

Long- And Short-Term Laboratory Testing Of UNS N06985 For OCTG In Extreme Sour Environments

Wenle He
Sandvik Materials Technology
Tube R&D
Sandviken, STTPC-4371, 81181,
Sweden

Charlotte Ulfvin
Sandvik Materials Technology
Tube R&D
Sandviken, STTPC-4371, 81181,
Sweden

Per Olsson Artberger
Sandvik Materials Technology
Tube BU Energy
Sandviken, SMT-TTO, 81181
Sweden

ABSTRACT

UNS N06985 is a nickel-based alloy with additions of Mo, Cu and W. Because of its high strength and high resistance to stress corrosion cracking (SCC), cold worked UNS N06985 has been extensively used for OCTG (Oil Country Tubular Goods). To meet deeper and more challenging environments, tube products manufactured in high quality corrosion resistant alloys (CRAs) are required.

Recently, cold worked UNS N06985 tubes have been developed and produced for OCTG in specified minimum yield strength 110 ksi and 125 ksi in two dimensions. Laboratory testing has provided mechanical properties and corrosion resistance to localized corrosion and SCC. The SCC resistance has been verified using both short-term SSRT and long-term autoclave exposures using C-ring and mass loss for 90 days. The SSRT was performed in the maximum boundary conditions of environmental limits for 4d type nickel-based alloys per ISO15156-3, whereas the long-term autoclave exposures were performed in a condition outside of the limits for the 4d type materials.

With comparison to the commercial tube products of UNS N06985 in literature, the newly developed tube materials of grades 110 ksi and 125 ksi showed high SCC resistance and stable mechanical properties. The tube materials fulfill ISO13680/API-5CRA requirement and ISO15156-3.

Key words: corrosion resistant alloy (CRA), stress corrosion cracking (SCC), slow strain rate testing (SSRT), C-ring, laboratory testing, UNS N06985

INTRODUCTION

UNS N06985 is a nickel-based alloy with additions of Mo, Cu and W. Because of its high strength and high resistance to stress corrosion cracking (SCC), cold worked UNS N06985 has been extensively used for OCTG (Oil Country Tubular Goods). The SCC initiation has been studied in $\text{Cl-H}_2\text{S-CO}_2$ containing sour environments; and the SCC resistance was often evaluated using slow strain rate testing (SSRT) for screening by short-term testing and using tensile or C-ring for verification by long-term autoclave testing exposures¹. UNS N06985 showed high resistance to SCC at high temperatures in elemental sulfur containing environments with moderate chlorides². Acceptable environments were reported based on testing results³. The testing conditions were up to 25% NaCl (180 000 mg/l Cl^-), 100 psi H_2S , 200 psi CO_2 , at 218 °C for 60-120 days⁴. On another hand, 52 000 mg/l Cl^- , 1709 psi H_2S , 556 psi CO_2 , 100 g/l elemental sulfur at 149 °C for 90 days have been used for SCC testing to simulate a more severe reservoir in the Middle East, typically with high H_2S contents, high pressures, high temperatures and high salinity brines, by which UNS N06985 products from different material suppliers through distinct manufacturing routes showed acceptable SCC resistance in a fit-for-purpose testing program⁵. All these testing conditions were chosen within the environmental limits for UNS N06985 as 4d type nickel-based alloys per ISO⁽¹⁾ 15156-3⁶/ NACE⁽²⁾ TM0175⁷. Moreover, long-term exposure testing is necessary to ensure the SCC resistance of the high grade CRAs in the deep and sour wells with high concentrations of chloride and hydrogen sulfide at high temperature for at least 90 days, according to the ongoing rewriting of ISO 15156 committee activity.

This paper presents a recently developed tube product of Sandvik⁽³⁾ Sanicro 48⁽⁴⁾ (UNS N06985) and evaluations for an internal product qualification of the products in order to meet OCTG requirements in the more challenging sour environments using both short-term and long-term testing.

Two heats were produced by Sandvik in its fully integrated production and in total eight dimensions were produced, the production started with ingots and was finalized by cold pilgering with a reduction meeting the desired strength level. All dimensions, ranging in OCTG size from 4½" 12.6 ppf to 7" 29 ppf, were proved to fulfil the requirements stated in the standards ISO 13680⁸ and API⁽⁵⁾ Specification 5CRA⁹. 7" 29 ppf and 4½" 12.6 ppf with belonging Coupling Stock (CS) dimensions were in-depth characterized including mechanical and corrosion properties.

EXPERIMENTAL PROCEDURE

Materials

UNS N06985 OCTG tube materials are produced per ISO 13680/API 5CRA group 4, manufactured from ingot through extrusion, cold working and intermediate annealing. Two heats are involved for the evaluations. A commercial tube (from another manufacturer), material UNS N06985, 125 ksi, has been included as a reference material for a comparison in this study. The materials are shown in Table 1 and chemical compositions in Table 2. The tube size is described using both dimension in millimeter (mm) and OCTG denomination for outside diameter in inch (") and weight per length in pounds per foot (ppf).

(1) ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland

(2) NACE International, 15835 Park Ten Place, Houston, TX 77084-5145, United states

(3) Trade name

(4) Trade name

(5) API Standard Department, 1220 L Street, N.W., Washington, D.C. 20005, United states

© 2022 Association for Materials Protection and Performance (AMPP). All rights reserved. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means (electronic, mechanical, photocopying, recording, or otherwise) without the prior written permission of AMPP.

Positions and opinions advanced in this work are those of the author(s) and not necessarily those of AMPP. Responsibility for the content of the work lies solely with the author(s).

Table 1:
An overview of the materials

Materials	A	B	C	D	E	F, Ref
SMYS*	110 ksi	110 ksi	110 ksi	110 ksi	125 ksi	125 ksi
Heat, No.	xxx008, xxx407	xxx407	xxx008	xxx008	xxx008	xxx888
Lot, No.	xxx478, xxx460, xxx414	xxx875	xxx725, xxx832	xxx857, xxx840	xxx615	xxx999
Dimensions (mm)	177.8×10.36	196.7×23.53	114.3×6.88	127.5×16.03	114.3×6.88	88.9×7.34
OCTG Denomination	7" 29 ppf	7" 29 ppf CS	4½" 12.6 ppf	4½" 12.6 ppf CS	4½" 12.6 ppf	3½" 10.2 ppf
Hardness, Max (HRC)	29 / 28	32	27	28	32	32

* SMYS: specified minimum yield strength

Table 2:
Main chemical composition, wt%

Materials	C	Cr	Ni	Mo	Si	Cu	Co	Fe	W	P	S
Heat xxx008	< 0.015	21.5	47.9	6.8	< 0.25	1.7	< 0.5	Bal.	< 1.0	< 0.03	<0.001
Heat xxx407	< 0,015	21.7	48.0	6.8	< 0.25	1.6	< 0.5	Bal.	< 1.0	< 0.03	<0.001
F, Ref	< 0,015	22.5	47.4	6.7	< 0.25	2.3	0.6	Bal.	< 1.0	< 0.03	<0.001

Testing program

The mechanical properties and corrosion resistance were extensively tested for OCTG in two dimensions, 4½" 12.6 ppf (110 & 125 ksi), and 7" 29 ppf (110 ksi) including corresponding Coupling Stock dimensions. All applicable tests are performed in accordance with API 5CRA / ISO 13680.

Mechanical properties

All mechanical properties were evaluated on four quadrants along the tube circumference. Tensile tests were performed at five positions along the tube, see Figure 1, and in accordance with ISO 6892-1¹⁰. To prove the stable hardness properties along the tube, as many as 11 positions along the tube length were tested following ISO 6508-1¹¹ partly included in ISO 13680, using the Rockwell C scale. Inner, outer and also mid-wall locations were tested for the Coupling Stocks, for all locations an average of three indents was reported.

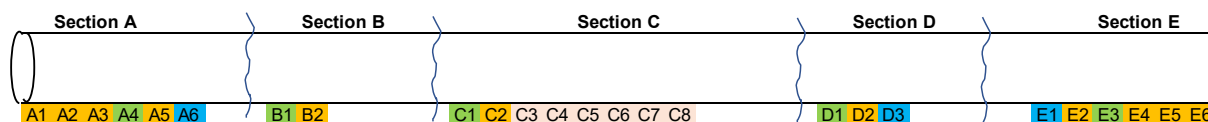


Figure 1: Schematically positions for sample take-outs. Hardness tests in yellow, tensile tests in green, corrosion tests in blue. Positions such as C3-C8 were used for investigations of other characteristics not included in this paper, e.g. anisotropy, de-rating factor of mechanical properties and impact strength, all with satisfactory results.

Pitting resistance

The resistance to pitting corrosion and effect of chloride concentration have been evaluated for 110 ksi 7", material A, in two different test solutions. The test coupons were taken from the tube section A, and prepared by grinding and final finishing using 600 grit SiC paper. The sample size is about 50×20×4 (mm) with a hole of Ø5 mm. Critical pitting temperature was determined according to ASTM⁽⁶⁾ G150¹² in 1M NaCl and in a more aggressive solution 3M MgCl₂. The test was started with the test solution at a temperature of 20°C, and heated at a rate of 1 °C/min. The assessments were carried out at a potential of +734 mV vs. Ag/AgCl. Six specimens were tested in 1M NaCl and four specimens in 3M MgCl₂. All specimens were naturally passivated after final finishing for at least 24 hours before testing. See Table 3.

Table 3:
Test matrix for pitting corrosion resistance

Material	Coupons	
	1M NaCl	3M MgCl ₂
110 ksi 7" (A)	Coupons ground with No. 600 grit abrasive paper	Coupons ground with No. 600 grit abrasive paper

SCC resistance

Stress corrosion cracking was evaluated by short-term slow strain rate testing according to NACE TM0198¹³ and long-term testing for 90 days using C-ring per NACE TM0177¹⁴. The SSRT was performed in the maximum boundary conditions of environmental limits for 4d type nickel-based alloys per ISO15156-3, whereas the long-term autoclave exposures were performed in a condition outside of the limits for the 4d type materials. All tested materials, testing methods and conditions have been summarized in Table 4. See an overview of the materials and testing conditions in relation to ISO 15156-3 limits for Ni-based alloys in Figure 2. The SCC testing has been performed by a third part laboratory, where OLI⁽⁷⁾ simulations were used for preparation of the brine and gaseous components to ensure the target condition for each testing. Total pressure and temperature have been monitored during each of the entire testing period.

The slow strain rate testing has been performed according to NACE TM0198. The samples were taken from the tube wall for manufacture of the SSR specimen. One set testing for each material and test condition included two specimens that have been tested in a specified sour environment and one specimen has been tested in an inert environment (N₂) as a baseline at the same test temperature and total pressure as the sour environments, strain rate 4×10⁻⁶ in/in/s. Environment 1 contained 1 g/l elemental sulfur (S⁰) whereas Environment 2 was without sulfur. The elemental sulfur (375 mg) was added to a deaerated dry test vessel (500 ml) and deaerated brine solution (375 ml) was transferred into the test vessel and then further deaerated one hour.

After the testing, final pH has been measured after the testing at room temperature before removal of gases. Time to failure (TTF), ultimate tensile strength (UTS), plastic elongation (Ep) and reduction of area (RA) were determined for the test specimens. The ratios between the specimens tested in the sour environment and the specimen tested in N₂ were calculated for TTF, Ep, and RA. Observation for possible secondary cracks has been performed on the specimens tested in the sour conditions. A criterion applied

⁽⁶⁾ ASTM International, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959, United states

⁽⁷⁾ Trade name

© 2022 Association for Materials Protection and Performance (AMPP). All rights reserved. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means (electronic, mechanical, photocopying, recording, or otherwise) without the prior written permission of AMPP.

Positions and opinions advanced in this work are those of the author(s) and not necessarily those of AMPP. Responsibility for the content of the work lies solely with the author(s).