# API Ballot id# 5531 SC5 TGOCTG & RGSSP

Work Item	2401 – Nickel Content Limits for API 5CT Sour Service Products
<b>Distribution-type</b> (Ballot, Comment-only Ballot, Recirculation (comment resolution), Re-ballot, etc.)	Comment-only (i.e. no voting) Ballot
API Document	Technical Report (TR) 5NCL [proposed identifier]
Other Impacts	SPEC 5CT
Revision Key	NA—This is a 1 <sup>st</sup> Ed. with no Track Changes for its circulation.

**Work Item Charge:** This TR will summarize the testing and provide information respective to the update in API 5CT regarding the allowable Nickel content-limit to make its Q125 grade products consistent with current knowledge regarding Sulfide Stress Cracking (SSC).

**Ballot Rationale:** Manufacturers are increasing their interest with higher Ni-content grades (e.g. P110). For example, although P110 is not a "sour grade", its use above 175°F in a sour environment is allowed by NACE MR0175. There is a potential performance impact for such applications, where cracking mechanisms (such as SSC) can cause premature failures which could then lead to leaks and possible well control situations.

For HPHT applications, the use of commercial nickel-containing grades (such as UNS G43XX for heavy section components with >80 ksi minimum yield strength) must meet the hardenability and toughness requirements that some non-nickel containing low-alloy steels (LAS) would not. Also, qualifying high-strength and high-toughness LAS with nickel contents above current limits for sour service will be a technology enabler when developing  $H_2S$  containing reservoirs. The information this TR will provide would encourage investigating the Ni-effects on cracking behavior in other pipe grades and expand the selection of products available for operating in a harsh environments.

NOTE See the ballot email notification for additional information regarding this ballot.

# Nickel Content Limits for API 5CT Sour Service Products

API TECHNICAL REPORT 5NCL FIRST EDITION, [MONTH] [YEAR]

The *Special Notes*, *Foreword*, and *Contents* will be generated by API during the page proof stage before publication.

## INTRODUCTION

API 5CT restricts the maximum nickel (Ni) content is some grades – such as Q125 – but not for others – such as P110. NACE MR0175/ISO 15156-2:2015, which provides guidelines for sulfide stress cracking (SSC) resistane and selection criteria for use of carbon/low-alloy steel (LAS) in sour service does restrict the maximum Ni-content to < 1 % for low-hardness steels in A.2.1.2. These two documents have led the industry to mainly not use higher nickel (1 % or greater) steels in sour service. A literture survey however suggests that Ni-content may not predict SSC resistance of steels and instead the underlying microstructure obtained from processing – such as heat treatment – plays an important role.

With this background, limited testing based on voluntary contributions, was done to compare AISI 4140 (low Ni-content) and AISI 4340 (high Ni-content) steels for their SSC resistance when heat treated to similar yield strength. The yield strength selected was close to the upper limit of Grade P110, which also overlaps with Grade Q125, in API 5CT. This study showed that AISI 4340 could have higher SSC resistance relative to its 4140 at similar strength levels thus again alluding to the point Ni-content in the steel may not predict SSC resistance. It is recommended that a more detailed study, with various steel grades having different Ni-content, be carried out with API assistance and the chemical composition limits of the API 5CT grades – such as Q125 – be revisisted especially with respect to restriction of max Ni-content.

Low-alloy steels have been traditionally restricted by NACE MR0175/ISO 15156 to Ni-contents below 1.0wt % on the assumption that higher nickel concentrations could negatively affect SSC resistance. Accessory manufacturers have suggested, for API 5CT Grade P110 specified components, the use of commercial Nicontaining grades – such as unified numbering system (UNS) G43XX – which have better hardenability and can be heat treated to a high-toughness.

NACE MR0175/ISO 15156-2:2015 A.2.1.2 states that: "Parent metal composition, heat treatment and hardness Carbon and low-alloy steels are acceptable at 22 HRC maximum hardness provided they contain less than 1 % mass fraction nickel". Similarly, API 5CT (9<sup>th</sup> Edition) Table E.4, indicates that Group 2 materials have max nickel restrictions (except M65); however, Group 3 Grade P110 has no nickel restriction, but for Group 4 Grade Q125, it does limit the Ni-content to 0.99 % max.

The limitation suggested by NACE MR0175/ISO 15156 was mostly based on a body of research conducted during the 1960s and 1970s. In their pioneering work, R. S. Treseder and T. M. Swanson [16] and A. K. Dunlop [19], found that LAS with more than 1.0-wt % nickel were more susceptible to SSC under similar loading and environmental conditions. Treseder and Swanson also reported that steels with a Ni-content higher than 1.0-wt% were susceptible to SSC even if hardness was kept under 235-BHN, which is equivalent to the 22-HRC limit now adopted by NACE MR0175/ISO 15156.

This limitation has created some challenges for HPHT applications. The use of commercial nickelcontaining grades – such as UNS G43XX – for heavy section components with > 80 ksi MYS, are required to meet the hardenability and toughness requirements that some non-nickel containing low-alloy steels would not meet. However, SSC resistance could be reduced for G43XX grades at temperatures permitted by NACE MR0175/ISO 15156 for OCTG grades. But, using high-nickel alloys might increase the risk of environmental cracking at elevated temperatures. Cracking mechanisms like SSC can cause premature failures leading to leaks and possible loss of well control. This occurring in a sour well may then lead to HSE issues. Although, qualifying high-strength and high-toughness LAS with Ni-contents above the current 1-wt % limit for sour service will be a technology enabler for the development of the reservoirs.

Thus, it would be beneficial to investigate the effect of nickel on cracking behavior; currently chemistry of Grade P110 in API 5CT has no limit on Ni-content while for similar higher-strength steel – such as Grade Q125 – there is a 0.99 %-max on Ni-content because a higher Ni-content in Q125, when used in sour conditions according to NACE MR0175/ISO 15156, could increase risk for cracking; accessory manufacturers are particularly challenged to design components to withstand higher loads and requiring the use of thicker P110 or Q125 grades, and due to the hardenability limitations of grades typically used (i.e. AISI 4140 mod) they consider higher Ni-content grades (i.e. AISI 4330) for such components. Although API 5CT Grades P110 or Q125 are not "sour grades", their use above 175 °F in sour service is allowed by NACE MR0175/ISO 15156; that gives a potential reliability impact for those applications. But, if nickel bearing low-alloy steel's advantages and disadvantages are defined based on current understanding of the technology, this would enable the possibility of lower cost solutions for HPHT accessories.

## 1 SCOPE

Research indicates that the SSC resistance of both Ni-free and Ni-bearing low-alloy steels is contingent on the interdependency of heat treatment, microstructure, and strength. The basis for the current 1 % Ni would need to be revised and make the API 5CT grades chemistry's consistent with current understanding of SSC. For that purpose, this report was created with the intent of generating data that could help and to summarize the testing that was done.

## 2 LITERATURE REVIEW

Regardless of the actual cracking mechanisms, SSC resistance is related primarily to the alloy's microstructure. In this regard, fully tempered martensite showed the best sour service performance, followed by lower bainite. In contrast, untempered martensite and upper bainite reduce SSC resistance significantly and are to be avoided.

## A) Waid and Stiglitz, Offshore Technology Conference, 1979 [Ref. 15]

The effect of nickel on SSC resistance is presented in Figure 1 for AISI 4130-type steels and for a medium carbon alloy steel (0.22 % C, 2.00 % Cr, 0.60 % Mo, 0.20 % V), respectively. As both figure parts (a) and (b) show, Ni-contents up to about 1.00 weight-percent have no effect on SSC resistance, but above about 1.0 % cause SSC resistance to decrease.

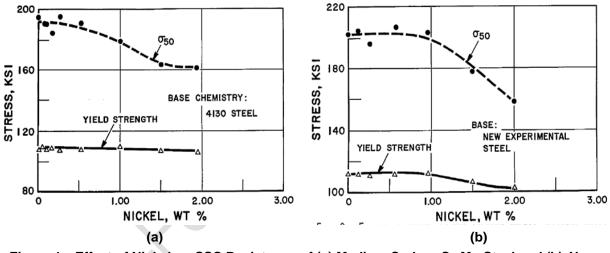


Figure 1—Effect of Nickel on SSC Resistance of (a) Medium Carbon Cr-Mo Steel and (b) New Experimental Cr-Mo-V Steel

## B) J. H. Payer, S. P. Pednekar, and W. K. Boyd [Ref. 21]

The objective of this study was to compare the performance of selected nickel containing low-alloy steels in a standard H<sub>2</sub>S environment with that of nominally nickel-free AISI 4130 steel at comparable strength and hardness levels (yield strength (YS)  $\approx$  690 MPa, HRC  $\leq$  22).

Five commercial low-alloy steels with nominal Ni-contents ranging from 0-3 % were selected. These were quenched and tempered to the desired hardness. In addition, five different tempering treatments were used for one of the steels (AISI 4330) to establish the effect of minor microstructural changes on the SSC cracking susceptibility.

At the time when this paper was published, there was conflicting evidence on whether the presence of nickel increases the susceptibility of steels to sulfide stress corrosion cracking. In some studies, comparable resistance of nickel containing and nickel-free steels was found, while in other studies, nickel containing steels at the same hardness and strength level as nickel-free steels performed much worse.

The results of the constant-load and DCB-specimen (double cantilever beam) tests show that two of the tempered, low-nickel steels used in this study closely approached the sulfide cracking resistance of AISI 4130 and ASTM A514-77 steels that contain less than 1 % Ni. Thus, given proper heat treatment, low-alloy steels containing greater than 1 % Ni can have comparable sulfide cracking resistance.

Microstructural changes brought about by minor variations in heat treatment affect the SSC susceptibility of low-nickel steels, even though these changes cannot always be resolved by conventional microscopy.

#### C) Y. Yoshino and Y. Minozaki [Ref. 18]

The SSC resistance of Ni-containing low-alloy steels was studied using laboratory and commercial heats over the range of 600 to 800 MPa yield strength (700-900 MPa tensile strength). The results were interpreted with regard to observations by metallurgical and electrochemical analyses. In the steel containing 1 % Cr and 0.5 % Mo, the SSC resistance is not affected by up to 2 % Ni.

A commercial steel with 3.7 % Ni, 1.8 % Cr, 0.4 % Mo exhibits the same  $K1_{ssc}$  as Ni-free steels. The cracking resistance begins to deteriorate when fresh martensite exceeds 5 %-vol. The deterioration is associated with intergranular fracture.

#### D) B. D. Craig, J. K. Brownlee, and T. V. Bruno [Ref. 20]

The question of whether nickel in low-alloy steels is detrimental to their resistance to SSC has been addressed extensively in the literature. Much of the confusion surrounding this question has been created by research that did not properly control important variables. Principal among these variables are strength level, hardness, chemical composition, microstructure, and heat treatment.

This investigation achieved control of these variables and was able to demonstrate that Ni-content of lowalloy steels does not have a direct effect on SSC. The SSC resistance of both Ni-free and Ni-bearing lowalloy steels depends upon the heat treatment, microstructure, and strength, which are interdependent.

#### E) TWI Group Sponsored Project [Ref. 22]

For C-Mn steel components requiring good low-temperature toughness in the weld metal, nickel containing consumables are usually utilized. ISO 15156-2:2003 required experimental demonstration of resistance to SSC for C-Mn steel welds with more than 1 % Ni in the deposit; despite work carried out in the 1980s, which has been taken on board by DNV OS-F101, this standard allows up to 2.2 % Ni.

The work undertaken comprised metallographic examination and hardness testing of the welds, determination of retained austenite, chemical analysis, plain four point bend testing in the NACE MR0175 Solution A environment and K<sub>Issc</sub> testing in that environment and in a milder sour environment.

#### F) M. Kappes, M. lannuzzi, R. Rebak, and R. Carranza [Ref. 17]

Although some research has shown that nickel might not have direct deleterious effect on SSC resistance, crack initiation and propagation mechanisms in both nickel containing and nickel-free LAS are still unclear. Despite the effort to date, more research is necessary to separate the effect of nickel from that of other variables such as alloy composition, microstructure, and loading conditions. Elucidating if LAS with Nicontents above the current 1-wt % limit are fit for sour service will be a technology enabler for the development of the oil and gas reservoirs of the future.

# 3 TESTING PLAN

#### 3.1 Materials

AISI low-alloy steel Grades 4140 and 4340 were selected for testing as part of this work item. AISI 4140 grade is a popular grade within the 41XX series chromium-molybdenum low-alloy steel grades and is widely available in tubing, bar product. Nickel is a residual element in AISI 4140 low-alloy steel and is mostly below 0.25 % mass-fraction, which would be an example of low-Ni containing low-alloy steel. AISI 4340 grade is a commonly used grade within the 43XX series chromium-molybdenum-nickel low-alloy steel grades and is primarily available as bar product. Nickel is intentionally added in AISI 4340 low-alloy steel within the range of 1.65-2.0 % mass-fraction and would constitute high-Ni containing (much over 1 % mass-fraction) low-alloy steel. AISI 4340 typically has better hardenability than its 4140.

The raw material heats used for testing were originally bar stock that were drilled and heat treated which reflects typical use of these grades for downhole thick walled tubular components. One of the steel manufacturers donated the hot rolled raw material for the study and helped in getting them drilled to the required ID. Table 1 provides the details regarding product form, size at both initial raw material condition and during heat treat of material investigated. The reduction ratio refers to hot work ratio where in the ingots were hot forged and rolled to final bar size. Table 2 provides the chemical analysis of the two heats. The hardenability (DI) of the two heats were calculated according to ASTM A255 and is provided in Table 3 which shows higher hardenability of AISI 4340 heat over its 4140 heat.

		Or	iginal Raw Mate	Raw Material during Heat Treat		
Heat ID	AISI Grade	Product Form	Size, mm (in.)	Reduction Ratio	Product Form	Size [OD x wall], mm (in.)
1	4140	Bar	228.6 (9.0)	12.3:1	Hollow bar	228.6 x 50.8 (9.0 x 2.0)
2	4340	Bar	228.6 (9.0)	6.8:1	Hollow bar	228.6 x 50.8 (9.0 x 2.0)

Table 1—Product Form, Size of Heats Sele	cted for Testing
--	------------------

#### Table 2—Chemical Composition of Heats Selected for Testing

ŀ	leat ID	% C	% Mn	% Si	% Cr	% Mo	% Ni	% Cu	% Al	% S	% P
	1	0.41	0.97	0.29	1.02	0.20	0.17	0.21	0.028	0.006	0.006
	2	0.43	0.78	0.27	0.86	0.26	1.83	0.21	0.026	0.019	0.008

#### Table 3—Hardenability of Heats Selected for Testing

Heat ID	AISI Grade	DI, mm (in.)
1	4140	165 (6.6)
2	4340	224 (8.5)

## 3.2 Heat Treatments

The heats were normalized, quenched and tempered in accordance with Table 4 to mainly aim at yield strength around 965 MPa (140 ksi). A steel distributor facilitated the heat treatment through a batch heat treatment shop. AISI 4340 steel having higher Ni-content does have a lower austenitizing temperature compared to its 4140 and 4340 having higher hardenability would be tempered at higher temperatures compared to its 4140 steel for getting similar target mechanical properties.

Heat ID	AISI Grade	Normalizing [for 5 h then air cool]	Austenitizing [for 4 h then polymer quench]	Tempering [for 5 h then air cool]	
1	4140	899 °C (1650 °F)	857 °C (1575 °F)	566 °C (1050 °F)	
2	4340	899 °C (1650 °F)	843 °C (1550 °F)	582 °C (1080 °F)	

#### Table 4—Heat Treatment Details

# 4 TEST RESULTS

## 4.1 Mechanical Properties

As-quenched hardness in Rockwell C-scale in accordance with ASTM E18 was done after austenitizing and quenching operation to provide more insight into hardenability. Table 5 provides the results for the two heats of material, and as expected the AISI 4340 heat with higher hardenability has higher as-quenched hardness.

Table 6 gives the results of tensile testing that was done after heat treatment from the ends of the drilled bar and from at least one OD diameter length away from the end. Tensile testing was done in accordance with ASTM A370 with samples taken from mid-wall location of the drilled bar. For AISI 4140 heat, not much change was seen in yield strength values close to the quenched end and away from it; while for AISI 4340 heat, the yield strength away from the quenched end was almost 4 ksi lower than near quenched end. Do note the yield strength values away from the quenched end were used for any stress calculations in corrosion testing as the samples for corrosion testing was taken away from the ends too.

		Leastion	Average Hardness, HRC				
Heat ID	AISI Grade	Location	Q1	Q2	Q3	Q4	
		OD	57.0	57.0	57.3	57.1	
1	1 4140	Mid-wall	57.3	57.1	57.3	57.5	
		ID	57.3	56.8	56.7	56.7	
		OD	59.1	58.8	58.9	58.4	
2	4340	Mid-wall	57.9	58.1	58.9	58.7	
		ID	57.5	57.7	57.8	57.9	

 Table 5—Average Four Quadrant as Quenched Hardness Test Results

#### Table 6—Tensile Test Results

Heat ID	AISI Grade	Location	Yield 0.2 % Offset, psi	Yield 0.6 % EUL, psi	UTS, psi	% El	% ROA
		Close to quench end	136,400	135,800	154,000	18.5	59.5
1	4140	One OD diameter away from quenched end	136,500	136,000	153,600	20.7	60.6
		Close to quench end	140,000	140,000	156,400	18.9	58.1
2	4340	One OD diameter away from quenched end	135,800	135,300	154,400	20.4	58.9

Charpy V-notch impact toughness testing was done in longitudinal and transverse direction at 0 °C (32 °F) in accordance with ASTM A370. This testing was done by taking samples away from quenched end and the results are shown in Table 7. The AISI 4340 heat did show lower toughness than its 4140 heat in transverse direction but both had similar toughness in longitudinal direction. It is to be noted that the AISI 4340 heat did have higher S-content than the its 4140 heat, which is likely to have led to lower Chary impact toughness of its 4340 heat in the transverse direction.

			noten impact rest results		
	AISI		Transverse	Longitudinal	
Heat ID	Grade	Test Temperature	Average Charpy value, J (ft-lbf)	Average Charpy value J (ft-lbf)	
1	4140	0 °C (32 °F)	49 (36)	85 (63)	
2	4340	0 °C (32 °F)	31 (23)	80 (59)	

#### Table 7—Charpy V-notch Impact Test Results

#### 4.2 Microstructure

Microstructure and microhardness was eavlauted on both the AISI 4140 and 4340 DCB samples as shown in Figure 2. Hardness measured using Vickers 1 Kg microhardness method on the DCB samples of both grades was similar at around 340 HV1 (~34 HRC). The microstructure analyzed using optiocal microscope for both grades was composed of tempered martensite as seen in Figure 3. No large regions of bainite or other constituents rather than martensite were observed in the metallographic surfaces analyzed.

Scanning electron microscope (SEM) analysis of the microstucutre of both grades showed that the carbides observed were fine as seen in Figure 4. Further, while average grain size seemed to be 20-30  $\mu$ m in both steels under high-magnification, AISI 4140 showed some regions with abnormal grain growth with grain size larger than 60  $\mu$ m; while such grain growth was not seen in AISI 4340 (Figure 5). Large MnS inclusions were detected in both materials (Figures 6 and 7). The density and size of such particles seemed to be larger for the AISI 4340 steel.

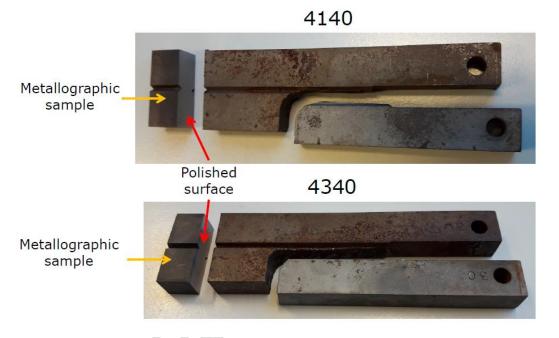


Figure 2—AISI 4140 and 4240 DCB Samples Used for Microstructure Evaluation

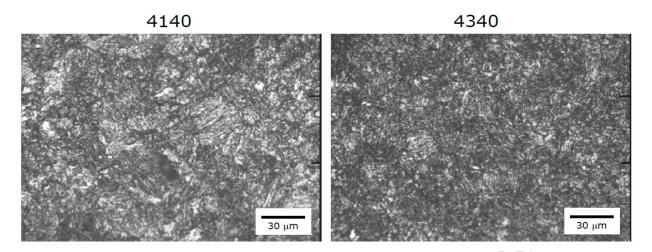


Figure 3—Optical Microscope Image of Microstructure Showing Tempered Martentise for Both AISI 4140 and 4340 Grades

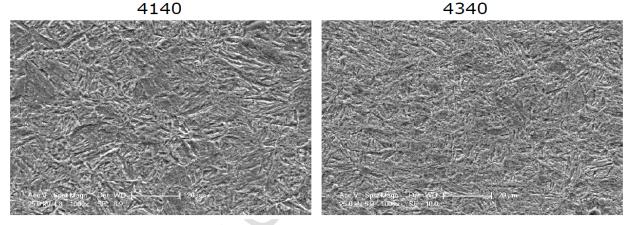


Figure 4—SEM Image of Microstructure Showing Tempered Martentise with a Fine Distribution of Carbides for Both AISI 4140 and 4340 Grades

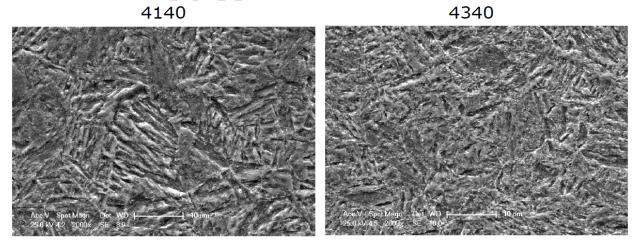


Figure 5—AISI 4140 Material Showing Regions of Large Grain Growth that is Absent in AISI 4340 Material

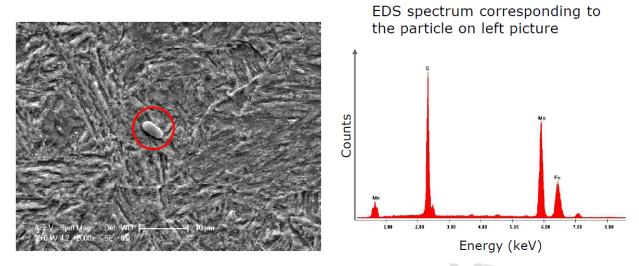


Figure 6—MnS Inclusion in AISI 4140 Material Analyzed

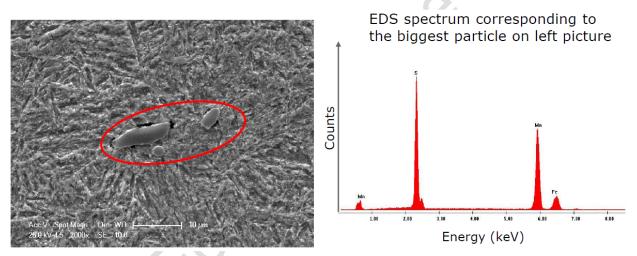


Figure 7—Larger Size and Higher Denisty of MnS Inclusions in AISI 4340 Material Analyzed

## 4.3 Corrosion Testing

The SSC testing using NACE TM0177 Method A was performed in two different test environments and using NACE TM0177 Method D in one environment. Both Method A and Method D testing was done voluntarily by two different corrosion test facilities.

Initial SSC testing of both heats was done at 0.003 bar (0.05 psi)  $H_2S$ , NACE TM0177 Method A Solution B buffered to pH of 4.0 at room temperature with specimens stressed to 90 % AYS (actual yield strength) where AYS was the room temperature yield strength of the corresponding heat in Table 6 (away from quenched end). Test duration was 30 days (720 hours) and each heat was tested using triplicate specimens. This would represent testing at the boundary limits of NACE MR0175/ISO 15156-2 Region 0 sour service. The results in Table 8 showed that both heats did pass this test so would be suitable for Region 0 sour service and the difference in Ni-content between the two heats did not change the cracking susceptibility in this environment.

Heat ID	Stress Level	Test Temperature	# of Specimens Tested	Initial pH	Final pH	Results
1	80 % AYS	80 °C (175 °F)	3	2.77	3.52 - 3.61	Failure after 14.5, 18.6 and 20.1 hours
2	80 % AYS	80 °C (175 °F)	3	2.77	3.56 - 3.66	Failure after 12.8, 14.9 and 17.3 hours
NOTE	Test cond	litions: 0.003 Bar (0.0	04 psi) H <sub>2</sub> S, 4.0 pH at	ambient te	mperature.	

#### Table 8—NACE TM0177 Method A Test Results in Solution B

Subsequently, NACE TM0177 Method A SSC testing of the two heats was done at 1 bar (14.5 psi) H<sub>2</sub>S, NACE TM0177 Solution A at 80 °C (175 °F) with specimens stressed to 80 % AYS (actual yield strength) where AYS was the room temperature yield strength of the corresponding heat in Table 6 (away from quenched end). This test condition would represent testing for use in all region sour service at or above 80 °C (175 °F). Both heats failed quickly within 24 hours of this test in accordance with Table 9. Again overall the test result did not show any difference in cracking resistance between the two grades having widely different Ni-content. However, this result is contrary to NACE MR0175/ISO 15156-2:2015 limit in Table A.3, which allows proprietary Q&T low-alloy steel with 965 MPa (140 ksi) or less maximum yield strength to be used at service temperatures of 80 °C (175 °F) for all sour service regions. Hence, more testing may be needed to further investigate this.

Table 9—NACE TM0177 Method A Test Results in Solution A

Heat ID	Stress level	Test Temperature	# of Specimens Tested	Initial pH	Final pH	Results			
1	90 % AYS	Room Temperature	3	3.95	4.12 - 4.17	No Failure after 720 hours			
2	90 % AYS	Room Temperature	3	3.95	4.09 - 4.17	No Failure after 720 hours			
NOTE	NOTE Test conditions: 1 Bar (14.5 psi) H <sub>2</sub> S at 80 °C (175 °F).								

Finally, NACE TM0177 Method D SSC testing of the two heats was done at 0.007 bar (0.1 psi) H<sub>2</sub>S, Solution B buffered to 4.5 pH at room temperature. This environment represents a point with NACE MR0175/ISO 15156-2 Region 1. The arm displacement used for the testing was 0.036 mm (0.014 in.). According to Table 10, the test results show that AISI 4340 heat had much better fracture toughness than its 4140 heat in these sour conditions with K1<sub>ssc</sub> values of the AISI 4340 heat being around 25 % higher than those for its 4140 heat. This would actually indicate the higher nickel containing grade of AISI 4340 showing better SSC resistance than lower nickel containing grade of its 4140 for similar yield strength and hardness.

Heat No.	AISI Grade	K1 <sub>ssc</sub> , MPa-m <sup>0.5</sup> (ksi-in. <sup>0.5</sup> )	Avg. K1 <sub>ssc</sub> , MPa-m <sup>0.5</sup> (ksi-in. <sup>0.5</sup> )	Std. Dev.	HRC
		21.8 (19.8)			34.65
4	4140	25.1 (22.8)		1.78	34.55
1	4140	22.9 (20.8)	23.9 (21.77)		33.72
		26.0 (23.7)			34.25
		Edge cracks			34.10
	10.10	30.3 (27.6)			34.58
2	4340	29.1 (26.5)	29.8 (27.14)	0.55	34.50
		30.0 (27.3)			34.45
NOTE	Test con		psi) H₂S, 4.5 pH at ambie	nt tempe	

#### Table 10—NACE TM0177 Method D Test Results in Solution B

# 5 COMMENTARY

AISI 4340 heat of material with higher Ni-content had higher hardenability (DI), higher as-quenched hardness than its 4140 heat of material. The AISI 4340 heat was tempered at higher temperature than its 4140 though the yield strength after heat treatment for both grades was similar. Higher hardenability in an alloy system can lead to higher as-quenched hardness and providing similar yield strength after tempering at higher temperatures. The S-content in AISI 4340 heat was 0.019 % while that in its 4140 was 0.005 % which would have resulted in relatively lower transverse Charpy impact values in the 4340 heat. Microstructure of both grades after quench and temper heat treatment is mainly tempered martensite. SEM analysis of microstructure showed some regions of high-grain growth in AISI 4140 heat which was not present in its 4340 heat. However due to higher S-content in the AISI 4340 heat, it showed more presence of MnS inclusions which also would have played a role in lower transverse Charpy impact toughness for the 4340 heat.

NACE Method A testing in Region 0 at room temperature with 90 % AYS showed that both heats passed and were compatible with NACE MR0175/ISO 15156-2 for use of steels with 140 ksi MY or lower in Region 0 conditions. NACE Method A testing in Solution A at 80 °C (175 °F) with 1 bar H<sub>2</sub>S at 80 % AYS showed rapid failures for both heats bit contrary to Table A.3 of NACE MR0175/ISO 15156-2:2015 which allows use of proprietary Q&T grades with 965 MPa (140 ksi) or less maximum yield. NACE TM0177 Method D testing done at 0.007 bar (0.1 psi) H<sub>2</sub>S, Solution B buffered to 4.5 pH at room temperature showed that AISI 4340 heat had 27.14 ksi-in.<sup>0.5</sup> average K1<sub>ssc</sub> value which was almost 25 % higher than that of its 4140 heat at 21.77 ksi-in.<sup>0.5</sup> average K1<sub>ssc</sub>. Overall while NACE Method A showed both the AISI 4340 and 4140 heats performed similarly in the two tests; NACE Method D did show that its 4340 heat had better K1ssc value than its 4140 heat with similar yield strength. This demonstrates that the Ni-content percentage is not a good indicator SSC resistance for low-alloy steels. Other published work [Ref. 14] also shows that AISI 4340 grade heat treated to yield strength of around 140 ksi had better SSC resistance than its 4140 grade heat treated to similar yield strength. This would suggest that some of the API 5CT grades which have max-% Ni-limit – such as for API 5CT Grade Q125 – which has 0.99 %-max nickel requirements should be revisited. A more detailed study that can evaluate various steels with differing Ni-content, yield strength ranges would help in providing a comprehensive evaluation and help in providing better guidance on chemistry ranges for the different API 5CT grades.

## 6 CONCLUSIONS

- (1) Both AISI 4340 and 4140 heat of material heat treated via quench and temper to similar yield strength of around 135 ksi showed SSC resistance when tested in NACE MR0175/ISO 15156-2 Region 0 limits at room temperature via NACE TM0177 Method A.
- (2) The two heats failed when tested in NACE TM0177 Solution A with 1 bar H<sub>2</sub>S at 80 °C (175 °F) via NACE TM0177 Method A.
- (3) AISI 4340 heat of material showed almost 25 % higher K1<sub>ssc</sub> when tested in mild sour conditions of 0.007 bar (0.1 psi) H<sub>2</sub>S, NACE TM0177 Solution B buffered to 4.5 pH at room temperature.
- (4) This study, along with other literature, suggests simple use of % nickel in low-alloy steel is not recommended for use as predictor of SSC resistance and instead a more holistic approach with emphasis on microstructure resulting from the processing be used.
- (5) Based on this study, it would be recommended that 0.99 % max Ni-content for API 5CT Grade Q125 Type 1 be revisited. Additionally, a more detailed study is recommended to look at chemical composition limits of other API 5CT grades especially with regards to maximum Ni-content.

#### **BIBLIOGRAPHY**

- [1] API SPECIFICATION 5CT, Casing and Tubing
- [2] API SPECIFICATION 5CT, Casing and Tubing, 9th Edition
- [3] API TECHNICAL REPORT 5C3, Equations and Calculations for Casing, Tubing, and Line Pipe Used as Casing or Tubing; and Performance Properties for Casing and Tubing, 1st Edition (2008)
- [4] ASTM A255, Standard Test Methods for Determining Hardenability of Steel
- [5] ASTM A370, Standard Test Methods and Definitions for Mechanical Testing of Steel Products
- [6] ASTM A514-77, Standard Specification for High-Yield- Strength, Quenched And Tempered Alloy Steel Plate, Suitable For Welding
- [7] ASTM E18, Standard Test Methods for Rockwell Hardness of Metallic Materials
- [8] DNV OS–F101, Submarine Pipeline Systems
- [9] ISO 15156-2:2003, Petroleum and natural gas industries Materials for use in H2S-containing environments in oil and gas production Part 2: Cracking-resistant carbon and low alloy steels, and the use of cast irons
- [10] NACE MR0175/ISO 15156, Petroleum and natural gas industries Materials for use in H2Scontaining environments in oil and gas production
- [11] NACE MR0175/ISO 15156-2:2015, Petroleum and natural gas industries Materials for use in H2S-containing environments in oil and gas production — Part 2: Cracking-resistant carbon and low alloy steels, and the use of cast irons
- [12] NACE TM0177, Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H<sub>2</sub>S
- [13] NACE TM0177-2016-SG, Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H<sub>2</sub>S
- [14] NACE Paper #12900, Sulfide Stress Cracking Resistance of High Strength, Low Alloy Steels With Varying Nickel Content, K. Krishnan, NACE International Corrosion Conference, March 2019
- [15] OTC 3509-MS, The Development of High Strength Casing Steels with Improved Hydrogen Sulfide Cracking Resistance for Sour Service
- [16] Corrosion, Factors in Sulfide Corrosion Cracking of High Strength Steels, Vol. 24, No. 2, p. 31-37, February 1968
- [17] Corrosion, Sulfide Stress Cracking of Nickel-Containing Low-Alloy Steels, Vol. 32, No. 3-4, p. 101-128, September 2014
- [18] Corrosion, Sulfide Stress Cracking Resistance of Low-Alloy Nickel Steels, Vol. 42, No. 4, p. 222-233, April 1986
- [19] Corrosion, Stress Corrosion Cracking of Low Strength, Low Alloy Nickel Steels in Sulfide Environments, Vol. 34, No. 3, p. 88-96, March 1978
- [20] Corrosion, The Role of Nickel in the Sulfide Stress Cracking of Low-Alloy Steels, Vol. 46, No. 2, p. 142-146, February 1990
- [21] Metallurgical Transactions A, Sulfide Stress Cracking Susceptibility of Nickel Containing Steels, 17, 1601-1610, September 1986
- [22] TWI Group Sponsored Project (GSP), Raising the Acceptance Level for Nickel in C-Mn steel Welds for Sour Service, Ref: 16721/10/13, March 2013