



# Performance of High Strength UNS N08830 in Hydrogen Stress Crack Testing to Simulate Conditions Arising from Subsea Cathodic Protection

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## ABSTRACT

The study of hydrogen stress cracking of various Nickel based alloys has recently been reported by others and is a subject of increasing interest. This paper describes results for different test methods, including Slow Strain Rate Testing (SSRT), Incremental Step Loading (ISL), and Constant Load Verification (CLV).

A new Ni-Cr-Mo-Fe alloy UNS N08830 alloy was recently evaluated for resistance to Hydrogen Induced Stress Cracking (HISC), simulating conditions arising during cathodic protection in a subsea environment.

The unique set of test methods and conditions included SSRT and ISL, all using pre-charged specimens with ongoing continuous charging during testing. Test specimens utilized different geometries, including smoothed and notched, along with round and rectangular cross sections.

The paper draws key conclusions based on comparisons of test methods, and also compares UNS N08830 alloy results to other high strength CRA's used in Oil and Gas subsea production equipment.

Key words: Oil & Gas, Hydrogen Cracking, HISC, Subsea, CRA, SSRT, Cathodic Protection

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## INTRODUCTION

Cases of Hydrogen Embrittlement affecting subsea equipment and components have been well documented for some time.<sup>1,2,3</sup> The source of embrittlement investigated in this study is theorized to emanate from the evolution of hydrogen gas generated by cathodic protection systems in seawater, with several factors possibly contributing to a material's susceptibility.<sup>4</sup>

A joint industrial project (JIP) was formed in 2005 out of Norway to investigate the susceptibility to Hydrogen Induced Stress Cracking (HISC) of duplex and martensitic stainless steels, which resulted in a recommended practice issued in 2008.<sup>5,6</sup> More recently, Ni based alloys have been investigated after some field experiences demonstrated susceptibility to HISC.<sup>7</sup> NTNU<sup>(1)</sup> in Norway performed HISC testing in 2014-16 on various austenitic alloys for comparative purposes. To this end, Incremental Step Load (ISL) testing was performed on precipitation hardened nickel allovs along with strain hardened austenitic allovs. One of the allovs included in the study was UNS N08830, a recently introduced new grade suitable for drilling tools for severe oil & gas downhole environments. The nominal chemical composition of the alloy is listed in Table 1.

The new alloy can achieve high yield strength approaching 1170 MPa (170 ksi) through strain hardening, making it a candidate material for various components in tools used for drilling, completions and subsea applications. The alloy retains significant strength at elevated temperatures, giving it advantages over leaner austenitic alloys and super-duplex stainless steels, while offering economic advantages as compared to higher cost Precipitation Hardened (PH)nickel alloys.<sup>8</sup>

## Table 1

Cr	Ni	Мо	Со	Cu	Mn	w	Si	С	N	Fe
20- 24	29- 34	4.5- 6.5	0.5- 4.5	0.5- 2.0	3.0- 6.0	0.2- 1.8	1.0	0.015	0.2- 0.55	Bal.
Maxim	Maximum wt.% unless range is indicated									

# Compositional Range of UNS N08830 (Wt. %)

UNS N08830 is a single phase, non-precipitation hardenable Ni-Fe-Cr-Mo-N super-austenitic alloy. The alloy maintains good pitting and crevice corrosion resistance in chloride environments, while achieving high strength, excellent toughness and wear resistance in the strain hardened condition. This solid solution strengthened alloy has additions of Cu, Co, Mn and N resulting in improved microstructural stability, allowing for better manufacturability of thick sections up to 254 mm (10 in) diameter compared to other super-austenitic alloys. The relative phase stability of UNS N08830 is illustrated Figure 1 where the TTT diagram of the sigma phase is compared to UNS N08367.

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<sup>&</sup>lt;sup>(1)</sup> Norwegian University of Science and Technology (NTNU), Trondheim, Norway (www.ntnu.no)



Fig 1: Time-Temperature-Transformation (TTT) curve for sigma phase.

The new alloy compares favorably in terms of pitting and crevice corrosion resistance to conventional high strength Ni alloys 718 and 925 as shown in Figure 2, while demonstrating good general corrosion resistance as detailed in Table 2.<sup>9,10</sup>



Fig 2: Comparison of common nickel alloys to ASTM G48C pitting corrosion test at 50 °C a) UNS 07718 Aged. b) UNS 09925 Aged. c) UNS 08830 Cold Worked.

## Corrosion

The alloy has a PRENw of about 46 and Critical Pitting Temperature,  $CPT_{min}$ , in acidified ferric chloride per ASTM<sup>(2)</sup> G48C of 75°C (167°F) and Critical Crevice Temperature,  $CCT_{min}$ , per ASTM G48D of 35°C (95°F).

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<sup>&</sup>lt;sup>(2)</sup> ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

## Table 2

## Intergranular Attack Test Results. Mass Loss in mpy (mm/y).

Alloy	N08367	N10276	N06022	N06625	N08830
G28 Practice A*	15.35	262.2	40.1	14.4	10.8
ferric sulfate-	(0.39)	(6.66)	(1.02)	(0.37)	(0.27)
50% sulfuric acid	. ,	. ,	. ,	. ,	. ,

\*Equivalent to ASTM A262 practice B

# Stress Corrosion Cracking – NACE<sup>(3)</sup> MR0175/ ISO<sup>(4)</sup> 15156<sup>11</sup>

Based on the composition, N08830 falls within the chemistry limits of NACE MR0175 / ISO 15156-3 for Type 4c solid solution and strain hardened nickel alloys. The alloys confined within the 4c group are generally limited to 1034 MPa (150 ksi) yield strength and 40 HRC hardness maximum, for use in sour service.

Sour service testing was performed on N08830 alloy to evaluate yield strength levels greater than 1034 MPa (150 ksi). Testing was performed using C-rings per TM0177-2016<sup>12</sup> at the following conditions as defined in MR0175 / ISO15156-03

- Level II (sulfide stress cracking (SSC))
- Level III (galvanic hydrogen stress cracking)
- Level V (stress corrosion cracking (SCC) 150 °C, 0.003 MPa p<sub>H2S</sub>, 101,000 mg/L Cl<sup>-</sup>)

Additional testing was performed using modified Level V conditions at a higher temperature of 175°C (347°F). All tests were conducted with nine samples representing three heats for 90 days of exposure.

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<sup>&</sup>lt;sup>(4)</sup> International Organization for Standardization (ISO), Chemin de Blandonnet 8, CP401, 1214 Vernier, Geneva Switzerland.

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NACE Level	Test Period	pН	cı-	Temp.	ppH2S	ррСО2	ppH2O	Comments	Results: 3 hts x 3 Tests		Results: 3 hts x 3 Tests		Yield Strength
	Days	Start - Fin	mg/L	Deg F	psia	psia	psia		T1	T2	T3		ksi
		2.72	2.72 - 33,700 75			<1		Normal Test	Р	Р	P	1	159
II 90	90	2.72-		75	14.7		<1	Conditions	P	Р	P	2	167
		2.72						Conditions	P	Р	P	3	167
		2.72 -		75	14.7		<1	Calv. Couple	Р	P	P	1	159
III	90		33,700					to c-steel	P	Р	P	2	167
	3.00						(normal)	Ρ	Р	P	3	167	
		90 3.65 101,000 302 100 200 70 N						Normal Test	Р	Ρ	Р	1	159
v	90		Normal Test	Р	Р	F	2	167					
								Conditions	Р	F	F	3	167
V						Temp. Higher	P	P	F	1	159		
High	90	3.6	101,000	347	100	200	70	That Normal	P	F	F	2	167
Temp.								Level V	P	P	P	3	167

Table 3: N08830 Sour Service Test Results at High Strength (>150 ksi YS)

The results column in Table 3 shows the pass (P) and fail (F) for 3 different heats with each heat producing 3 different test specimens. The results showed good SCC resistance for the >1034 MPa (150 ksi) higher strength material, at level II and III. Note that condition II and III were tested for duration of 90 days instead of 30 days prescribed by MT0177-2016. At test condition V, cracking susceptibility was evident by the mixed pass/fail results, particularly above 1103 MPa (160 ksi) yield strength.

## **EXPERIMENTAL PROCEDURES**

#### Hydrogen Induced Stress Cracking (HISC).

The HISC resistance of UNS N803380 was evaluated using several testing methods which consisted of two different pre-charging methods, two test environments, as well as two different test specimen geometries. For simplicity and future reference, the test conditions are separated into method I and method II and are listed in Table 4 below.

The test conditions listed in method I were selected so that a direct comparison of UNS N08830 to previously tested high strength materials using identical testing procedures and environment for commonly used Ni-based alloys such as grades API<sup>(5)</sup>-6ACRA<sup>13</sup> 718 and 725, cold worked 625 and fine grained super duplex stainless steel (SDSS) alloy. This comparison will be discussed in greater details in the following section.

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<sup>&</sup>lt;sup>(5)</sup> American Petroleum Institute, (Washington DC: API)

Method II introduces more severe test conditions than method I by decreasing the cathodic protection (CP) potential, lowering the test temperature, using rectangular tensile specimens to promote more plain strain conditions during the test and by introducing machined notches in selected test samples.

	Specimen Geometry	Test Environment	Pre-Charging	Test Method
	Round	Solution: 3.5% NaCl	Solution: 2:1 Glycerol and 85% H <sub>3</sub> PO <sub>4</sub>	
Method	Smooth	Temperature: 20°C	Temperature: 120°C	101
I	Tensile	Potential: -1050mV	Potential: -1050mV Ag/AgCI reference	13L
		vs. Ag/AgCl	Duration: 5 days	
	Rectangular	Solution: 3.5% NaCl	Solution: 3.5 % NaCl	
Method	smooth and	Temperature: 5°C	Temperature: 80°C	
II	notched	Potential: -1100mV	Potential: -1100mV <sub>Ag/AgCl</sub> reference	SORI
	tensile	vs. Ag/AgCl	Duration: 7 days	CLV

 Table 4

 Method I and II Test Conditions for the HISC Evaluation of UNS 08830

## Material Method I.

The N08830 material used for testing per Method I was machined from forged bars from two different heats, with longitudinal yield strength of 1270 MPa (184 ksi) and 1178 MPa (171 ksi) respectively. Four samples from each heat were prepared and tested. The dimensions of the tensile specimens used for the ISL testing where machined to gage diameter of 3.0 mm and gage length of 24.5 mm. After hydrogen pre-charging per Table 4, both pre-charged and a single reference samples from each heat were placed in a Cortest<sup>(6)</sup> proof ring as shown in Figure 3. A reference sample that was not pre-charged was tested in air only and was subjected to the same loading schedule as the pre-charged samples.

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<sup>&</sup>lt;sup>(6)</sup> Cortest is a product of Cortest Inc. 38322 Apollo Pkwy, Willoughby, OH 44094.

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# Figure 3. Cortest ring setup for Method I testing showing the configuration of the precharged sample in the test environment.

The ISL specimens per method I were initially loaded to 70% of actual yield strength and held there at a constant load for 6 days while adjusted for cold creep. After the 6-day holding period the load on the samples was increased by 4% of actual strength every 24 hours until the specimen fractured.

# Material Method II.

The material that was used for the HISC screening per method II was obtained from a forged bar that was cold worked to a yield strength of 1236 MPa (179 ksi). Tensile test samples were machined from the bar longitudinal direction with a rectangular geometry of the gage section to promote more triaxial state of stress during straining. Both smooth and notched samples were prepared for the SSRT, ISL and the CLV testing. The cross-sectional dimensions of the test specimens measured 4.5 x 3.5 mm (0.177 x 0.138 in) (for the smooth samples and 5.75 x 3.5 mm (0.226 x 0.138 in) for the notched samples. The depth of the notch was machined to 1.25 mm (0.05 in) with an estimated stress intensity factor K<sub>t</sub> of 6 and was produced on the 5.75 mm (0.226 in) side of the gage section which resulted in the same effective cross section as the smooth samples.

# Slow Strain Rate Testing (SSRT)

Slow Strain Rate Testing was performed using a Cormet C-176 SSRT<sup>(7)</sup> machine, which is shown in Figure 4. Testing was first carried out in air on un-charged smooth and notched rectangular samples to establish a baseline to which the pre-charged samples could be compared.

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# Figure 4: Shows a notched SSRT hydrogen charged sample. The Ag/AgCl reference electrode is seen in the upper left, and the Pt-counter electrode on the right.

Following the testing of the reference samples, two notched and two smooth hydrogen precharged samples were then tested in SSRT mode while emerged in a 3.5% NaCl solution detailed in Table 4 per method II. The CP was maintained by a potentiostat and to simulate the environment at the seafloor, the testing was performed in a refrigerated room at  $4^{\circ}$ C (39°F).

The strain rate of the test was set at  $10^{-6} \text{ s}^{-1}$  and after the samples fractured, the specimens were rinsed in distilled water, ethanol and dried. To minimize hydrogen diffusion out of the spent samples, they were stored at  $-20^{\circ}\text{C}$  (-4°F), until measurements of the atomic hydrogen could be performed.

# Incremental Step Loading Test (ISL) and Constant Load Verification (CLV)

Both the ISL and the CLV tests per method II were performed using the same Cormet C-176 SSRT machine. The pre-loading levels for both the ISL and the CLV tests were determined using the fracture stress (FS) which is the average UTS based on the tensile results from the pre-charged SSRT samples.

The ISL evaluation was carried out on one smooth and one notched hydrogen pre-charged test samples. The test samples were tested to failure and compared to the sample tested in air. The loading schedule for both the smooth and notched rectangular specimens consisted of pre-loading to 90% of FS and holding for 48 hours. After the initial 48 hours hold period, the load was increased by 2% of the FS every 24 hours until the samples fractured.

Note that the ISL test per method I was performed by pre-loading the specimen to 70% of the actual yield strength and held for 7 days. After the initial 7 day holding period, the load was increased in increments of 4% of the actual yield stress every 24 hours until the sample fractured.

To help further assess if hydrogen diffusion can cause longer-term HISC effects, CLV testing was performed on a pre-charged rectangular smooth and a notched sample in test environment per method II. This was done by applying a constant load corresponding to 90% and 95% of fracture

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strength as determined by the previous SSRT results. One smooth and one notched sample were tested at a constant load that was adjusted for cold creep for a period of 30 days or until failure which ever came first.

#### **RA-Values and calculating RA-Ratios**

The percentage reduction in cross-sectional area (%RA) was determined after fracture for all test methods. Comparing %RA results for samples tested in air versus hydrogen charged samples enabled calculating the Reduction of Area Ratios (RAR) as follows:

$$RAR = (RA_{hyd} / RA_{air}) \times 100$$
(1)

#### Analysis of hydrogen content

Hydrogen measuring was performed on the spent samples at the SINTEF<sup>(8)</sup> corrosion laboratory by melt extraction. Gases released during melting were passed through a thermal conductivity measurement cell to allow for determining the hydrogen content of the sample. The hydrogen content of the samples was measured within an accuracy of 0.001 ppm wt%.

## **RESULTS AND DISCUSSION**

## SSRT results

Two smooth and two notched pre-charged samples were tested at a slow strain-rate. The resulting mechanical properties from the SSRT of the rectangular specimens are summarized in Table 5. The ductility of the notched samples was approximately half of that of smooth for both air and hydrogen charged samples. This would be expected based on the effect of the notch providing a pre-crack. However, average elongation ratios were similar, measuring 0.85 and 0.75 for smooth and notched specimens respectively. The RAR calculation for the smooth sample was found to be 55.2% and the average RAR for the notched sample was 67.5%

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<sup>&</sup>lt;sup>(8)</sup> SINTEF, www.sintef.no, P.O. Box 4760 Torgarden, NO-7465 Trondheim, Norway.

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#### Table 5

	SSRT	Smooth	SSRT Notched			
	Air	Hydrogen	Air	Hydrogen		
YS: Mpa (ksi)	1230 (178)	1195 (173)	1301 (189)	1265 (183)		
UTS: Mpa (ksi)	1297 (188)	1260 (183)	1330 (193)	1271 (184)		
Elongation (%)	13.7 11.6		7.5	5.6		
Ratio El <sub>H2</sub> /El <sub>Air</sub>	0.8	85	0.	75		

## SSRT at Cross Head Speed of 1x10<sup>-6</sup> s<sup>-1</sup>. Comparison of Rectangular Smooth Versus Rectangular Notched Samples.

The mean fracture stress (FS) of the hydrogen charged samples were determined to be 1260 MPa (183 ksi) and 1271 MPa (184 ksi) for the smooth and notched samples respectively.

## Incremental Step Loading- Rectangular specimens per test method II

One smooth and one notched sample was evaluated using ISL test technique. The results of the ISL testing for the rectangular samples tested per method II are summarized in Table 6. The total duration of the smooth sample test was 247.4 hours or 10.31 days and failed after reaching 100% of the mean FS that was established by the SSRT. The fracture occurred after increasing the final load to 1261 MPa (183 ksi) and the duration of the final step was 9 hours and 10 minutes.

#### Table 6

#### ISL Results – Rectangular Sample Results

	Smooth Sample	Notch Sample
% of SSRT Fracture Stess	100	98
Time to Failure TTF (hrs)	247.4	202.3
Duration of Final Step	9 hrs, 18 min.	6 hrs, 10 min.

The notched sample failed after total time of 202.3 hours or 8.44 days at a final load increase of 1246 MPa (181 ksi) which represents 98% of the mean FS from the SSRT of the notched samples. The duration of the final load step before fracture measured 6 hours and 10 minutes.

#### **SEM Analysis**

SEM fractographs were taken of the fracture surfaces and the sides of the fractured ISL specimens to assess the nature of the fracture and gage the amount of secondary cracking. Figure 5 shows the secondary crack formation that resulting in ISL tests performed per method I and Figure 6 shows the fracture surface and secondary cracking resulting in the failed ISL specimens tested by method II.

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The fracture surfaces of both the round and rectangular smooth N08830 samples showed indications of mostly ductile fracture but also contained brittle fracture morphology around the periphery of the fracture surface. The brittle zone was relatively small with the ductile-brittle transition measuring only few micrometers in from the outer surface of the samples.

The notched rectangular samples of alloy N08830 also showed mostly ductile fracture morphology but unlike the smooth rectangular sample, the only brittle fracture morphology was only observed in the area ahead of the notched surface. The amount of secondary cracking for the round samples is shown in Figure 5 and is compared to alloys API-6ACRA 718 and 725, cold worked alloy 625 and 25Cr super duplex stainless alloy. Figures 6b and 6e show the secondary cracking for the rectangular samples. The round smooth samples made from alloy N08830 contains relatively small amount of secondary cracking and the smooth rectangular sample had no observable secondary cracking. However, few microcracks were observed on the notched rectangular sample shown in Figure 6b.



Fig 5: ISL round-smooth fracture samples comparing alloys' secondary cracking.



Fig 6: ISL fracture surfaces for rectangular test specimens of N08830 alloy. Top a and b) fracture surface and secodary cracks of the smooth sample. Bottom c and d) fracture surface of the notched sample showing mostry ductile fracture with no secodndary cracking.

## **RA Ratios for Incremental Step Loading (ISL)**

The ISL results for N08830 smooth round tensile bars per test method I gave an average RAR value of 60.0% for the material with yield strength of 1270 MPa (184 ksi) with the fracture occurring at 109.5% of the YS. The material with yield strength of 1178 MPa (171 ksi) fractured at 110% of YS which resulted in average RAR value of 57.9%.

These results were compared to various PH nickel alloys namely alloys 718, 725 and cold worked alloy 625 that had been previously tested using identical techniques outlined in table 4 under method I. This comparison is shown in Figure 7 were the RAR is plotted against the yield strength of the alloys. For comparison, the RAR values of UNS N08830, API-6ACRA grades 718, 725, and similar Ni PH alloys were plotted (red squares) against their yield strength in Figure 7. An additional comparison is provided in terms of SEM photo-micrographs (Figure 5) showing the extent of secondary cracking on the tensile gage section of various alloys tested by method I.

Plotting the ISL results for both round and rectangular testing using the ISL method gives a comparison for the degee of HISC susceptability, and also enables a comparison of test geometry effects (specific to N08830), for round versus rectangular, and smooth versus notched. For this study, the RAR for Ni PH alloys was 18-28%, or a drop in excess of 70% of ductility due to HISC. N08830 smooth sample dropped up to 40% for both round and retangular geometries. The notched geometry was seen to drop an additional 25% versus round specimens.

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Fig 7: Comparison of %RA ratios versus yield strength for various alloys tested to method I (red squares). The rectangular samples were tested to method II (blue squares).

#### **Constant Load Verification (CVL) Test Results**

To better understand the long-term effects of hydrogen on alloy N08830, both smooth and notched rectangular pre-charged specimens were loaded to 90% of fracture strength. No cracking was observed after 30 days hold period.

However, at a pre-load of 95% of FS, the notched samples broke about half way through the 30 days test period, with no substantial secondary cracking. The smooth rectangular sample did not crack during the 30 days period while pre-loaded to 95% of FS.

#### Hydrogen Analyses

The hydrogen concentrations of the N08830 charged samples are listed in Table 7. The notched SSRT samples showed a slight increase hydrogen concentration compared to the smooth SSRT and ISL tested samples, but of the same order of magnitude. These results were obtained by melt extraction, and therefore do not pinpoint an exact location in the cross section but indicate that the test methods used in this study were effective in charging the samples with hydrogen.

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#### Table 7.

# Measured hydrogen content of the fractured charged rectangular samples for N08830 alloy.

Test Samples	Smooth SSRT Sample 1	Smooth SSRT Sample 2	Smooth ISL	Sm ooth CLV	Notched SSRT Sample 1	Notched SSRT Sample 2	Notched ISL
ppm wt.	14.22	14.41	15.58	23.58	25.59	24.85	15.85

#### CONCLUSIONS

UNS N08830 is a solid solution, single phase, superaustenitic alloy with a unique combination of general and local corrosion resistant properties, while maintaining high strength in the strain hardened condition. Based on ISL results the alloy shows resistance to HISC as compared to Nibase PH API-6ACRA alloys 718 and 725 as well as Ni-base cold worked alloy 625, and duplex stainless steel.

Hydrogen pre-charging the test specimens before environmentally controlled load testing was effective in differentiating resistance to HISC for different alloys. Precharging was effective at both temperatures of 120°C (248°F) and 80°C (176°F) in producing the same level of HISC susceptibility, based on ISL testing of N08830 alloy. Post-fracture hydrogen analyses confirmed effective hydrogen uptake from the procedures used in this study.

Secondary cracking of N08830 was minor compared to API-6ACRA alloys 718 and 725 for all ILS test methods.

Comparing different N08830 test sample geometries for the ISL Method, measured results for reduction in %RA were similar for smooth-round, smooth-rectangular, and 25% lower for notched-rectangular samples. ISL testing did not appear to be sensitive to minor differences in the initial loads, nor times for loading, based on the parameters used in this study.

Based on the results of this study, round specimen, smooth bar, pre-charged ISL testing can be considered as a discriminant test method for evaluating an alloys' susceptibility to HISC.

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