

Effect of Nb Addition and Processing Path on The K_{ISSC} of a Low Alloyed OCTG Steel

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ABSTRACT

Due to their low ratio of cost versus performances, quenched and tempered (Q&T) high strength low alloyed steels are widely used as tubular goods. For several decades, steel suppliers and academics are investigating the mechanisms of sour corrosion of these materials. Several publications highlighted the different parameters influencing the K_{ISSC} of Q&T high strength low alloyed steels like the prior austenitic grain size, the dislocation density or the precipitation state. Niobium is known to have an impact on these 3 parameters, but authors often looked at its impact separately. Additionally, complete data relating the Niobium content and the K_{ISSC} are extremely rare in the open literature.

The present study focuses on the impact of Nb on the Prior Austenite Grain Size (PAGS) refinement for several quenching and tempering cycles and additionally on the role of Nb on the critical stress intensity factor for sulfide stress cracking (K_{ISSC}). A common Cr-Mo high strength OCTG steel alloyed with three different Nb contents ranging from 300 to 1000 ppm is used. It was found that Nb addition allows the use of higher strength material without compromising the resistance to crack propagation (K_{ISSC}). The results are discussed in terms of microstructure, hydrogen pick-up and precipitation state.

Key words: OCTG, K_{ISSC} , Niobium, SSC

INTRODUCTION

The use of Oil Country Tubular Goods (OCTG) material in harsher environments (deeper wells, higher H_2S concentrations and/or higher pressure and temperature) is pushing suppliers to design new steel grades with higher strength and better SSC resistance. This is extremely challenging since it is well known that the SSC resistance of a given material decreases with increasing its strength. Since the late

80's, people have been studying the metallurgical factors of low alloyed steels playing a role on the SSC resistance [1]–[3]. Quenched and tempered (Q&T) steels progressively became the reference since this processing allows to meet a high yield to tensile ratio answering the hardness limit imposed by successive API standards.

Obtaining a fully martensitic structure after austenitization and quenching is key. Upper bainite, even present in low amounts, drastically lowers the SSC resistance [4], [5], Figure 1a) and b). An increased final tempering temperature allows to reduce the dislocation density and has been observed to have a beneficial effect on the SSC resistance [6].

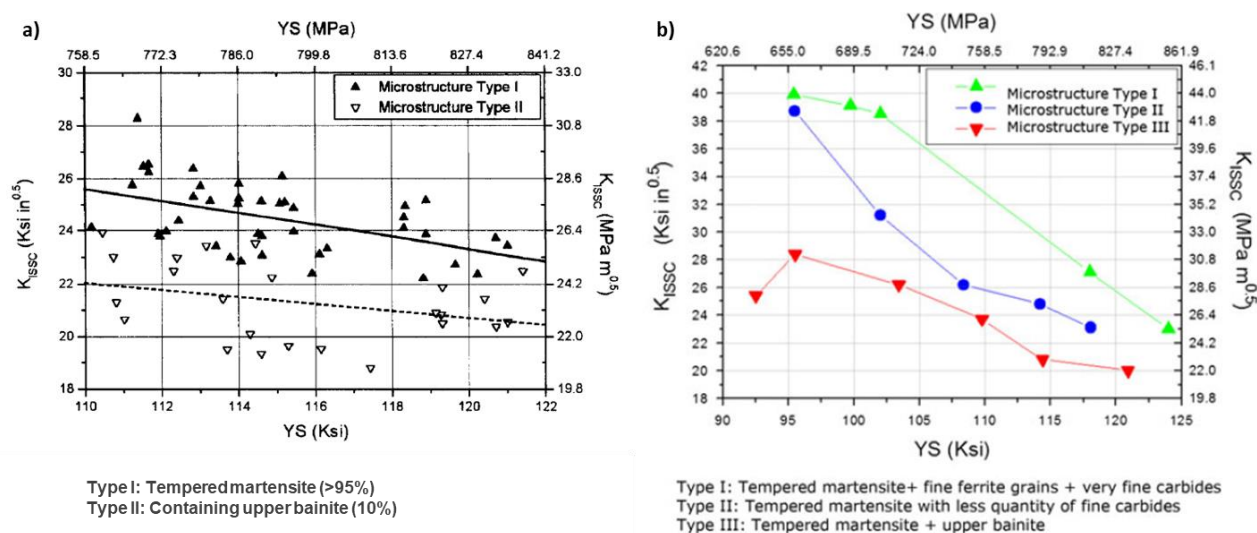


Figure 1: Effect of the microstructure on the K_{ISSC} , a) from [4] and b) from [5].

Microalloying addition like Niobium is also of major importance. Precipitation of fine and homogeneously distributed Nb-carbides (NbC) during the processing allows to control and refine the Prior Austenite Grain Size (PAGS) together with multiple quenching leading to an improved SSC resistance [3], [4], [7]. Fine carbides are potential trapping sites for absorbed hydrogen and are believed to improve the overall hydrogen cracking resistance [8]–[12].

However, very few studies focused on the direct relationship between Nb and K_{ISSC} . Zhang et al. [13] showed for iso-strength materials tempered at 700°C that the K_{ISSC} is improved by increasing the Nb content, Table 1. However, some data are missing to confirm and understand this beneficial effect like the PAGS or the processing and heat treatment details of each material. Complete data linking the Niobium content and the K_{ISSC} are extremely rare in the open literature.

Table 1: Effect of Nb content on the K_{ISSC} of C110 casing steels [13].

Nb content (wt%)	0	0.015	0.030	0.060
Tempering temp (°C)	650	675	700	700
Yield strength (MPa)	792	787	776	783
K_{ISSC} ($MPa \cdot m^{0.5}$)	23.6	25.2	29.5	31.5

Therefore, for the present study we chose to characterize, for a common low-alloyed Cr-Mo high strength steel, the impact of Nb on the PAGS refinement for several quenching and tempering cycles and then the Nb effect on the K_{ISSC} by correlating microstructure, hydrogen pick-up and precipitation state.

EXPERIMENTAL PROCEDURE

Laboratory cast ingots (100 kg each) of low alloyed OCTG steel were realized with 3 different niobium levels at ArcelorMittal Global R&D Gent. The base composition is taken within the API 5CT requirements with the C, Mn, Cr and Mo level kept constant in each material. The target concentrations of Nb were 300, 600 and 1000 ppm. The realized compositions of these 3 grades are available in Table 2 and hereafter labelled as Low, Medium and High Nb.

Table 2: Realized chemical composition in wt% of the 3 Nb low alloyed OCTG steels. The C, Mn, Cr and Mo level is constant for each material.

	C	Mn	Cr + Mo	Nb
Low Nb	< 0.35	< 1.2	< 2.5	0.033
Med Nb				0.067
High Nb				0.111

Hot rolled plates of 20 mm final thickness are obtained from the cast blocks and heat treatments to get either a single quenched and tempered (SQT) plate or a double quenched and tempered plate (DQT). Austenitization is done at 890°C for 30 minutes followed by water quenching. In the case of DQT plates, a second cycle of austenitization + water quenching is applied. For both cycles, the final tempering is done at 675°C with a soaking time varying from 30 to 90 minutes depending on the Nb content in order to target the middle of the yield strength range of C110 grade. The final mechanical properties are given in Table 3.

Table 3: Mechanical properties obtained after the final tempering.

	Yield strength (MPa)			
	SQT		DQT	
	Plate 1	Plate 2	Plate 1	Plate 2
Low Nb	813	815	789	795
Med Nb	811	813	806	810
High Nb	823	823	820	832

DCB testing is performed according to NACE TM0177 for 14 days in NACE solution A saturated with 1 bar of H₂S. Chevron notched samples are used. Specimens are machined at mid-thickness of the plates in the rolling direction. The wedge thickness is selected to provide an arm displacement value in the center of the range recommended by NACE TM0177 for grade C110. Six specimens are tested for each material and process condition.

To study the hydrogen pick-up and trapping behavior of each Nb grade, an internally designed test called “fast extraction” was used. In this test, samples are submerged in NACE solution A saturated with 1 bar of H₂S and extracted after different exposure times in such a way that the extraction does not perturbate the rest of the test. Once extracted, samples are directly measured by hot extraction at 300°C or Thermal Desorption Spectroscopy (TDS) up to 700°C after removal of the corrosion products. For each composition, 5 extraction times are chosen, 5h, 1, 2, 5 and 14 days with 3 samples per exposure time.

RESULTS

The Prior Austenite Grain Size (PAGS) was measured according to ASTM E112-96(04) standard with the linear intercept method for the 3 Nb materials right after each austenitizing and quenching (A+Q) cycle, Figure 2. For the sake of trend illustration, a third quenching cycle was applied and measured. Figure 3 illustrates the microstructure for each combination of Nb content and processing path. From

Figure 2, it is clear that the PAGS decreases with increasing the number of A+Q cycles. After a single A+Q, the PAGS is in a range of 15 to 18 μm while it is about half that size after a double A+Q.

However, it should be borne in mind that the PAGS can be quite heterogeneous. This is illustrated on Figure 4 where the PAGS distribution of each point of Figure 2 is represented. A double A+Q clearly reduces the spread in size and narrows the distribution towards smaller grains.

The Nb content also influences the PAG refinement. Indeed, from Figure 2 we can see that a higher Nb content slightly decreases the average PAGS for a single A+Q. Moreover, the grain size dispersion is slightly reduced with the medium and high Nb content, Figure 4. Thermodynamic calculations confirmed that a higher Nb concentration increases the driving force for NbC precipitation which pins the austenite growth earlier in the process leading to a smaller PAGS after rolling and single A+Q. Nevertheless, the effect of the Nb content on the PAG size and spread is not noticeable anymore once a double or triple A+Q is performed.

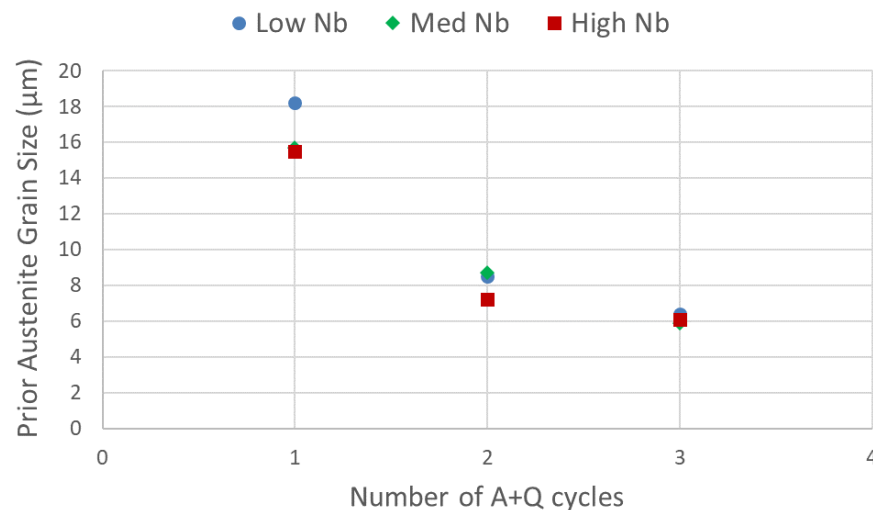


Figure 2: Prior Austenite Grain Size of the 3 Nb steels in function of the number of quenching cycles.

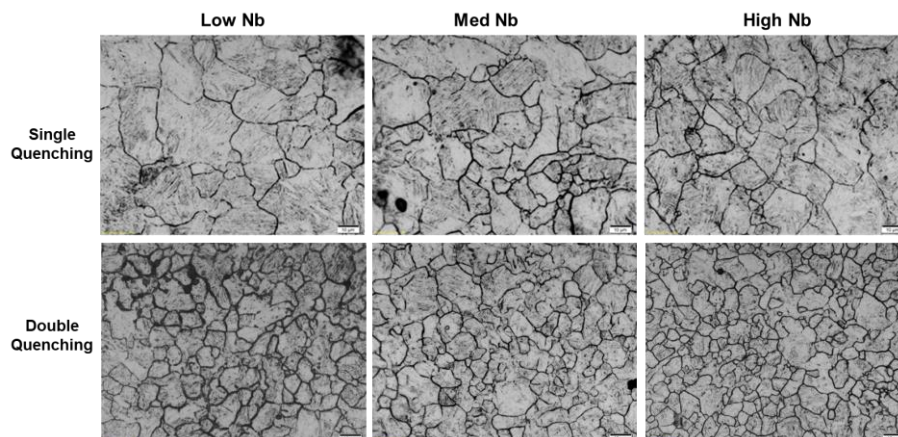


Figure 3: Prior Austenite Grains revealed after Béchet-Beaujard etching.