accordance with ASTM F 21.

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LOAD APPLICATION

After degreasing, the specimens were sealed in the test cells. The specimens were stressed to 66 percent of the specified minimum yield strength (SMYS). The SMYS was 85 ksi.

The test cells were then deaerated with nitrogen for minimum 1 hour with a flow rate of 500 mL/min before introducing the test solution.

TEST CONDITION

De-ionized water and reagent grade chemicals were used for the test solution preparation. The test solutions were then deaerated with nitrogen over the weekend with a flow rate of 500 mL/min. The deaerated solution was transferred into each cell, and they were further deaerated with nitrogen for one hour at a flow rate of 500 mL/min. After deaeration, the flow was switched to the test gas at a flow rate of 500 mL/min for one hour and then at a reduced maintenance flow rate for the remainder of the test period (720 hours). The test gas was 100% H₂S (14.7 psi ppH₂S) in the case of environment 1 and 35% H₂S (5 psi ppH₂S) with balance of CO₂ in the case of environment 2.

One specimen per coating system and environment was photographed at approximately 100-hour intervals. The pictures are shown in Figures 4 through 7.

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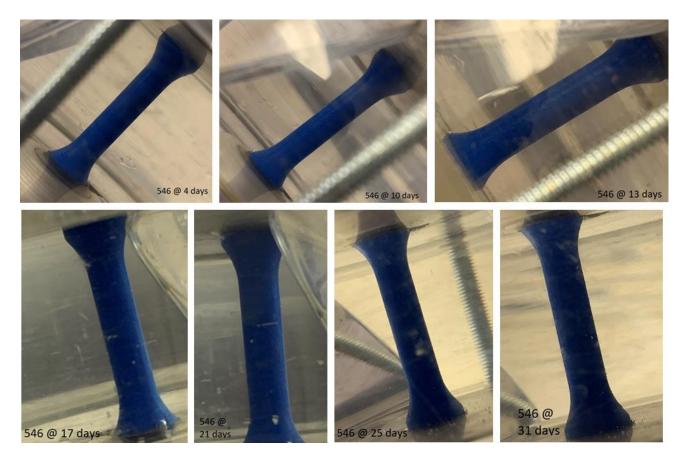


Figure 4: Test specimen 546 with Coating A (1.5-2 mil total TDZ & 1 mil Armorplex) at 100-hour exposure intervals in environment 1.

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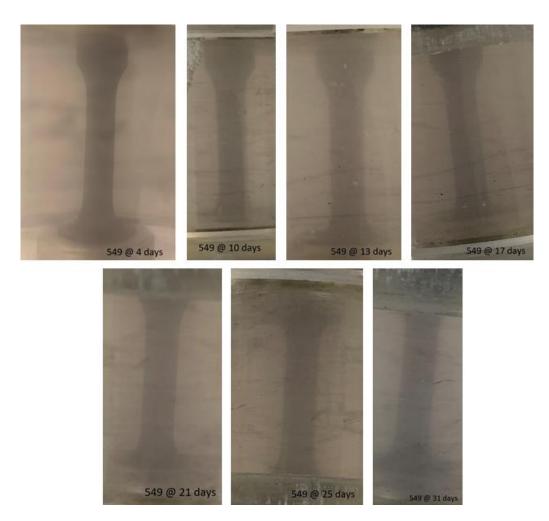


Figure 5: Test specimen 549 with Coating B (1.5-2 mil total TDZ & 1 mil Sealer only) at 100-hour exposure intervals in environment 1.

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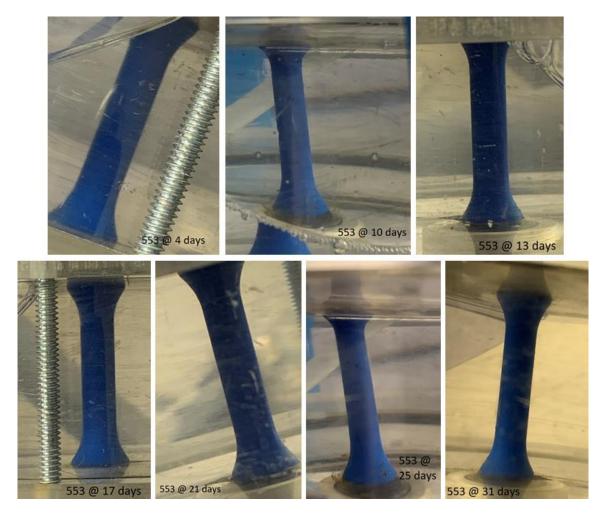


Figure 6: Test specimen 553 with Coating A (1.5-2 mil total TDZ & 1 mil Armorplex) at 100-hour exposure intervals in environment 2.

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Figure 7: Test specimen 556 with Coating B (1.5-2 mil total TDZ & 1 mil Sealer only) at 100-hour exposure intervals in environment 2.

RESULTS AND CONCLUSIONS

At the end of the test, the final pH from each cell was measured and the H₂S concentration of the last cell was measured. The cells were then purged with nitrogen at a flow rate of 500 mL/min for 30 minutes to remove the H₂S. The initial pH, final pH and H₂S concentration (cH₂S) and test results are listed in Table 1 below. The specimens were then removed from the cells, cleaned and visually evaluated. Figures 8 and 9 show all specimens after the exposure. Figures 10 through 13 show representative detail of reduced section of one specimen per coating system and environment at 20X magnification.

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Table 1: Test Results Details

Environment	Specimen ID	Initial pH	Final pH	Final cH ₂ S (mg/L)	Result
	545 (Uncoated)	2.6	3.2		Pass
	546 (Coating A)	2.6	2.6		Pass
	547 (Coating A)	2.6	2.6		Pass
1	548 (Coating A)	2.6	2.7	2812*	Pass
	549 (Coating B)	2.6	2.7		Pass
	550 (Coating B)	2.6	3.0		Pass
	551 (Coating B)	2.6	2.9		Pass
	552 (Uncoated)	3.4	3.5		Pass
	553 (Coating A)	3.4	3.4		Pass
	554 (Coating A)	3.4	3.4		Pass
2	555 (Coating A)	3.4	3.4	1045**	Pass
	556 (Coating B)	3.4	3.4		Pass
	557 (Coating B)	3.4	3.4		Pass
	558 (Coating B)	3.4	3.4		Pass

Note: * Minimum cH $_2$ S for environment 1 (100% H $_2$ S / 14.7 psi ppH $_2$ S) is 2300 mg/L.

All specimens passed the test.

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^{**} Minimum cH_2S for environment 2 (35% H_2S balance CO2 / 5 psi ppH_2S) is 805 mg/L.





Figure 8: Test specimens after the 720-hour exposure in environment 1.

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Figure 9: Test specimens after the 720-hour exposure in environment 2.

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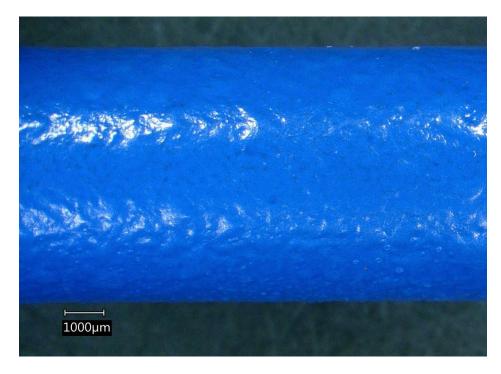


Figure 10: Test specimen 546 with Coating A (1.5-2 mil total TDZ & 1 mil Armorplex) after exposure in environment 1, at 20X magnification.

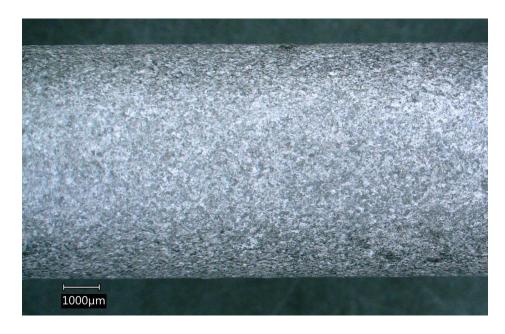


Figure 11: Test specimen 549 with Coating B (1.5-2 mil total TDZ & 1 mil Sealer only) after exposure in environment 1, at 20X magnification.

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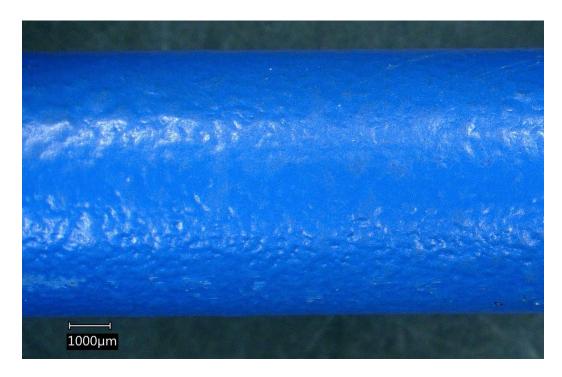


Figure 12: Test specimen 553 with Coating A (1.5-2 mil total TDZ & 1 mil Armorplex) after exposure in environment 2, at 20X magnification.

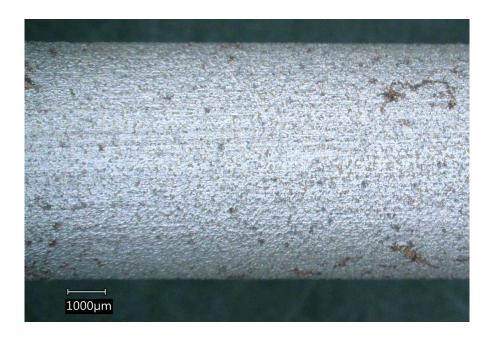


Figure 13: Test specimen 556 with Coating B (1.5-2 mil total TDZ & 1 mil Sealer only) after exposure in environment 2, at 20X magnification.

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Please call us if you have questions about this information or if we may serve you further.

Sincerely,

Eva Coronado, Ph.D.

Eva Coronedo

Corrosion Laboratory Manager

Telephone : (713) 696-6384

E-mail : eva.coronado@element.com

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Appendix A

Base material (1" OD x 70" LG bar identified as MS-004643-03) MTR documents. Note: None of the test results were generated by Element

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TURN



TECH

INCORPORATED

CERTIFICATION PACKAGE FOR

PO#

45/27/09729-120

THIS CERTIFICATION PACKAGE INCLUDES THE FOLLOWING:

	,,,,,		TOTAL INOLUDES THE FOLLOWING.
	< 9 >		CHEMICAL CERTIFICATION(S)
	< 10 >		PHYSICAL CERTIFICATION(S)
	< 11 >		HARDNESS CERTIFICATION-WITNESS
	< 12 >		FURNACE CHART(S)
	< 13 >		STRESS RELIEVE CERTIFICATION
	< 14 >		DESCRIPTIVE HEAT TREAT REPORT
	< 15 >		CERTIFICATE OF COMPLIANCE
	< 16 >		NITRIDE CERTIFICATION
	< 17 >		WELD CERTIFICATION(S)
	< 18 >		VISUAL EXAMINATION CERTIFICATION(S)
	< 19 >		VOLUMETRIC NDE (U/T) CERTIFICATION(S)
	< 20 >		SURFACE NDE CERTIFICATION(S) (M/P AND/OR L/P)
	< 21 >		SURFACE NDE PRIOR TO WELD CERTIFICATION(S)
	< 22 >		POSITIVE MATERIAL INSPECTION (P.M.I.)
	< 24 >		COATING CERTIFICATION(S)
	< 25 >		PRE-MANUFACTURING MEETING
	< 26 >		RADIOGRAPH CERTIFICATION(S)
_	< 27 >		BOND INTEGRITY/FUSION LINE (U/T) CERTIFICATION(S)
	< 28 >		OVERLAY THICKNESS (EDDY CURRENT)
	< 29 >		THIRD PARTY INSPECTION WITNESS FORM(S)
	< 30 >		SET-OUT SHEET(S)
	< 31 >		PROPRIETARY THREAD CERTIFICATION(S)
_	< 32 >		STRESS/LOAD TEST CERTIFICATION(S)
-	< 33 >		HYDRO-STATIC TEST CERTIFICATION(S)
٠_	< 34 >	-	RUBBER MOLD CERTIFICATION(S)
_	< 53 >		PLATING CERTIFICATION

INSPECTED AND PACKAGED BY:

DATE:

08 | 21 | 20 ISO Certified

32007 Industrial Park Dr. Pinehurst, TX 77362

Phone: (281) 356-1290 E-mail: turn-tech@turn-tech.com



Certificate of Compliance

Order

01875100

Customer

TURN-TECH INC

Customer PO

97438

Size

1.250 ROUND BAR

Grade

HF 4140 (217-237)

Heat Number

H11805912WX

Lot Number

481385

Specification

MS-4643-03 REV 02

Customer PT#

LG MS-004643-03

Quantity

1 PC 0'-70.50", 1 PC 0'-74"

- 1) Sigma Tube & Bar LLC hereby certifies that the material described above and on the attached Test Report(s) complies with the terms of the order contract, as presently agreed upon.
- 2) This order was processed in accordance with Sigma Tube & Bar's Quality Manual Rev F, dated 22 February 2019.
- 3) No weld repair was performed on this material.

Cody Willeford Sales Assistant

VNDR: TURN-TECH

PO# 4512769729-120

P/N: 1" OD X 70" LG

QP# N/A

SIGN: Kristen Troquille

VNDR# 10006457

CC SER# 01

MS# HT#

4643-03 REV 02 H11805912WX-481385

CTNG:

N/A



JIANGYIN XING CHENG SPECIAL STEEL WORKS CO., LTD.

NO.297, BINJIANG (E) ROAD, JIANGYIN CITY, JIANGSU PROVINCE, CHINA PC:214429

PRODUCER'S CERTIFICATE OF CHEMICAL AND PHYSICAL ANALYSIS

- EN10204 3.1

DESCRIPTION OF GOODS:HOT ROLLED AND FORGED ROUND STEEL BARS CONTRACT NO.:XBUSA18303HOU INVOICE NO.:XBUSA18303HOU-2C PO.212278
STEEL IS FREE FROM MERCURY; NO REPAIR WELDING; STEEL IS FREE FROM ANY HARMFUL RADIOACTIVE CONTAMINATION John Deere

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MAX-	0.43	0.35	1.00	0.025	0.025	0.25	0.25	1.10	0.05	0.	.25	0.050	0.0099	0.0003	99.0	0.040														Ь			
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VNDR: TURN-TECH

VNDR# 10006457

PO# 4512769729-120 P/N: 1" OD X 70" LG

CC SER# 01

QP# N/A

MS# 4643-03 REV 02

H11805912WX-481385 HT#

SIGN: Kristen Troquille

CTNG: N/A

1/38

TURN-TECH INC PO: 97438, PT# LG MS-004643-03, SP# MS-4643-03 REV 02 SO:01875100 pg 2 of 4 approved by CW 08/04/2020



JIANGYIN XING CHENG SPECIAL STEEL WORKS CO., LTD.

NO.297, BINJIANG (E) ROAD, JIANGYIN CITY, JIANGSU PROVINCE, CHINA PC:214429

PRODUCER'S CERTIFICATE OF CHEMICAL AND PHYSICAL ANALYSIS

-- EN10204 3.1

DESCRIPTION OF GOODS:HOT ROLLED AND FORGED ROUND STEEL BARS
CONTRACT NO::XBUSA18303HOU INVOICE NO::XBUSA18303HOU-2C PO:212278 STEEL IS FREE FROM MERCURY; NO REPAIR WELDING; STEEL IS FREE FROM ANY HARMFUL RADIOACTIVE CONTAMINATION John Deere

Gra	ide	В	atch No.	Hea	t No.	Size((mm)	Size	(inch)	Bundles	Pi	ieces	Weig	ht(MT)	Leng	th(Feet)	Condition	n of Deli	very .		Manufach	re Proces	S	Free from	n mercury a	nd any harmful r	adioactive
AISI4140	QT P1	M	1811670	H11805	912WX	31.	.75	1	1.25	3		190	7	.92		22	. (Q+T		E	AF+LF+V	/D+CC+I	IR.	,	ontaminati	on;NO weld repa	dr.
												Energ	y Charpy	Impact Tes	t (10×10	×55mm V N	lotch)										
Specime			Long.			Long.			Long.			Long.		Soccime													
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		ft-Ibs	%	mm	ft-Ibs	₹	mm	ft-Ibs	%	mm	ft-Ibs	%	mm	Localion	l :	ft-Ibs	%	mm	ft-Ibs	%	mm	ft-Ibs	%	mm -	ft-Ibs	%	mm
Core	min	40		1	30			1			20				min												
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actu	al	74	0.76	70	68	0.70	70	70	0.69	70	65	0.63	60	ac	tual												
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37	Actor ASTM A29-16,A322-13,A304-16,A751-14a,ASTM E381-17,E112-13,E81-6a,E10-15,EE10-15,
Remark	

QUALITY MANGER:Bai Yun



VNDR: TURN-TECH

VNDR# 10006457

PO# 4512769729-120

CC SER# 01

P/N: 1" OD X 70" LG

MS#

4643-03 REV 02

QP# N/A

HT#

H11805912WX-481385

SIGN: Kristen Troquille

CTNG: N/A

SPECIALTY HEAT TREAT, INC.

CERTIFICATE OF HEAT TREATMENT

CUST NO:

S196

SIGMA TUBE & BAR LLC

CUST PH:

281-369-5525

CUST NAME:

14315 W. HARDY RD.

CUST PO:

\$ 01875100 01 SR

CUST ADDR:

HOUSTON, TX 77060

CUST HN:

4140

7-29-20

H11805912WX

QUANTITY:

3

MATL:

DUE DATE:

WGT:

52

LOCATION: DATE RECD: NS-2 7-22-20

STICKER #:

99568

DESC:

LT# M1811670

1 1/4"OD X 1 @ 70 1/2"LG; 1 @ 80"LG; 1 TP @ 7"LG

PROCESS INSTRUCTIONS: HEAT TREAT PER MS-004643-03 REV 02

PROCESSED AS FOLLOWS IN A BATCH FURNACE:

MCS:

1 1/4"

CONTROLLED BY FURNACE INSTRUMENT

PROCESS	TIME	TEMP IN DEG F	COOLING METHOD
HARDEN	3 HOURS	1575	QUENCHED TO QUENCH TEMPERATURES
OIL QUENCHED		106-114	
TEMPER	4 HOURS	1290	A/C TO AMBIENT

HARDNESS:

CK 6" PROLONGATION (2 PLCS) = 237-237 HBW; CK TP = 237-237 HBW

CK 1 PC(s) = 228 HBW/P

MECHANICAL PROPERTIES OF A 0.503" DIA TP PER ACCU-TEST LAB #: 936235.00

CORE

TENSILE	YIELD .2% OFF	ELONG IN 2"	REDUCTION
111,200 PSI	96,400 PSI	25.4 %	63.2 %

CHARPY IMPACTS 10X10 MM CORE

DIRECTION	TEMPERATURE	FT/LBS	MILS LAT EXP	%SHEAR-%D/F
LONGITUDINAL	-20 °F	97-102-94	70-73-68	100-100-100

HT LOT#

481385

ALL MATERIALS ON THIS ORDER WERE PROCESSED IN ACCORDANCE WITH OUR QUALITY MANUAL SHT 01.2 REV 2

SPECIALTY HEAT TREAT, INC.

VNDR: TURN-TECH PO# 4512769729-120

P/N: 1" OD X 70" LG

QP# N/A

SIGN: Kristen Troquille

VNDR# 10006457

CC SER# 01

MS#

4643-03 REV 02

HT#

H11805912WX-481385

CTNG:



TURN-TECH, INC.

32007 INDUSTRIAL PARK DRIVE

PINEHURST, TEXAS 77362

Certificate of Conformance

From: Turn-Tech, Inc

Packing List No: 64189

Today's Date: 08/21/20

Shipping Date: 08/21/20

PO Number: 4512769729

Item #: 120

Part Number: 1" OD X 70" LG MS-004643-03

Material Specification: MS-4643-03 REV 02

Quality Plan: None Coating Certification: None

Quantity

Shipped <u>Unit</u> **Description** Job Number

1 EΑ 1" OD X 70" LG MS-004643-03 1" OD X 70" LG MS-004643-03

79077

VNDR: TURN-TECH

10006457 VNDR#

PO# 4512769729-120

CC SER# 01

P/N: 1" OD X 70" LG

4643-03 REV 02 MS# H11805912WX-481385

HT#

QP# N/A

SIGN: Kristen Proquille

N/A CTNG:

We hereby certify that the above mentioned parts meet the requirements of all drawings and specifications listed on the above purchase order, including material, heat treatment, plating and special procedures as applicable.

Clete Jacger

Authorized Signature Turn-Tech, Inc



Turn-Tech, Inc

32007 INDUSTRIAL PARK DRIVE PINEHURST, TX 77362

Phone: 281-356-1290 Fax: 281-356-1293

Visual Examination Certification

From: Turn-Tech, Inc

32007 INDUSTRIAL PARK DRIVE

PINEHURST, TX 77362

Report Date: 08/20/20

PO Number: 4512769729

Item #: 120

Part Number: 1" OD X 70" LG MS-004643-03

Drawing No: None

Weld Specification: None

Heat No: H11805912WX-481385

Material Specification: MS-4643-03 REV 02

Procedure: X-008060 REV 06

Quality Specification: None

Coating Specification: None

Quantity

Description

Job Number

1 Pcs.

1" OD X 70" LG MS-004643-03 1" OD X 70" LG MS-004643-03

79077

Area(s) Examined:

THIS IS A VISUAL EXAMINATION AFTER FINAL MACHINING FOR SN# 01.

VNDR: TURN-TECH

PO# 4512769729-120

VNDR# 10006457

CC SER# 01

P/N: 1" OD X 70" LG

QP# N/A

MS#

4643-03 REV 02 HT# H11805912WX-481385

SIGN: Kristen Troquille

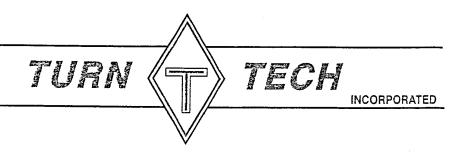
CTNG:

N/A

1 Pcs. Examined

1 Pcs. Accepted

Inspected By:



QUALIFIED INSPECTION METHOD VISUAL EXAMINATION PERSONNEL

ALL TESTING DONE IN ACCORDANCE WITH CAMERON X-008060 REV 06

ID NUMBER	NAME	QUALIFIED THRU DATES LISTED
<t>2</t>	LORENZO NUNEZ	11/19-11/20
<t>7</t>	SONNY SEBASTIAN	11/19-11/20
<t>9</t>	JORGE GARCIA	11/19-11/20
<t>12</t>	FERNANDO SANCHEZ	11/19-11/20
<t>14</t>	ROBERTO ESPINOZA	11/19-11/20
EMP# 345	CLETE JAEGER	11/19-11/20
<t>10</t>	JUAN LONGORIA	11/19-11/20
<t>15</t>	LUIS MATA	03/20-03/21
<t>17</t>	EDUARDO PENAFIEL	11/19-11/20
EMP# 573	BLAKE URBANOSKY	12/19-12/20
CERTIFIED BY:		DATE
JORGE GARCIA	Jorge García	Mar 2020

Q.A. MANAGER

VNDR: TURN-TECH PO# 4512769729-120

P/N: 1" OD X 70" LG

QP# N/A

SIGN: Kristen Troquille

VNDR# 10006457

CC SER# 01

MS# 4643-03 REV 02

H11805912WX-481385 HT#

CTNG: N/A

ISO Certified

32007 Industrial Park Dr. Pinehurst, TX 77362

Phone: (281) 356-1290 E-mail: turn-tech@turn-tech.com

Hardness Value Report Job# 79077 Turn-Tech, Inc.

	L
PART #:	1" OD X 70" LG MS-004643-03
MACH DET:	None
PROC:	X-008065 REV 09

HARDNESS VALUE

,	SHEETOF
QUANTITY:	1
MS #:	MS-4643-03 REV 02
HEAT #:	H11805912WX-481385
QP #:	None
DESC:	1" OD X 70" LG MS-004643-03
HARDNESS	PERFORMED PER BOM & DWG

217-237 HB MATERIAL HARDNESS RANGE: 4BW 710F 7-14 TESTING TYPE (A, B, OR C: AMBIENT TEMPERATURE:

EMPLOYEE #:

1) 229	11)	21)	31)	41)	51)	61)	71)	81)	91)
2)	12)	22)	32)	42)	52)	62)	72) .	82)	92)
3)	13)	23)	33)	43)	53)	63)	73)	83)	93)
.4)	14)	24)	34)	44)	54)	64)	74)	84)	94)
5)	15)	25)	35)	45)	55)	65)	75)	85)	95)
6)	16)	26)	36)	46)	56)	66)	76)	86)	96)
7)	17)	27)	37)	47)	57)	67)	77)	87)	97)
8)	18)	28)	38)	48)	58)	68)	78)	88)	98)
9)	19)	29)	39)	49)	59)	69)	79)	89)	99)
10)	20)	30)	40)	50)	60)	70)	80)	90)	100)

NOTES:

SERIAL#

REVIEWED BY:

8,6,20

VNDR: TURN-TECH PO# 4512769729-120 P/N: 1" OD X 70" LG

4643-03 REV 02 H11805912WX-481385 N/A

SIGN: Kristen Troquille

ΤL	JRN	N-T	EC	Η.	INC	.
				,		•

CONTROL CHART

JOB#

79077

	- T			PART#:					MS #:	MS-4643-03 REV 02	
CUST #:	A916 4512769729 ITEM#: 120				MACH. DET.: None				HEAT #:	H11805912WX-481385	
PO# - ITEM#:					DESC.: 1" OD X 70" LG MS-004643-03				COATING:	None	
QUANTITY:	1			QP#:	None	None			WELDING:	None	
	. Е	EMPLOYEE #:	537								
SN		SER #:									
PRINT DIM.	TOLERANCE		PART DIM.	PART DIM.	PART DIM.	PART DIM.	PART DIM.	PART DIM.	PART DIM.	PART DIM.	PART DIM
1.000	+.005		1.004								
70.00	45		70.500				10 to				
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					-				/ -		
						,					
GAGE#)							,	,	
INSPECTOR:	for	× 2.	" Fire	au_	DATE: 8	120 120			SHEET _		

VNDR: TURN-TECH PO# 4512769729-120

P/N: 1" OD X 70" LG

QP# N/A

SIGN: Kristen Troquille

VNDR# 10006457

CC SER# 01

MS# 4643-03 REV 02

HT# H11805912WX-481385

CTNG: N/A