

**Impact of material properties on the performance of thick-wall
X65 TMCP plate steel for sour service**

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ABSTRACT

Low-alloy steel of API 5L grade X65 is commonly used as line pipe material for sour service applications. Within recent years more challenging requirements have been introduced, including more severe corrosion test conditions, increased mechanical properties, and extended limitations regarding hardness, to enable application to increasingly severe sour service conditions. Within this study a systematic investigation of material properties related to corrosion resistance was performed for low sulfur X65 TMCP-based steel plates with a thickness of 30 mm and 35 mm. Plate-to-pipe forming simulation to different wall thickness to diameter ratios (t/D) was undertaken and HIC testing according to NACE TM0284 was performed to investigate the HIC resistance after cold forming of heavy plates to SAWL pipes. Wall thickness to diameter ratios of 5 % and above were found to be accomplishable. In addition, hydrogen permeation measurements were used to further investigate the influence of the microstructure on the hydrogen diffusion. SSC resistance tests according to NACE TM0316 were carried out under standard test conditions (1 bar H_2S) and elevated pressure for fully machined specimens and for specimens containing the as-received plate surface. The SOHIC resistance was assessed using the new "Twist-and-Bend"-SOHIC test, which is currently under standardization within AMPP, based on applied twist and load. The results of electron backscatter diffraction (EBSD) measurements on plate material were correlated with the hydrogen permeation measurements. The results support the understanding of significant material-related factors, which is essential for the development of high-quality line pipe steel for sour service applications.

Key words: HIC, SSC, SOHIC

INTRODUCTION

Low-alloy steel of API⁽¹⁾ 5L grade X65 is commonly used as line pipe material for sour service applications, where, due to stringent operation limits, resistivity to Hydrogen Induced Cracking (HIC), Sulfide Stress Cracking (SSC) and Stress-Oriented Hydrogen Induced Cracking (SOHIC) is required and tested before and after plate into pipe forming. Within recent years more challenging requirements have been introduced, including more severe corrosion test conditions, increased mechanical properties and extended limitations regarding hardness, to enable application to increasingly severe sour service conditions. For steel plants and plate manufacturers this entails the necessity to further improve the quality of their products together with separate improvements and modifications on the production plants and processes that have been implemented anyway. As the complete production cycle from blast furnace to finished pipe is expensive and time consuming, understanding the influence of the relevant production steps is vital for plate and pipe development and manufacture. Within this work the impact of material properties of thick-wall X65 TMCP (Thermo-Mechanically Controlled Processed) plate on sour service performance is shown and discussed for the various steps of the production cycle.

STEELMAKING AND PLATE ROLLING

The investigated steel for sour-service applications was produced at an integrated iron and steel plant. Two hot-metal desulphurization stations, two converters with a change-vessel converter installation, two ladle furnaces, one ladle treatment station and four vacuum installations are operated to permit flexible adaptation to the production conditions. **Figure 1** gives an overview of the installed production facilities in our hot metal shop.

The production of slabs for sour service applications commences with hot-metal desulphurization, in which the hot metal is desulphurized down to ultra-low sulphur contents. In the subsequent converter process, necessary low phosphorus contents are adjusted. Until the second half of the year 2020 tapping, desoxidation and alloying was followed by intensive argon stirring. In the second half of the year 2020, the argon stirring stations were replaced by two ladle furnaces, which are capable of reheating the melts to a given temperature. This was a major step, allowing to lower the tapping temperature and to achieve lowest phosphorous contents. After reheating the melts to ladle treatment temperature, the adjustment of chemical composition, ultra-low nitrogen, hydrogen and sulphur contents as well as Ca-treatment is conducted in the vacuum degassing unit. Continuous casting is done on a caster which dissipates the heat from the solidifying slab primarily via radiation to assure production of zero-defect surfaces and a very uniform solidification. This, in combination with the applied soft reduction, achieves an extremely low segregation center zone.

For this investigation, plates originating from two different heats were processed. The first heat for the production of 30 mm plates was produced with argon stirring after converter tapping whereas the second heat for 35 mm plates took advantage of the benefits of the new installed ladle furnace with a significant lower phosphorous content. The chemical composition of the heats is shown in **Table 1**. Plates were rolled using TMCP with accelerated cooling.

¹ American Petroleum Institute (API), 1220 L. St., N.W., Washington, DC 20005-4070

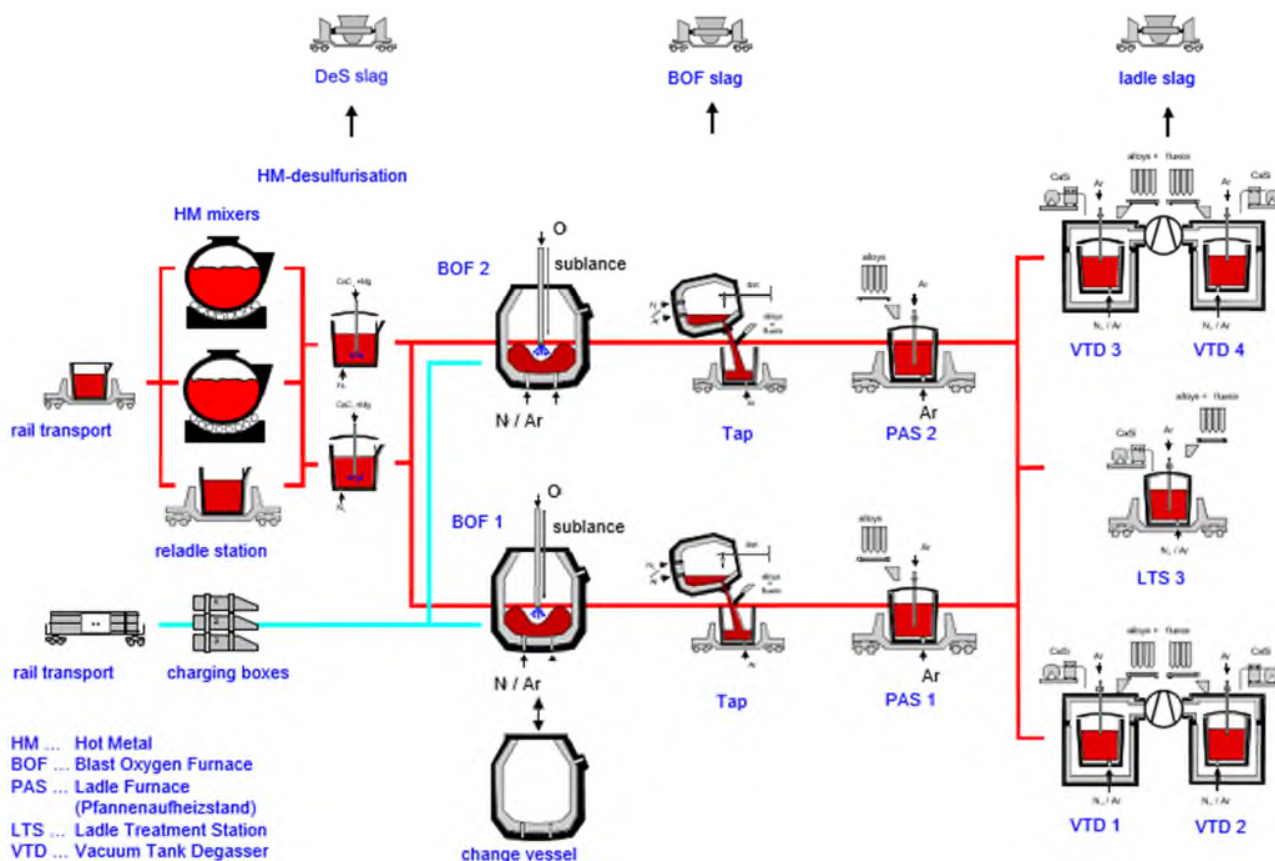


Figure 1: Production facilities at hot metal shop

Table 1
Chemical Analysis of Investigated Steel Heats

Heat	C	Si	Mn	P	S	Al	Cu+Cr+Ni	Mo+V	Ti+Nb
30 mm	0,043	0,27	1,35	0,013	0,0005	0,038	0,87	0,02	0,01
35 mm	0,037	0,25	1,39	0,006	0,0004	0,013	0,88	0,01	0,04

HYDROGEN INDUCED CRACKING (HIC) RESISTANCE

The resistance of pipeline steel to HIC is typically tested according to NACE TM0284.¹ HIC tests have been performed for the rolled plates with 30 mm and 35 mm thickness with 528 individual specimens in total.

Experimental Procedure

At least three individual specimens have been machined from each plate. Testing has been performed under standard conditions in NACE TM0284 test solution A for 96 h under 1 bar H₂S. Evaluation has been made only by the ultrasonic method as per NACE TM0284 Annex A determining the CAR (Crack Area Ratio). Detailed evaluations have shown that a CAR of up to 23 % will represent the CLR limit of maximum 15 % as given in NACE MR0175/ISO⁽²⁾ 15156-2.^{2,3}

² International Organization for Standardization (ISO) Chemin de Blandonnet 8, CP 401 1214, Vernier, Geneva, CHE

Results and Discussion

The results from ultrasonic evaluation are shown in **Table 2**. For both plate thicknesses about 86 % of the test specimens showed no indications with CAR = 0 %. However, the results differ for higher CAR ranges. For 30 mm thick plates 9 % of the specimens showed a CAR between 0 and 1 % and for 5 % the CAR was above 1 %. For the plate variant from the new installed ladle furnace with lower phosphorous content - rolled to 35 mm - improvement on HIC resistivity has been achieved as the amount of specimens with CAR > 1 % was reduced to 1.5 %, despite the fact that more specimens have been tested.

Table 2
HIC Results for Plates with 30 mm and 35 mm Thicknesses

Plate thickness	CAR	Number of specimens	Percentage of specimens
30 mm	0 %	169	86,0%
	0 - 1 %	17	9,0%
	> 1 %	9	5,0%
35 mm	0 %	287	86,5%
	0 - 1 %	40	12,0%
	> 1 %	6	1,5%

Influence of pipe forming on Hydrogen Induced Cracking resistance

Acceptable HIC results on a plate are not transferred into the finalized pipe by itself. In the pipe manufacturing step, the flat plate is formed into a pipe or tube, e.g. with a UOE process for large-diameter pipes, where the plate is formed into an U-shape in the first step and subsequently into a circular O-shape using an O-press and, after longitudinal submerged arc welding (SAW), expanded by internal sizing to the final geometry. The degree of cold deformation can be assessed as the ratio of wall thickness "t" to final pipe outer diameter "D". For a 42" pipe with a wall thickness of 30 mm t/D is 2.8 %, in case of 32" and a wall thickness of 35 mm t/D is calculated to 4.3 %. As pipe forming can influence the HIC resistivity, knowledge on the maximum achievable deformation ratios is thus necessary for a plate manufacturer for safe design of HIC resistant plates.

For this purpose, a lab-scale simulating pipe forming installation has been developed and validated. In a four-point bend geometry a plate strip taken transverse to the plate axis is deformed, giving an area in the mid-length, where controlled deformation is achieved. From this region HIC specimens, oriented longitudinally to the rolling direction (pipe axis) were taken. Individual strips of approximately 1 m length taken from some of the plates with 30 mm and 35 mm thickness, which showed no HIC (CAR = 0 %) have been formed to t/D of 2.5 %, 3.5 %, 4.2 % 5.0 % and also 6.0 % and tested for HIC. The highest individual values for CAR have been taken for further qualitative evaluation.

For each investigated plate with a thickness of 30 mm or 35 mm the influence of pipe forming (t/D ratio) on the HIC resistivity has been investigated by HIC testing of deformed plate sections. The dependency for nine individual plates (30 mm: 391, 381, 380, 377; 35 mm: 159, 157, 147, 141, 137) is shown in **Figure 2** based on t/D and CAR. At 2.5 % and 3.5 %, which are typical of large-diameter pipes, no effect of pipe forming on HIC can be detected. Further forming to 4.2 % only induces minor HIC in 2 of the 9 plates. A visible effect of forming on HIC starts at 5 %, where 5 of the 9 plates showed visible HIC indications in a wide CAR range between 0.2 % and 1.2 %. For two plates in addition a deformation of

6 % has been applied, resulting in higher CAR up to 1.5 %. All indications can be found in general near the plate surface, related to small nonmetallic inclusions. Their interaction with the strained matrix is assumed responsible for the HIC initiation. The results show limiting t/d ratios, above which HIC initiates. The maximum CAR values obtained after HIC tests at t/d up to 6 % were, however, far below well-established acceptance limits. To be noted, in a HIC test on a formed pipe an additional HIC specimen transverse to the weld seam is required.

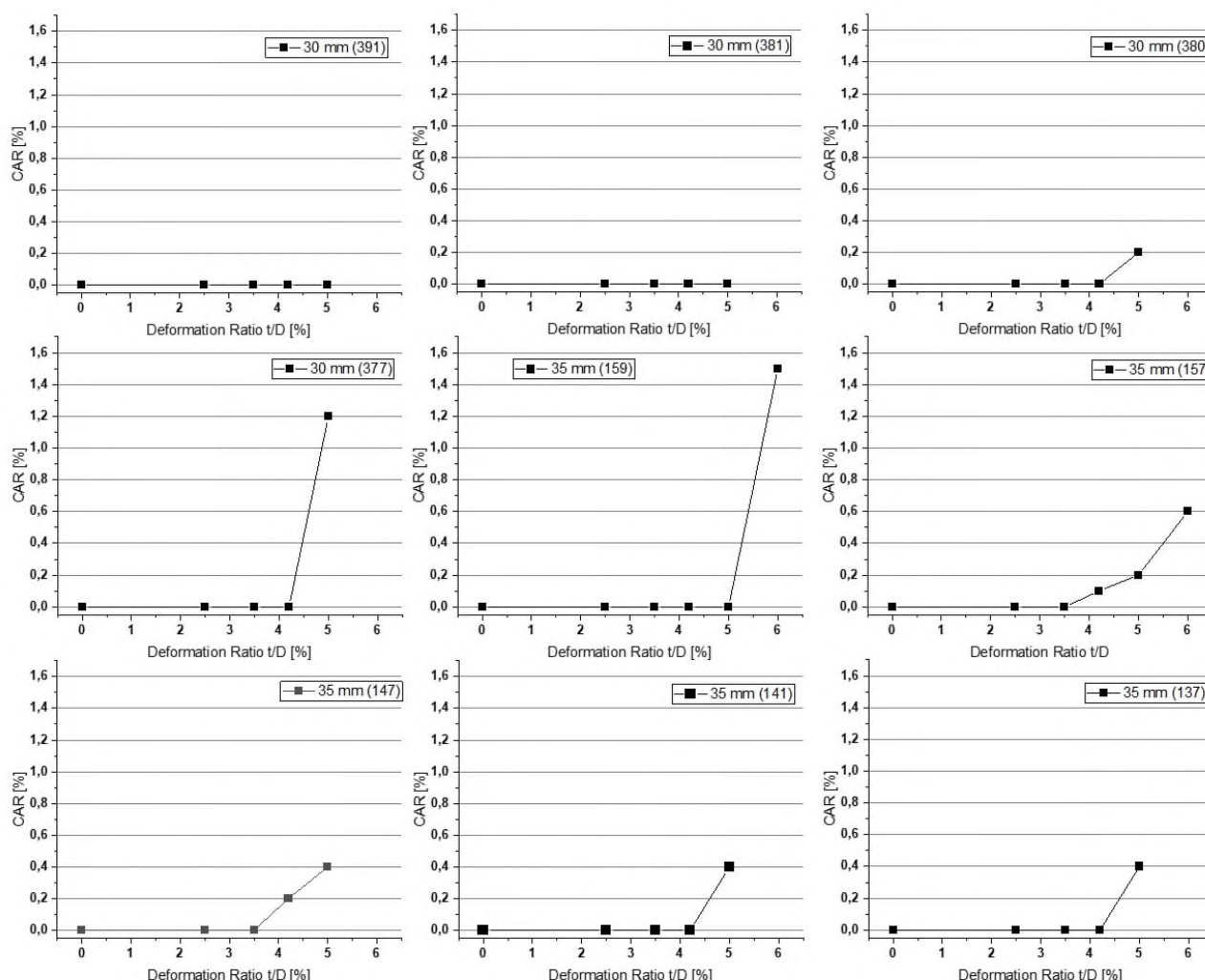


Figure 2: Influence of simulated pipe forming on HIC for investigated plates

SULFIDE STRESS CRACKING (SSC) RESISTANCE

SSC resistance testing for TMCP line pipe steels is typically performed as a four-point bend test according NACE TM0316.⁴ In the past testing was mostly based on the NACE MR0175/ISO 15156-2 pH-H₂S regions of environmental severity diagram.² As qualification for the most severe SSC region 3 tests at 1 bar H₂S were supposed to cover the full region 3. Recently, concerns have been raised on the validity of this approach for H₂S partial pressures above 1 bar,^{5, 6} and thus requirements for SSC testing under elevated pressure of H₂S with the addition a CO₂, which more represents in-field conditions, were defined together with more stringent hardness limits below the NACE MR0175/ISO 15156-2² requirement of 250 HV10 maximum.