# OPTIMIZATION OF THE PRODUCTION PROCESS FROM THE MELT SHOP TO THE ROLLING MILL TO GET HEAVY PLATES FOR SOUR SERVICE APPLICATIONS*

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**Abstract**
The use of heavy plates resistant to hydrogen induced cracking in sour service applications has become fundamental to satisfy the greater demands that are required worldwide in the works and facilities of large diameter pipes. The industrial production of heavy plates is continuously being refined from the steelmaking process to hot rolling, through special techniques of mechanical and microstructural characterization. This paper shows the main metallurgical concepts that are established in plate specification engineering; in the definition and optimization of the parameters of the steel production process in the melt shop and the rolling of thick plates; and in the detection and analysis of cracks and internal defects arising from the presence of hydrogen, to optimize and make economically viable the full production chain.

**Keywords:** Sour service; Ladle metallurgy; Plate rolling; Microstructural characterization.

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## 1 INTRODUCTION

The great challenge for using steel pipes in acidic environments, (sour service) is to establish criteria for the specification and production of steel, from the melt shop to the rolling mill, which are a priori economically viable and mitigate the formation of internal defects, especially hydrogen induced cracks (HIC) and stress corrosion cracking (SCC), as well the appearance of microstructurally hardened regions (hard spots) that induce the generation and propagation of these cracks, which can be catastrophic.
The alloy design development, which includes the definition of the chemical composition, the manufacturing process with its control items and tests for the production of heavy plates for the most demanding applications of API tubes has become an extremely complex task. Applications with strict safety requirements, such as offshore pipelines built on ocean beds, subjected to high external pressures, low temperatures, transporting fluids containing $CO_2$ and/or $H_2S$, require skilled designers to develop extremely strict technical specifications.
In this type of application it is common to use pipes with greater thickness/diameter ratios compared to on-shore pipelines, associated with minimum resistance levels of 65 to 70 ksi and an extensive list of requirements, such as chemical composition of ladle and product (individual restrictions, by sum and by relations between chemical elements), tensile strength (longitudinal and transversal), toughness (Charpy, DWTT, CTOD), hardness, microstructure (ferritic grain size, type and level of inclusions), mechanical properties after aging and after stress relief treatment, resistance to HIC, SCC, etc. In addition to these requirements, issues related to the behavior of the plate when manufacturing the tubes must also be considered during the development of the alloy design, such as formability and variation of properties between the original plate and the formed pipe.
Countless technologies have already been developed, based on studies and metallurgical insights of the phenomena involved and the operational practice in the facilities that manufacture plates and the corresponding pipes, as well on the technical observations made in the pipelines already implanted.
The occurrence of HIC and SCC has already been understood since its origin, with basic countermeasures already established. Currently, the great progress that is still expected is how to promote these countermeasures in an economically feasible way, on a large scale, with adequate performance in the production, inspection and laboratory evaluation stages. That is why there are few plants in the world that can reach an adequate level of supply, having heavy plates in their portfolio that meet the strict specifications for sour service application and have good profit from them.
Figure 1 [1] shows schematically the SCC formation mechanism that starts in the presence of hydrogen in the internal inhomogeneities of the heavy plates. Cracking occurs under the combined action of tensile stress and corrosion in the presence of water and hydrogen sulfide. It became noticeably clear that the first action to eliminate the appearance of HIC and SCC is to mitigate the formation of these defects. This can be achieved through specific techniques at each stage of the plate production process. There is a need to have absolute control of the process parameters in liquid steel refining, casting and rolling, as is shown schematically in figure 2 [2]. And, finally, it is necessary to have adequate procedures to reveal the presence of cracks in metallographic tests in a well-equipped testing center with skilled personnel. Figure 3 [3] shows how the factors of the environment where the

pipe is being used are correlated and the basic characteristics of steel in the appearance of corrosion cracks.
This work aims to show how cracking due to the presence of hydrogen (HIC and SCC) occur and the countermeasures that must be taken and managed throughout the plate production chain. A very recent topic which will be also discussed here is the occurrence of the so called "hard spots" in TMCP plates, which still requires more detailed studies.

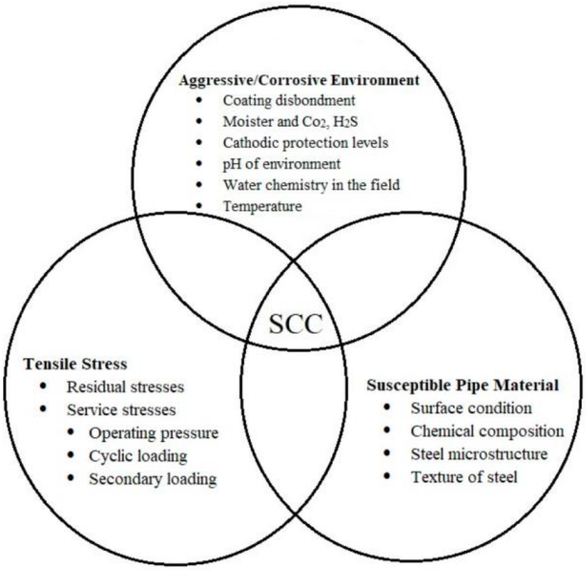



**Figure 1.** Factors that promote SCC in pipeline steels [1].

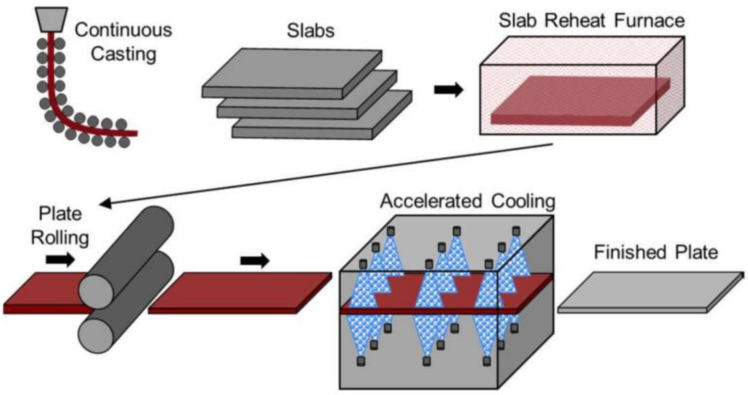



**Figure 2.** Schematic of thermomechanical controlled processing (TMCP) of plate [2].

## 2 PRODUCT SPECIFICATION: STRATEGY AND ALLOY ELEMENTS

The development of the alloy design of steels for sour service applications requires the definition of the restrictions of the external specification, possible variations in the contents of alloy elements in the steel slab (positive and negative segregations), the manufacturing processes to be used in the different stages (as well as its limitations) and the metallurgical strategy to be adopted during plate rolling, considering the specific microstructure morphology targeted.
Of the elements added intentionally, the most common are C, Mn, Si, Al, Nb and Ti. Elements such as V, Cr, Ni, Cu and Mo are also often used, depending on the

specification restrictions, plate rolling strategy and type of microstructure wanted. The choice of elements and their proportions must be made very carefully, because when certain limits are exceeded, the resulting microstructure may come to present characteristics that affect the diffusion of hydrogen, generating effects on the susceptibility to HIC. Additionally, issues related to the cost of ferroalloys must also be considered.

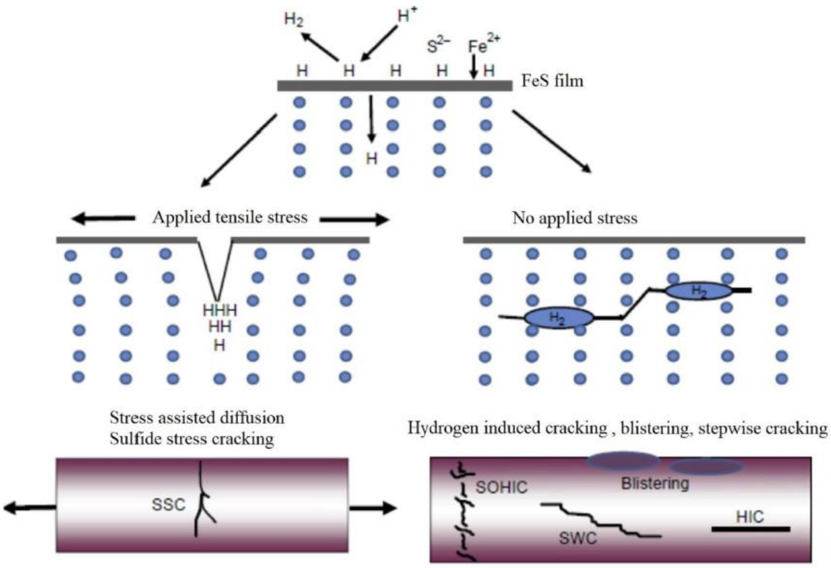



Figure 3. Formation of HIC and SSC cracks [3].

Impurities such as P and S must be kept at the lowest possible levels allowed by the limitations of the refining processes. Ca may or may not be added, depending on the external specification and the level of S targeted. The addition of CaSi is sometimes mandatory, regardless of the level of S.

Analyzing the chemical compositions that are being used in the current projects and evaluating the specific influences of each chemical element, together with the correlations of all the variables of each production process from the melt shop to the rolling mill in the mechanical properties and hydrogen cracking, it can be stated that there are optimal ranges for heavy plates produced with yield strength of 500 MPa or higher: C between 0.03 to 0.08%, Si less than 0.5%, Mn less than 1.5%, 0.3% Cu maximum, 0.3% Ni maximum, 0.1% Nb+V+Mo maximum, 0.005% S maximum, 0.005% P maximum and 0.005% O maximum. Figure 4 [4] shows the effect of some alloy elements over hydrogen cracking susceptibility.

The high atomic mobility of hydrogen in the iron lattice makes its interaction with the microstructure particularly complex. Microstructural features, such as non-metallic inclusions, precipitates, dislocations, micro voids, grain boundaries and the specific type of constituent present, reduce the mobility of hydrogen in steel, acting as traps.

Traps are classified as reversible or irreversible, depending on their hydrogen bonding energy. Reversible traps, e.g., grain boundaries, dislocations and micro voids, have low binding energy and does not permanently trap hydrogen, reducing the risk of this element to reach high levels in sites with stress concentration that can lead to the nucleation of microcracks. Many researchers consider reversible traps to be the most relevant factor in relation to susceptibility to hydrogen-induced cracking [3].

On the other hand, irreversible traps, such as non-metallic inclusions and precipitates, have high binding energies. Non-metallic inclusions, depending on their morphology, size, distribution and volumetric fraction, are, in many cases, the predominant factor in the fracture nucleation process. As for precipitates, coherent and semi-incoherent ones have low binding energies, while incoherent ones have high energies. Coarse precipitates, like those of cubic shaped TiN, act as microcrack nucleators. The presence of fine precipitates (smaller than 100 nm) is considered beneficial because they behave as innocuous traps that help to redistribute hydrogen at numerous points in the microstructure, contributing to improve resistance to HIC. Niobium is particularly useful to reduce HIC, as it not only creates strong irreversible hydrogen traps by the formation of copious NbC precipitates in the matrix, but also increase the density of reversible hydrogen traps by enhancing the proportion of low angle grain boundaries, resulting in a more uniform hydrogen distribution [5]. Figure 5 illustrates the various types of traps commonly found in steel [3].

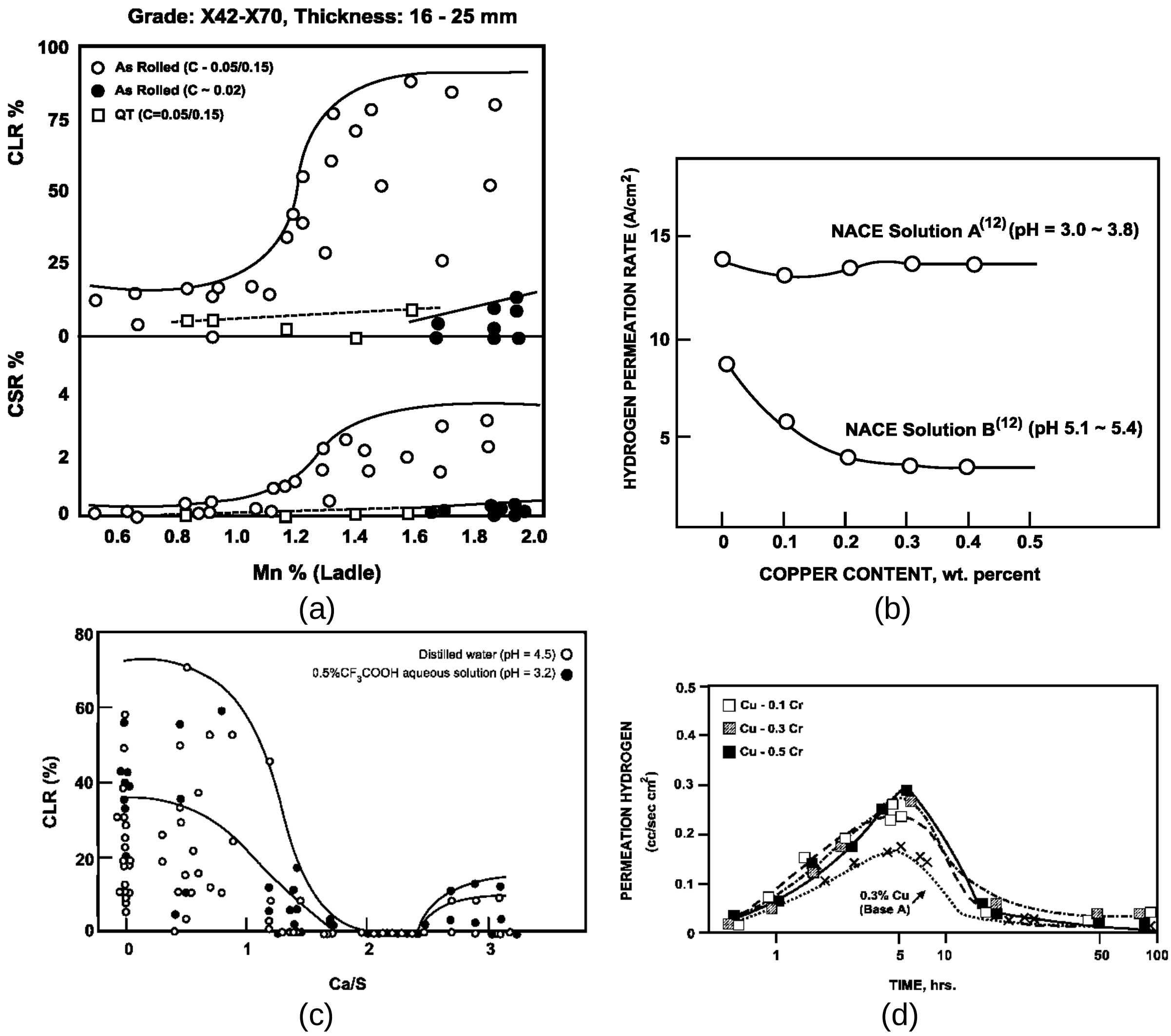



**Figure 4.** Effect of alloy elements on HIC: a) Mn; b) Cu; c) Ca/S ratio and d) Cr. CLR: crack length ratio; CSR: crack sensitivity ratio [4]. See Figure 7.

The most suitable microstructure depends on each case. However, it is common sense that a uniform microstructure, with a minimum of defects, is the most efficient way to increase resistance to hydrogen-assisted cracking. Microstructures with predominance of acicular ferrite or ultrafine ferrite tend to present the best results in terms of resistance to HIC and SCC [6]. It is believed that, since acicular ferrite has randomly oriented grain boundaries and high levels of dislocations, its efficiency in trapping hydrogen is superior to that of other microstructures. On the other hand, microstructures containing hard phases, such as martensite, bainite, MA (martensite-austenite) and ferrite/pearlite banding, should be avoided as they provide favorable sites for the initiation and propagation of cracks.

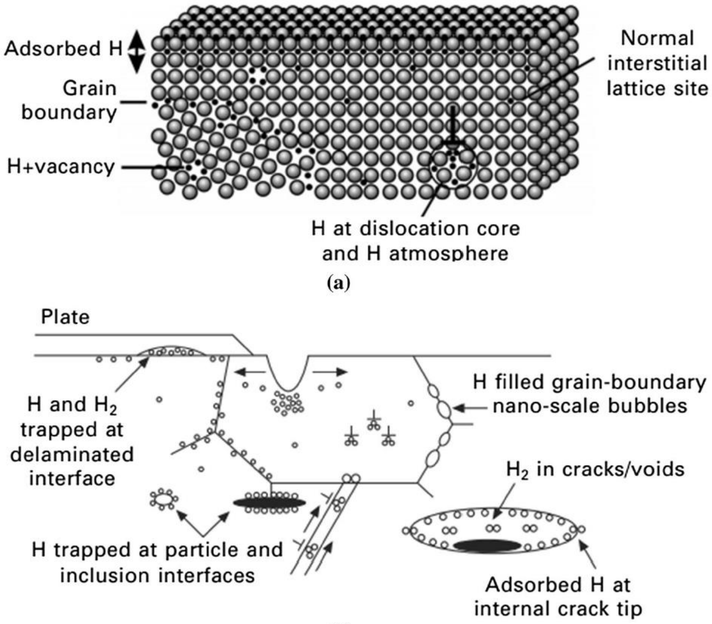



**Figure 5.** Trap sites for hydrogen in steels: a) on atomic scale; b) on microscopic scale [3].

The detrimental effect of high Mn contents for API 5L plates and pipes (e.g. X65MS and X70MS) on HIC performance used to be considered very tricky as quite high and risky Mn additions are needed to grant the strength levels that are required by the higher grades ones. As an innovative proposal, the Low Mn HTP alloy design is a promising solution to this tricky scenario, by substituting Mn additions by Nb microadditions [7].

## 3 LIQUID STEEL REFINING

The production route at the melt shop based on the sequence Kanbara Reactor (KR) – Basic Oxygen Furnace (BOF) with double slag - Ladle Furnace (LF) – RH vacuum degasser - Ca Injection – Continuous Casting with dynamic soft reduction proved to be highly efficient to produce highly clean sour service steels, with few and finely dispersed precipitates and inclusions, where there is a major suppression of sites for HIC, already in the coarse structure of the as-cast slab.
For optimal results, the production route in the steelworks must have as main goals: S content less than 10 ppm, P content less than 100 ppm, H content less than 1.8 ppm, N maximum content generally in the range between 40 and 50 ppm and total O less than 10 ppm (cleanliness) [4].
**Sulfur.** The final hot metal S content sent to the LD converters can even vary in a range of 300 to 900 ppm. The KR process proves to be the most efficient for hot metal ladle desulphurization (De-S), where levels between 5 to 10 ppm of S are the normal practice. To compensate the S pick-up, a second De-S step in ladle refining is

required to adjust the S content below 10 ppm. This is usually achieved in Ladle Furnace (LF), but not exclusively. For instance, after 50 minutes in the LF, values below 15 ppm can be obtained at the end of this treatment. To prevent S pick-up, it is necessary to strictly control the chemical composition of lime, dolomite, nepheline and other slag-forming additions and Fe-alloys, besides the suppression of slag carryover from the primary refining (BOF or FEA), i.e., use of a slag stopper system.

**Phosphor.** Despite the high rates of dephosphorization (De-P) during the blow at BOF (85 to 90%), this index is not sufficient to obtain consistent levels of P in the product below 100 ppm. Therefore, as in the case of De-S, it is necessary a hot metal dephosphorization process (De-P). The current pattern of De-P in greenfield plants (mainly in Asian countries), converges to the use of a BOF #1 (exclusive or not) for desiliconization (De-Si) and De-P, and another BOF #2 for decarburization (De-C). Values below 50 ppm of P are normally obtained. When this technology is not available, double slag techniques are used in conventional BOF steelworks, with the proper handling of the scorifiers and fluxes added during the blow, to achieve the largest possible P partition. To minimize the P pick-up (30 to 70 ppm), like S, it is also necessary to use an efficient slag retention technique.

**Hydrogen.** To obtain H values below 1.8 ppm, a value close to the thermodynamic equilibrium, the maintenance conditions of the vacuum equipment, for instance, the RH-OB, must be such that the leakage is suppressed, making it possible to sustain a vacuum pressure of the order of 2 to 4 mbar throughout the treatment.

**Nitrogen.** After tapping there will be a N pick-up in the order of 16 ppm until slab casting, considering the partial De-N of the RH-OB process. This pick-up is a direct indicator of the degree of reoxidation of the process, which indirectly measures the level of liquid steel cleanliness. Practical data indicates that to prevent the formation of edge crack in slabs, a Ti/N ratio below 3.0 is necessary; this condition would also, in general, meet the TiN precipitation ratio.

Sour service steels require a very well-conceived non-metallic inclusion engineering. Consolidated practice is to define a Ca/S ratio between 2.0 and 2.2 to prevent MnS and $Al_2O_3$ clusters precipitation during secondary refining processes, as shown in Figure 4c [4]. The greatest operational difficulty is to maintain a constant and reproducible content of Ca dissolved in the heat, due to the low solubility of this element in liquid Fe and the ease Ca vaporization and loss due to volatilization. One solution is the double treatment of Ca injection: the first CaSi injection, carried out in the Ladle Furnace, aims to modify the inclusions of $Al_2O_3$ in calcium aluminate, and the second injection, in the RH-OB degasser, has the objective of favoring CaS precipitation instead of MnS. Addition of Mg is a current trend to play the same role as Ca as a modifier for non-metallic inclusions. The formation of inclusions of high melting point of Mg oxide, sulfides and oxysulfides, with granulometry in the range of 10 to 100 nm, prevent the growth of austenitic grain ("pinning effect") and facilitate the nucleation of acicular ferrite (AF) in high energy welding processes. This effect is also reported for steels treated with zirconium and rare earths.

The liquid steel solidification during slab continuous casting also requires special care. Even with extremely low values of S in the steel, during the solidification process there will always be an increase in the concentration of Mn and S in the remaining liquid and consequent MnS precipitation tendency in the liquid during the solidification of the steel, that is, macro-segregation. This region is highly conducive to the generation of H traps, favoring HIC. The dynamic soft reduction technology during the continuous casting process of the slabs is a way to minimize this effect, by breaking the primary dendrites of the as-cast structure, reducing central segregation and preventing the generation of HIC sites. Coupled with this technology, recent studies with electromagnetic stirrers (EMS), in various sections of the solidification segments and also in the mold, demonstrate that it is possible to reduce the differences in overheat temperature of the liquid steel in the tundish, thus enabling the generation of several nuclei of heterogeneous precipitation, refining the coarse as-cast structure and minimizing macro segregation.

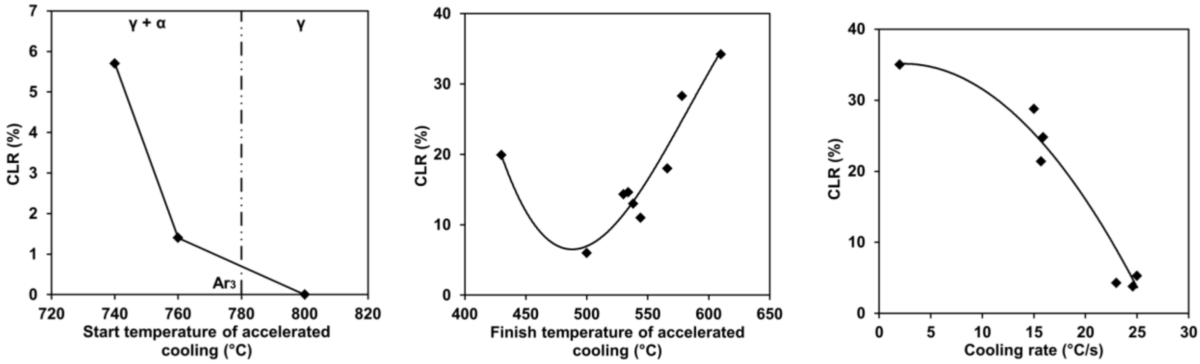
## 4 THERMOMECHANICAL CONTROLLED PROCESSING (TMCP)

When the application requires a combination of high strength (API X65 and X70, for example) with thick pipe wall thicknesses (above 30 mm), the chemical composition alone is insufficient to get the required strength level. In this case, it is necessary to increase strength by means of hardening mechanisms which can be promoted during the thermomechanical controlled processing (TMCP) of the plate.

The main objective of TMCP regarding HIC performance is to promote the formation of favorable microstructure morphologies and crystallographic textures which minimize the susceptibility to the appearance of cracks. To develop the plate rolling strategy that will lead to the required microstructure morphology it is necessary to understand the several interactions that can occur between chemical composition and hot rolling parameters. These interactions can be predicted through empirical equations that allow quantifying the influence of chemical elements on metallurgical parameters, such as critical rolling temperatures: dissolution of precipitates of microalloying elements (Ti, Nb, V, Al), recrystallization limit (RLT) and stop (RST) of austenite, non-recrystallization of austenite ($T_{nr}$), start of the austenite to ferrite transformation ($Ar_3$), start of formation of pearlite ($Ar_1$), start of bainitic transformation ($B_s$) and start of martensitic transformation ($M_s$). In addition, as the microstructure morphologies that enable the best results regarding HIC for these strength-thickness levels require accelerated cooling after rolling to be obtained, it is extremely important to know the continuous cooling transformation (CCT) diagrams of austenite for the chemical composition considered. This information is vital for the correct choice of the cooling rate and the start and end accelerated cooling temperatures that will generate the desired microstructure.

The microstructure most favorable to the increase the HIC resistance is predominantly ferritic, with smaller fractions of pearlite and bainite to promote the desired mechanical properties. Martensite and other hard constituents must be avoided, as they nucleate HIC. Some practical results indicate that a good HIC performance requires a minimum fraction of 60% of acicular ferrite in the microstructure, a constituent that, as already seen, is very favorable to restrict the occurrence of HIC [6].

It is generally recommended the adoption of lower finish rolling temperatures (FRT), as this yields a refined austenitic structure which is inherited by the ferrite that forms later. However, FRT must always be above $Ar_3$. Figure 6 shows the effect of some accelerated cooling parameters on HIC resistance of 20 mm low Mn microalloyed steel plates used for the manufacturing of API X65 pipes [8]. Figure 6(a) shows that best results regarding HIC resistance are achieved if accelerated cooling starts in a fully austenitic plate, as a more refined and uniform structure is obtained. For its turn, if accelerated cooling starts with some ferrite already formed in the microstructure, hard high carbon constituents are formed, which will promote the future nucleation of HIC. Lower temperatures of accelerated cooling finish are beneficial for HIC performance due to the formation of a progressively refined acicular ferrite and/or bainite. However, below a certain value, some austenite transforms in MA, a very hard microconstituent which intensifies HIC, as shown in figure 6(b). Finally, higher cooling rates, up to 25°C/s, refine and homogenize the final plate microstructure, as shown in figure 6(c). However, excessively high cooling rates can promote the formation of harder bainite and martensite, degrading HIC performance.




(a) (b) (c)

**Figure 6.** Effect of some parameters of accelerated cooling after plate rolling on HIC resistance [8]. CLR: crack length ratio, see Figure 7.

## 4 HARD SPOTS

It is very well known that the presence of Martensite and heavy deformed ferritic microstructures introduce a higher opportunity for hydrogen induced cracking of steels for sour service applications. As both metallurgical aspects are produced by specific thermomechanical processing conditions combined with segregation of C and other alloying elements, it is well accepted the need for very restrict control of the manufacturing conditions and the alloy design to avoid hydrogen induced cracking. Instead of checking for martensite using metallographic inspection, it is much more practical to use hardness limits (HV) as a criterion for qualifying a given steel plate or a large diameter pipe. Exogenous causes of martensite formation, as in the case of the hard spots, add new challenges to API 5L pipes for sour service purpose [8]. Finally, it is almost impossible to not to mention the potential effect of B residuals when talking about the risk of Martensite formation as very low content of this element is reported to increase the hardenability of steels. This increase on hardenability might not be in macroscopic scale, but it occurs in microscopic scale and are associated to undesirable brittle paths for hydrogen induced cracks and brittle fracture when the material is submitted to impact testing such as DWTT and CVN.

Until recently, it was believed that the occurrence of hard spots was restricted to pipes produced before 1960, having occurred due to specific manufacturing failures that happened in a few American plants. So much so that it has never been usual practice to carry out massive measurements of hardness in heavy plates for the manufacture of pipes. However, this problem came back to a dramatic extent in 2014, after a series of leaks occurred in a newly built gas pipeline in the Kashagan field, near the Caspian Sea. The origin of these cracks was attributed to the stress cracking induced by sulfide in tiny areas of the pipe, with few tenths of a millimeter in depth, which unexpectedly showed high values of hardness, both near welded joints and in the body of the pipe [8].

The complete novelty and the lack of concrete information about this new defect threatened to promote a retrocession in the specifications of materials for large diameter pipes, and there have even been proposals to re-use steels with lower levels of mechanical strength, such as API X52, and hardness levels below 220 HV. However, the consequent increase in the thickness of the pipe wall that would be necessary in this case would make it difficult to meet the high toughness and sour service requirements typical of this application [9].

The tiny size of the hard spots and the subsurface character make it extremely difficult to identify, requiring a full inspection of the heavy plates used in the manufacture of the pipes, like what already occurs in the online ultrasound tests applied to ensure its internal soundness. The Dillinger plant, in Germany, a benchmark for plate technology, soon installed an automatic system in its plate finishing area that allows the determination of plate hardness in a massive, quick and non-destructive way, using a magnetic method based on Eddy currents [9].

Subsequent research conducted by oil companies, the biggest interested in clarifying this issue, pointed out several causes for the hard spot. One of these would be the localized contamination of the liquid steel by the carbon present in the flux powder used in the continuous casting of the slabs. Another very intriguing possibility is based on the so-called “cooling paradox”, a phenomenon that has long been known by Japanese artisans who made samurai swords. They found that regions of the blade with thermally insulating coating may, interestingly, have higher local rates of cooling under the action of water compared to regions where the coating was not used. Therefore, small local heterogeneities in the thickness of the insulating scale (iron oxide) on the plate can lead to large variations in the local cooling rates, promoting the formation of superficially hardened spots where the scale was thicker. This raises the possibility of identifying the regions of a plate that could show hard spots from the thermal image of its upper surface, determined immediately after its accelerated cooling [2].

But why is the hard spot issue just getting attention? First, since it is a tiny, subsurface hardened region, it is practically impossible to detect these defects using conventional mechanical hardness tests. Therefore, this defect may have gone unnoticed over the decades of manufacturing large diameter pipes. This defect appears to be harmful only in cases where the hydrocarbon fluid being conducted by the pipe promotes an extremely aggressive attack by $H_2S$, which is relatively rare today, but may become more common in the future, with the aging of oil fields. It is interesting to note that several customers are looking to see if it is possible to live with this defect, determining the minimum level of toughness necessary so that a crack eventually nucleated in a hard spot is captured by the steel and, thus, does not offer risk to the integrity of the pipe 10].

## 5 LABORATORY CHARACTERIZATION OF HIC

The evaluation of pipeline steels for resistance to HIC is generally performed in the laboratory according to the NACE Standard TM 0284. The test method consists of exposing unstressed test specimens to one of the two standard solutions saturated with $H_2S$ at ambient temperature and pressure. After a specific period the test specimens are removed and evaluated. Each of them is sectioned according to the specifications of this standard and three surfaces examined metallographically for determination of the Crack Sensitivity Ratio (CSR), Crack Length Ratio (CLR) and Crack Thickness Ratio (CTR). They are calculated on basis of crack measurement in the focusing screen of a light microscope at a magnification of 100 x, as shown in Figure 7 [11,12].

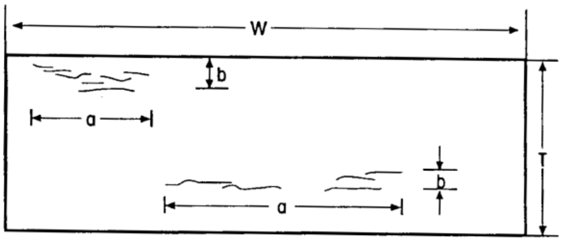



**Figure 7.** Test specimen and crack dimensions to be used for the calculation of CSR, CLR and CTR [12].

The formulas for calculation of these parameters are described below:

$$CSR = \frac{\Sigma\,(a\,.\,b)}{(W\,.\,T)}\,.100\% \qquad (1)$$

$$CLR = \frac{\Sigma\,a}{W}\,.100\% \qquad (2)$$

$$CTR = \frac{\Sigma\,b}{T}\,.100\% \qquad (3)$$

where **a** is crack length, **b** is crack thickness, **W** is section width and **T** is test specimen thickness. Examples of typical cracks induced by H can be seen in Figure 8 [11].

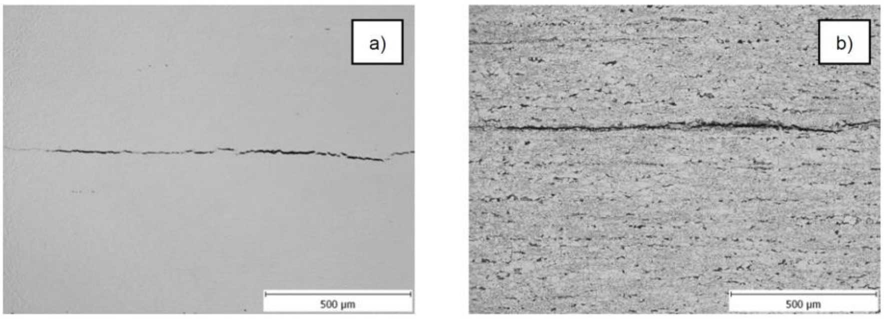



**Figure 7.** Typical cracks induced by H seen in the metallographic specimens of steel plates for pipes [11].

Usually, top level studies do prefer a more precise approach to get a more accurate understanding of the performance of plate or pipe for sour service purpose. One of the most common methodologies available to characterize hydrogen induced cracks is to measure the CAR (crack area ratio) of tested coupons. In this case, metallographic analysis approach is replaced by ultrasonic inspection based on C scan methodology, so the ratio of the cracked area gives a good complement to the other ones, CLR, CTR, and CSR. It is considered a faster alternative to the metallographic method [11]. Following the same goal that returns a more accurate understanding of the HIC performance, the Full Ring Test is a methodology that increase the sample scale to support the detection of abnormal HIC events in addition to the view offered by standard test specimen [13].

## 6 CONCLUSION

This paper sought to show, in a practical and objective way, the personal opinion of the authors on the main challenges involved in the design, manufacture and characterization of heavy plates made with microalloyed steel used in the manufacture of pipes for sour service. It is an application whose requirements for uniformity and performance have become increasingly severe over the past few decades, which is making its compliance increasingly challenging from an economic and technical point of view. After all, it is about extracting the best possible performance from very economical steels, fairly alloyed, which has been increasingly restricting the process windows applicable in the areas of steel refining and hot rolling. The most modern metallurgical simulation and characterization resources are being used to fulfill this complex task.

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