

Failure Analyses On A Super Duplex Stainless Steel Tube Material Before And After Slow Strain Rate Testing In Sour Conditions – Part 1: SCC Susceptibility

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ABSTRACT

For safely using duplex stainless steel and super duplex stainless steel, temperature-pH domains of stress corrosion cracking (SCC) resistance were provided by using slow strain rate testing (SSRT) screening in sour conditions in an earlier study.

To follow-up the study, a failure analysis has been performed on the tube material of UNS S32750 and the specimens after the SSRT in a sour environment in pH 2.8, pH 3.5 at 80 °C. Scanning electron microscope (SEM) equipped by energy-dispersive X-ray spectrometer (EDS) analysis and Electron backscatter diffraction (EBSD) measurements have been used for the investigation on specimen surface, fracture surface and cross-sections of the SSRT specimens. Initiation of selective dissolution (SD) and cracking has been identified. EBSD reveals that cracking initiated in ferrite with enhanced strain/deformation in frontend of selectively dissolved austenite, where possible SCC mechanism seems to be hydrogen embrittlement. It observed that SD stopped in the brittle-ductile transition area in the fracture surface, which probably attributed to repassivation of the material in the condition of pH 3.5. Combined SSRT and analysis methods by SEM and EBSD showed a good way for the material evaluation before decision making for the materials selections and supplementary long-term exposures.

Key words: Duplex stainless steel (DSS), super duplex stainless steel (SDSS), stress corrosion cracking (SCC), selective dissolution (SD), scanning electron microscope (SEM) equipped by energy-dispersive X-ray (EDS) analysis, and electron backscatter diffraction (EBSD) measurement.

INTRODUCTION

Standards NACE⁽¹⁾ MR0175 / ISO⁽²⁾ 15156-3¹ have temperature-H₂S-Cl-pH limits for safely using duplex stainless steels based on their Pitting Resistance Equivalent Number (PREN) of subcategory such as duplex stainless steel (DSS, PREN 30-40, Mo>1.5%) and super duplex stainless steel (SDSS, PREN 40-45), temperature-pH domain of stress corrosion cracking (SCC) resistance can be used for material evaluation and provided by using slow strain rate testing (SSRT) screening in sour testing conditions².

In earlier studies^{2,3}, the plate- and tube products were tested in NACE standard solution A (50 g/L NaCl, 5 g/L NaCH₃COO), under 0.5 bar (7.25 psi) H₂S at temperature 23 °C, 50 °C, 80 °C and pH 2.8, 3.5, 4.5, and 6. The super duplex tube product showed higher SCC resistance compared to the duplex plate product while the hyper duplex stainless steel plate product did not show SCC or selective dissolution (SD) in the same testing conditions. It has been found that a UNS S32750 tube product was SCC susceptible and underwent selective dissolution in austenitic phase (γ) at 80 °C when pH $\leq$ 3.5, see T-pH domain and stress-strain curves in **Figure 1**.

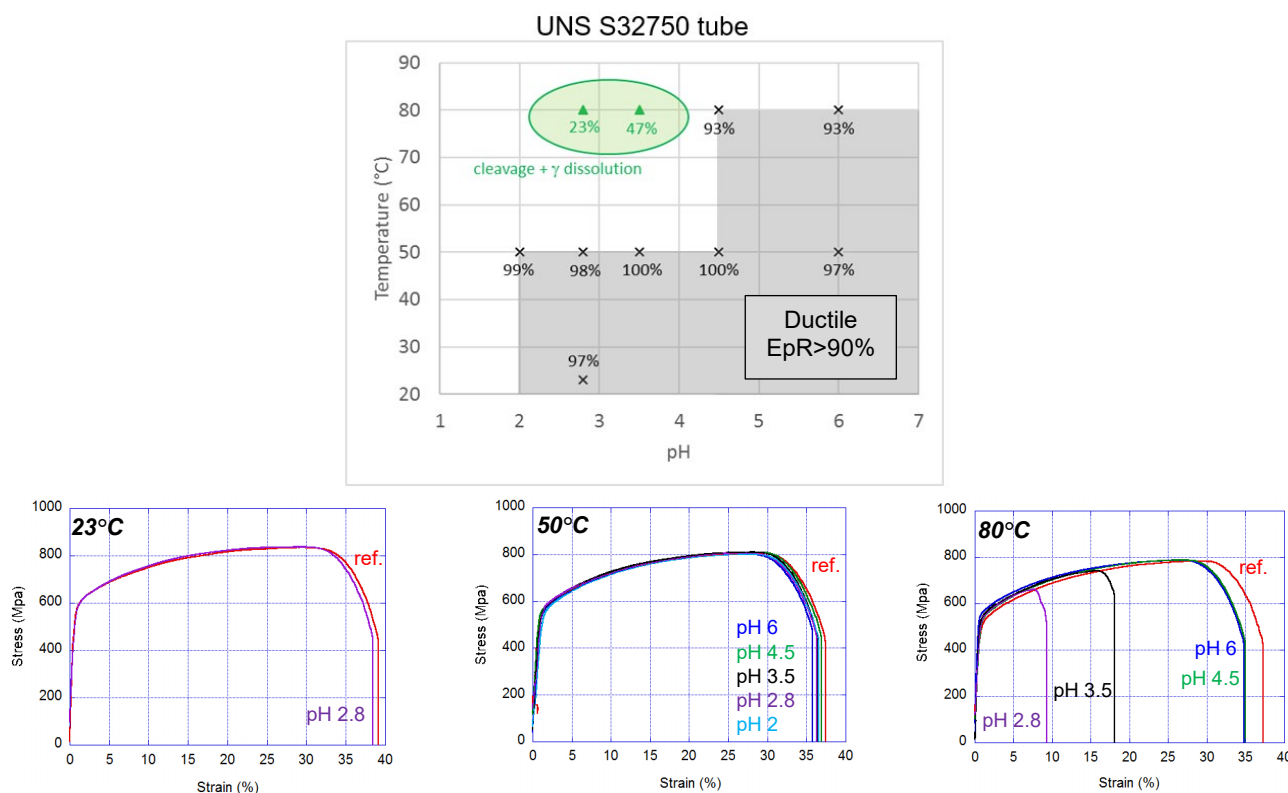


Figure 1: T-pH domain (upper picture) for a UNS S32750 tube material by SSRT in NACE Solution A: 50 g/L NaCl, 5 g/L NaCH₃COO, acidified by HCl for reaching the specified pH, 0.5 bar (7.25 psi) H₂S. Plastic elongation ratio (EpR > 90%) in the grey condition area; SCC and austenite SD were observed in the green condition area. Stress-strain curves in lower pictures.

Fracture surface observation on SSRT specimen UNS S32750 after testing in the sour environment at 80 °C, pH 3.5 showed that localized corrosion by SD in austenite has been observed near to the specimen surface. Cleavage is observed near the SD area, however deeper part of the fracture surface is ductile with dimples and voids. See **Figure 2**. Whereas, the specimen after the testing at 80 °C, pH 2.8 showed

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brittle fracture surface with selective dissolution in austenite and cleavage in ferrite in the edge of fracture surface; cleavage in both austenite and ferrite in deep part of specimen, see in **Figure 3**.

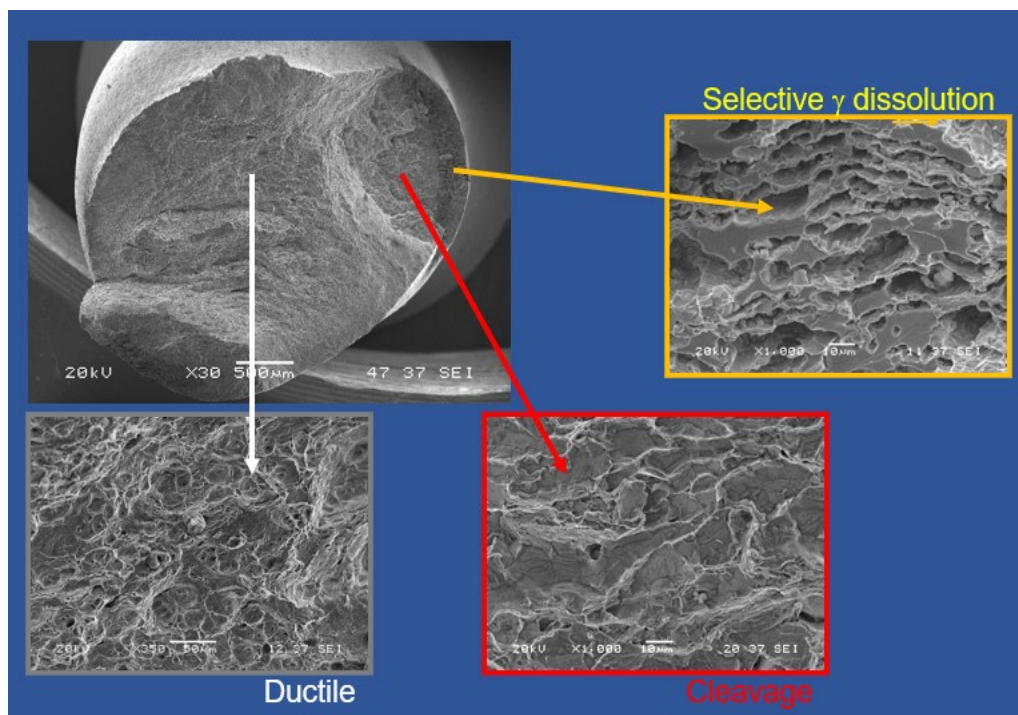


Figure 2: Fracture surface observation on SSRT specimen UNS S32750 after testing in the sour environment at 80°C, pH 3.5, showed selective dissolution in austenite in the edge of fracture surface, followed by brittle and ductile fracture into the center of specimen.

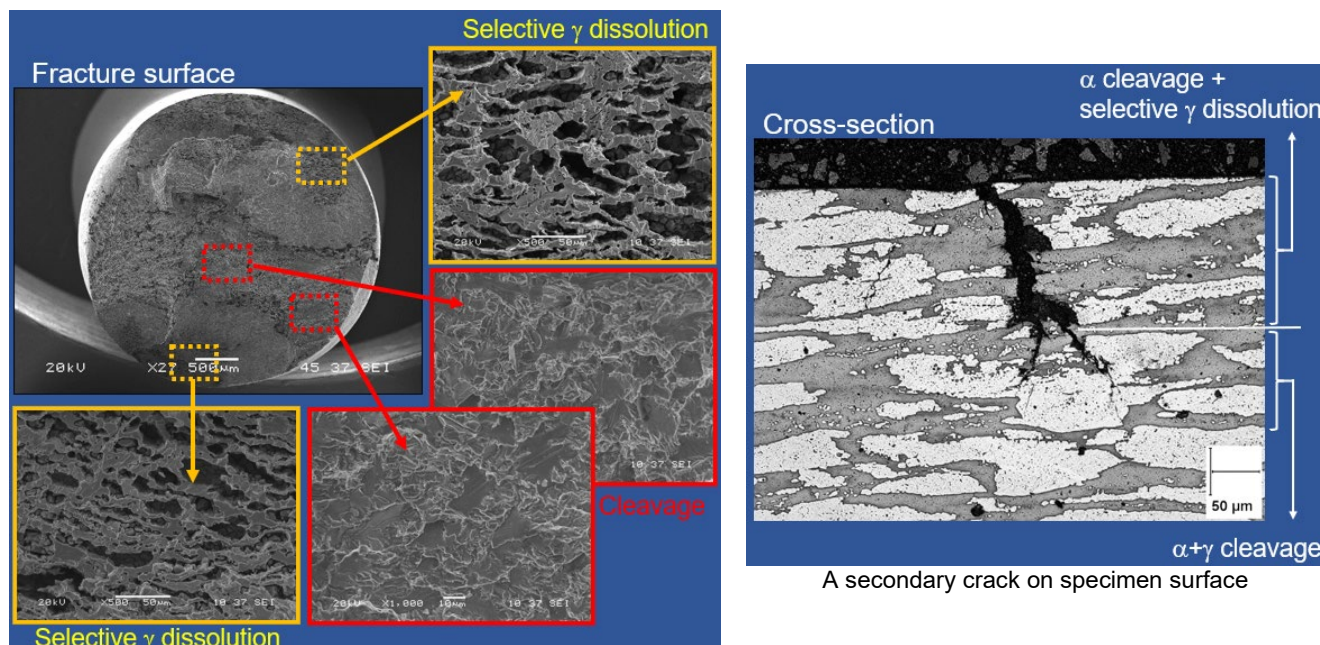


Figure 3: Fracture surface observation on SSRT specimen UNS S32750 after testing in the sour environment at 80 °C, pH 2.8, showed selective dissolution in austenite in the edge of fracture surface, followed by brittle fracture through whole specimen.

To follow-up the study², all SSRT specimens have been observed and an investigation has been performed on the tube material of UNS S32750 and the specimens which showed SCC and localized corrosion after the SSRT, using scanning electron microscope (SEM) equipped by energy-dispersive X-ray (EDS) analysis, and electron backscatter diffraction (EBSD) measurements.

EXPERIMENTAL PROCEDURE

Material and specimens

A final tube product of super-duplex UNS S32750 (Sandvik SAF 2507®)⁽³⁾, has been used in the previous study, which has specified minimum yield strength 80 ksi, in dimension outer diameter 140 mm and wall thickness 7.72 mm. Slow strain rate testing (SSRT) specimens were machined in the longitudinal direction taken from the tube wall, into standard cylindrical subsize as per NACE geometry. The chemical composition is given in **Table 1** including PRE number and austenite spacing.

Table 1: Nominal composition of main elements in the materials used for testing, wt%

UNS	Product	Cr	Ni	Mo	N	PREN*	Austenite spacing
S32750	Tube, mm OD140*WT7.72	25.59	6.47	3.84	0.3	43	6.2±5.8 µm

* Pitting resistance equivalent number, $PREN = \%Cr + 3.3 \times (\%Mo + 0.5\%W) + 16 \times \%N$

The SSRT specimens after the testing have been investigated for a better understanding of the selective dissolution and cracking, including initiation and propagation. NACE standard solution A was used containing 50 g/L NaCl, 5 g/L CH₃COONa (sodium acetate) according to standard NACE TM0177⁴. The pH was adjusted once the solution reached the test temperature and was saturated with the test gas using additions of HCl (18 wt%) to values from 6 down to 2.8, and even lower (pH 2) for one test. At least one specimen has been tested in each condition. A summary of the testing results is shown in T-pH domain in **Figure 1**.

Investigation methods

Optical stereo microscope as well as scanning electron microscope equipped with energy X-ray dispersive spectroscopy (SEM-EDS) have been used for observations and elemental investigation on the microstructure, specimen surface and fracture surface. A Dual-Beam FEGSEM instrument (Carl Zeiss⁽⁴⁾ CrossBeam 1540 EsB) was used, which consists of a Gemini-type Field Emission Gun (FEG) electron column and with a Focused Ion Beam (FIB) device. This instrument is equipped with an Oxford EDS-system to perform microanalysis and collect X-Ray elemental Maps. The elemental maps were collected as energy intensities and confirmed with EDS point analysis to avoid critical elemental overlaps. Typical primary energy in use was 10-20 keV. The SEM-EDS has also been used on cross-section samples which were taken in longitudinal direction of the SSRT specimens. Both secondary electron (SE) imaging and backscattered electrons (BSE) imaging were used for showing topographical difference, e.g., corrosion products, micro-cracks. EDS point analysis and elemental mapping provided information about selective dissolution phase and corrosion products formed on the surfaces. Notice that the Mo signal overlaps with S when analyzing the corroded phase and corrosion products, however it provides the corrosion in relation to the phases, i.e. ferrite phase has more Mo compared to austenite. Therefore,

⁽³⁾ Trade name

⁽⁴⁾ Trade name

some of the elemental mapping on S has been combined with Mo signal to provide the location information of the corrosion area for the selective dissolution in this study.

Further investigation using electron backscattered diffraction (EBSD) measurements has been carried out for microstructural characterization of the super duplex stainless steel UNS S32750 on the cross-section samples from both the original tube and the specimens after the SSRT in the sour environments.

The sample preparation involves grinding of embedded samples down to a fine finish obtained with 1 μm diamond paste and subsequently polished with colloidal silica (Oxide Polishing Suspension, OPS). The polished specimens were subjected to EBSD data acquisition in Carl Zeiss⁽⁵⁾ Field Emission Gun scanning electron microscope equipped with the Symmetry EBSD detector supplied by OXFORD Instruments⁽⁶⁾. The data acquisition was carried out at a detector speed of approximately 2000 Hz. The scan areas and step sizes varied depending on the sample conditions. In the case of as-received sample, a total area of approximately $0.077 \times 0.0288 \text{ cm}^2$ was covered with a step size of 0.3 μm . On the other hand, in the case of SSRT specimens, scan areas were varied and step sizes in the range of 0.15 μm to 0.3 μm were employed. Post processing analysis was carried out with OIM analysis 8⁽⁷⁾ software for undeformed specimens and 'HKL Channel5' for SSRT specimens. For EBSD analysis, grain tolerance angle of 5° to differentiate two neighboring grains was applied. Phase maps and fractions, grain boundary densities of 1°-15° and >15° grain boundaries, Kernel average misorientation (KAM) map and area weighted average grain size of ferrite phase were evaluated in previous study². A few of the parameters presented in this investigation are briefly described below:

- KAM is a local misorientation parameter which is the average misorientation of a pixel with the neighboring pixels and with a choice of excluding some pixels that exhibit higher misorientation angles. In this study, neighboring pixels higher than 5° misorientation angles were excluded. These regions of high KAM values indicate presence of residual strain or in other words the microstructure is not fully recrystallized.
- The high KAM regions/areas are noted to contain high density of 1°-15° grain boundaries which are termed as low angle grain boundaries (LAGBs) and are considered to be deformed regions². The presence of deformed regions could be due to remnants of residual strain in the case of undeformed material. However, in the case of SSRT samples, this could be due to either tensile loading and/or deformation induced during corrosion and cracking propagation. Hence, in order to understand the corrosion behavior of constituent phases of the selected steel, EBSD phase maps with different phases in different colors overlaid with LAGBs, >15° grain boundaries (HAGBs) and $\Sigma 3$ twin boundaries are presented in this study.

RESULTS AND DISCUSSION

Optical microscope observations of SSRT specimens

All the SSRT specimens have been observed using an optical stereo microscope. The specimens with SCC and local corrosion observed are shown in **Figure 4**, which are included in further investigations using SEM-EDS and EBSD in this study.

⁽⁵⁾ Trade name

⁽⁶⁾ Trade name

⁽⁷⁾ Trade name