

# **PROCESS AND QUALITY CONTROLS FOR PRODUCTION OF LINEPIPE SLABS FOR SOUR SERVICE APPLICATIONS AT ARCELORMITTAL LAZARO CARDENAS, MEXICO**

J. Nieto<sup>1</sup>, T. Elías<sup>1</sup>, G. López<sup>1</sup>, G. Campos<sup>1</sup>, F. López<sup>1</sup> and A.K. De<sup>2</sup>

<sup>1</sup>ArcelorMittal Lázaro Cárdenas, 26113 Lázaro Cárdenas, Michoacan, Mexico

<sup>2</sup>ArcelorMittal Global R&D, 3001 E Columbus Dr, E Chicago, IN 46312, USA

Keywords: Segregation, Soft Reduction, HIC, Linepipe, Slab Casting, Hydrogen, SSCC, X65, Steelmaking, Composition, NACE, Inclusions

## **Abstract**

There has been an increased interest in the requirements of API grade linepipe steels for the transportation of crude oil or sour gas. In addition, several recent pipeline projects have more stringent acceptance criteria than API. Successful production of sour service pipes depends to a large extent on the internal quality of the primary slabs in order to guarantee excellent cleanliness from non-metallic inclusions and least possible centerline segregation and porosity. Additionally, the steel slab also needs to contain the minimum possible dissolved hydrogen gas content for effective resistance to hydrogen flaking in the skelp and hydrogen induced cracking (HIC) in the final product. ArcelorMittal Lázaro Cárdenas (AMLC) in Mexico has implemented key steelmaking and casting technologies to cater for the challenges of sour service slab making. Key process controls and effective design of steelmaking and casting technologies have been introduced for the production of ultra clean, sound slabs with the least dissolved hydrogen content. Significant tonnages of sour service slabs have been made at AMLC and supplied to customers worldwide for making HIC resistant skelp and pipes. Excellent HIC resistance has been reported in all forms of final products thus qualifying AMLC's capability to produce quality slabs for sour service applications. The current paper discusses some of the salient features of steelmaking and casting processes at AMLC along with some HIC test results from the final products.

## **Introduction**

With increased interest in tapping sour gas-containing crude oil/natural gas reserves, demand for sour service linepipe steels has been growing worldwide. Unlike steels for sweet service, oil and gas pipeline steels for transportation of sour gas or oil require stringent control over internal quality for resistance against hydrogen induced cracking. Attack from hydrogen can be both environmentally assisted during service and internal, ie. metallurgical [1]. In order to guarantee excellent resistance to hydrogen induced cracking from both sources of hydrogen, linepipe steels should have minimum possible microscopic or macroscopic discontinuities that can attract atomic hydrogen, besides having minimum dissolved hydrogen content. Internal traps are commonly (i) centerline segregation, (ii) shrinkage cavities and (iii) inclusion-matrix interfaces [2-4].

Centerline segregation, caused mainly by segregation–active elements such as carbon, manganese, sulfur, phosphorus and oxygen [5], gives rise to hard transformation products at the mid-thickness of finished products thus providing an easy path for stepwise hydrogen induced cracking [4,6]. Centerline segregation is also primarily responsible for centerline porosity in continuously cast slabs [5]. These porosities are easy traps for hydrogen that gets dissolved in the steel during the steelmaking process. When excessive hydrogen accumulates, porosities will be difficult to annihilate during hot rolling and will exert high pressure resulting in delayed cracking in rolled products [4]. Both effective compositional design and continuous casting process controls are key to avoiding centerline segregation and shrinkage cavities in sour service slabs.

Non-metallic inclusion control is perhaps the most important consideration for production of HIC resistant steels. Non-metallic inclusions such as oxides and sulfides should be minimized with strict steelmaking and casting process controls. Oxide and sulfide inclusions remaining in the melt have to be very fine in size and globularized through steelmaking controls. Globular inclusions do not pose a risk with regard to hydrogen pressure development at the interface but the size needs to be very fine so as to be contained mostly within the grains [3]. Elongated MnS inclusions act as stress raisers in the matrix and provide micro-cavities at interfaces which attract atomic hydrogen [1,4,6]. Hence the steel sulfur content needs to be kept very low (preferably 10 ppm or less) and the shape of any MnS inclusions modified through Ca-treatment.

Dissolved hydrogen, deriving from steelmaking and casting processes, needs to be minimized with intelligent process control and slabs should be slowly cooled to allow hydrogen diffusion out of the center of the slabs. It is reported that a final hydrogen content of 2 ppm and less is preferred for effective HIC resistance in thick gauge linepipe plates [4].

This paper discusses state of the art steelmaking and casting practices adopted in the AMLC Steel Mill for the production of HIC resistant steel slabs for linepipe and offshore storage pressure vessel applications.

## **Technology of Production of HIC Resistant Slabs at AMLC, Mexico**

### **Steelmaking**

Key technological infrastructures were implemented at AMLC, Mexico for the production of prime quality slabs for advanced critical applications. Figure 1 shows schematically key steelmaking and casting unit installations [7]. Based on demands from plate, HR coil and pipe makers in North America, Europe and Asia, AMLC has embarked on the production of slabs for sour service application grades such as API X52 and X65 since September 2011. Significant tonnages of API X52 and X65 grade slabs have been cast for processing into linepipe HR coils, plates and subsequently to ERW and LSAW pipes. A typical chemistry of cast slabs is given in Table I.

During steelmaking at AMLC, the following route was used: Electric Arc Furnace (EAF) - Ladle Furnace (LF) – RH Vacuum Degassing (VD) - Continuous Casting (CC). AMLC uses 100% direct reduced iron (DRI) as its primary metallic charge for EAF steelmaking and is produced through MIDREX and HYL-III processes. Use of DRI ensured virtually residual-free input for steelmaking and hence, a guaranteed high level of cleanliness [7]. At the ladle furnace (LF), the first phase of Ar-bubbling was accompanied by Fe-alloy and aluminum additions followed by a lime addition for slag making and sulfur control. Towards the end, when oxygen activity was brought to a minimum, the liquid melt was treated with metallic calcium in the form of steel clad wire along with a gentle rinsing with Ar. This treatment was helpful in producing very fine globular Ca-aluminate and modified Ca or Ca-Mn sulfides and easy flotation to the surface. The ratio of Ca/S was maintained within the range 2 to 4 for guaranteeing effective shape control and alloy-hardening of MnS by Ca.

Table I. Typical Chemical Composition of Sour Service Cast Slabs, wt.%

| Grade   | C         | Mn max | P max | S max | Si max | Al          | Ti max | Nb max | N           | Ca/S | Others        |
|---------|-----------|--------|-------|-------|--------|-------------|--------|--------|-------------|------|---------------|
| API X52 | 0.02-0.04 | 1.00   | 0.012 | 0.001 | 0.25   | 0.025-0.045 | 0.02   | 0.05   | 0.003-0.006 | 2-4  | Ni            |
| API X65 | 0.04-0.06 | 1.35   | 0.012 | 0.001 | 0.15   | 0.025-0.045 | 0.02   | 0.07   | 0.003-0.006 | 2-4  | Cr, Cu, Mo, V |

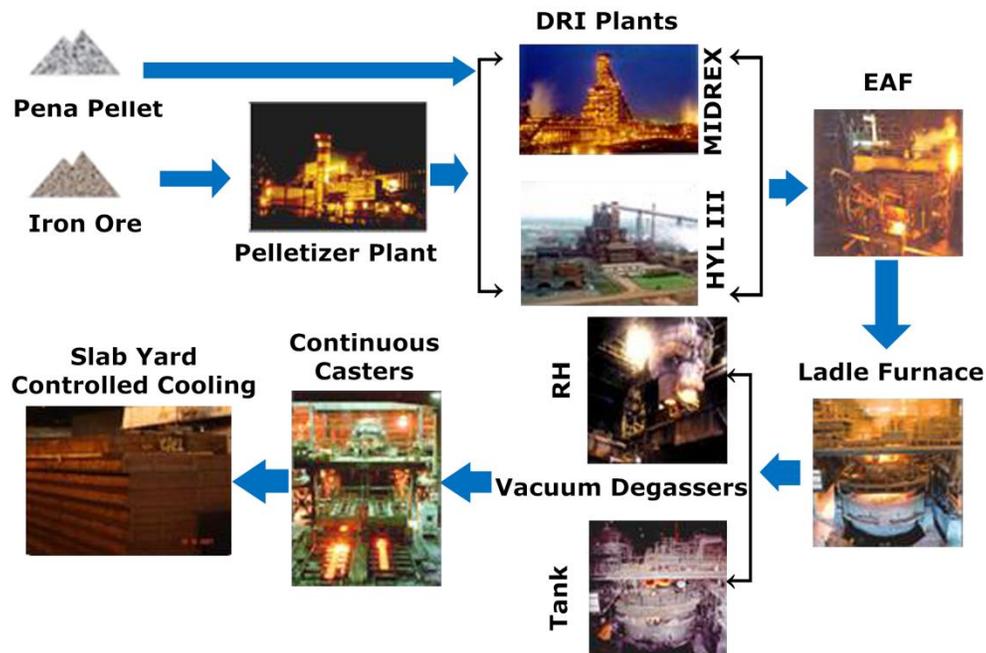


Figure 1. Steelmaking and casting process flow at AMLC for the production of HIC resistant slabs.

After ladle treatment and ensuring chemistry control, the steel is taken to an RH vacuum degasser for nitrogen and hydrogen control. Though AMLC has the option to use either the RH or tank degasser, it was found from a previous study that RH degassing produces better and faster removal of hydrogen from the melt, as indicated in Figure 2. This is probably because a greater volume of steel, for a specific period of time, is exposed to vacuum through the RH degasser than with the tank degasser. Tank degassing is also a slower process and demands tighter temperature control which is not advantageous for sour service slab casting where a stringent control over superheat is required.

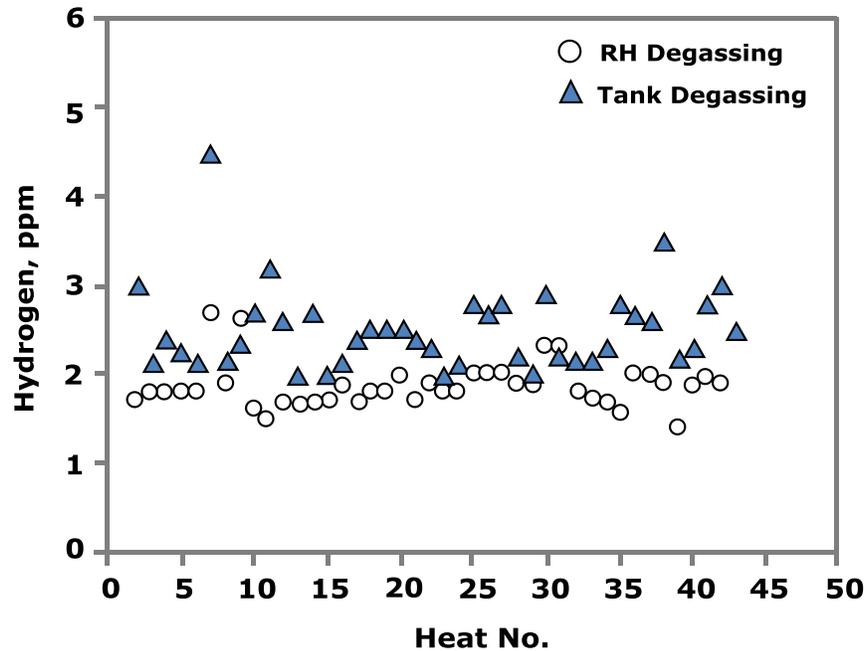


Figure 2. Hydrogen removal in linepipe steels during degassing at RH and Tank degasser.

Continuous Casting: Segregation Control

At the continuous casting station an argon-circulated ladle shroud ring is used between ladle and tundish for protection against air and thus hydrogen. A preheated (>900 °C) tundish (40 ton capacity) and submerged entry nozzles (SEN) were used to reduce hydrogen intake during pouring. Tundish design was improved to facilitate inclusion flotation and reduce skull formation thus guaranteeing steel cleanliness.

One important aim of current sour slab casting is to minimize centerline segregation. Since the chemistry (Table I) is low in C and Mn, centerline macro segregation is not as much of a concern as shrinkage cavities. Casting processes are therefore critically controlled to eliminate shrinkage cavities using a low superheat (<25 °C) in the tundish and a specific machine taper pattern that synchronizes the precise shrinkage of the solidifying slabs to reduce centerline cracks and segregation [5,8].

Identification of the solidification point within the caster and shrinkage correction is provided automatically by soft reduction technology powered by a Dynamic Solidification Control [8] (DSC) Model<sup>®</sup>. Soft reduction is applied at the rate of 0.2 to 0.8 mm/m. Typically, a total of 3-6 mm soft reduction is used. The full body rolls in the soft reduction zone are replaced by split rolls to reduce stress on the rolls and roll deflection thus reducing bulging of the strand, which is a major cause of centerline segregation. A 14-point unbending segment is used to reduce solid-liquid interface strain, thus reducing the unbending force, in turn, facilitating additional forces of soft reduction in this segment which allows better control over solidification shrinkage and hence, internal soundness [8,9].

### Slab Conditioning after Casting

Figure 3 shows the hydrogen content of heats as measured in tundish samples. The distribution shows that most of the heats have hydrogen contents of less than 3 ppm. Compared with hydrogen measured immediately after the RH station (Figure 2), it can be seen that there is a slight pickup of hydrogen in the tundish. Noting the fact that a steel melt is likely to pick up more hydrogen in the continuous casting area, care is taken to ensure use of moisture-dry mold and casting powder, preheated tundish and SEN. Furthermore, slabs meant for sour service applications are taken to a special controlled cooling enclosure for slow cooling after casting to allow further diffusing out of dissolved hydrogen.

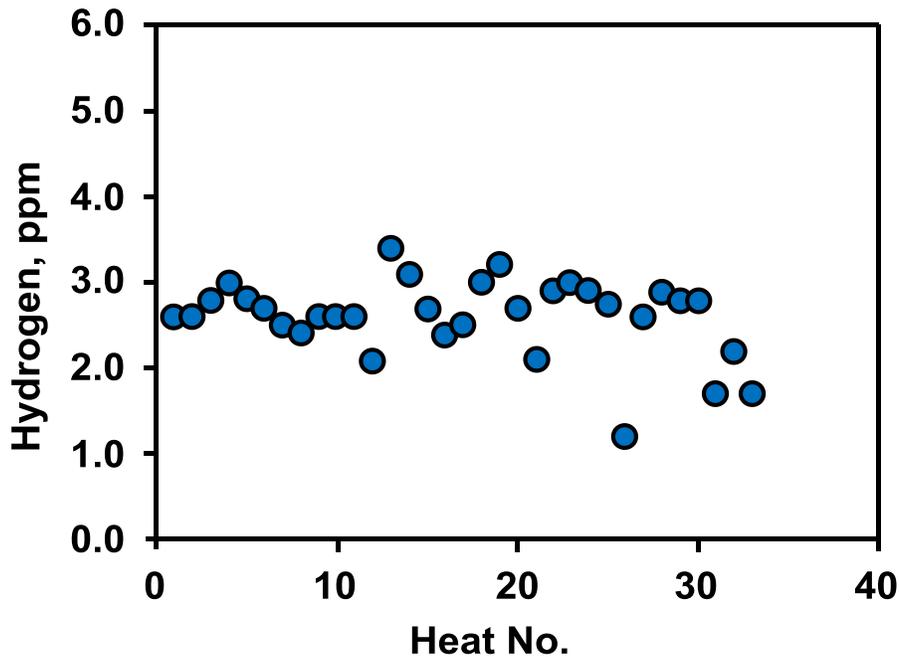


Figure 3. Hydrogen content measured in Hydris samples at tundish for linepipe grade heats.

This slow cooling area at AMLC is isolated from other areas with two meter high insulated walls on all sides. Hot slabs, in batches of six, are placed on an insulated slab and covered on the top by another insulated slab as shown in Figure 4. The stacks of these linepipe quality slabs are also surrounded by hot slabs of other grades. The cooling rate of slabs can be brought down to 5-6 °C/hr by this process and this is effective in diffusing out part of the dissolved hydrogen from the slab centerline.



Figure 4. Controlled slow cooling of slabs for hydrogen removal.

## Results

### Slab Macrostructure and Integrity

The slab dimensions are typically 250 mm thick x 1610-1900 mm width x 8000 mm long. Slab internal quality is examined through macroetching, using 30% HCl, in sections transverse to the casting direction and also along the longitudinal casting direction. The centerline quality is assessed using the Mannesmann slab internal quality rating system [10]. Figure 5 shows typical macrographs of macroetched transverse sections (transverse to casting direction) and longitudinal sections of cast linepipe grade slab indicating no apparent shrinkage cavities, dark spots or centerline segregation, which can be qualified as a Mannesmann rating of 1 or better. It is also seen that the columnar grains extend almost to the center of the slabs with no equiaxed zone at the slab center. The sound internal macrostructure suggests effective soft reduction control during continuous casting.

Chemical segregation is further checked with chemical analysis of drillings taken from through thickness sections of macroetched slabs at intervals of 5 mm. Internal soundness of the slabs is verified by ultrasonic testing of all macroetched slabs over the full length. A full body ultrasound scan of the slab did not reveal any internal inhomogeneities and thereby validated internal soundness as indicated by the macrographs in Figure 5. Chemical analyses of drillings from the macroetched slabs at different locations through the thickness are presented in Figure 6 and show no discernible segregation at the center of slab.

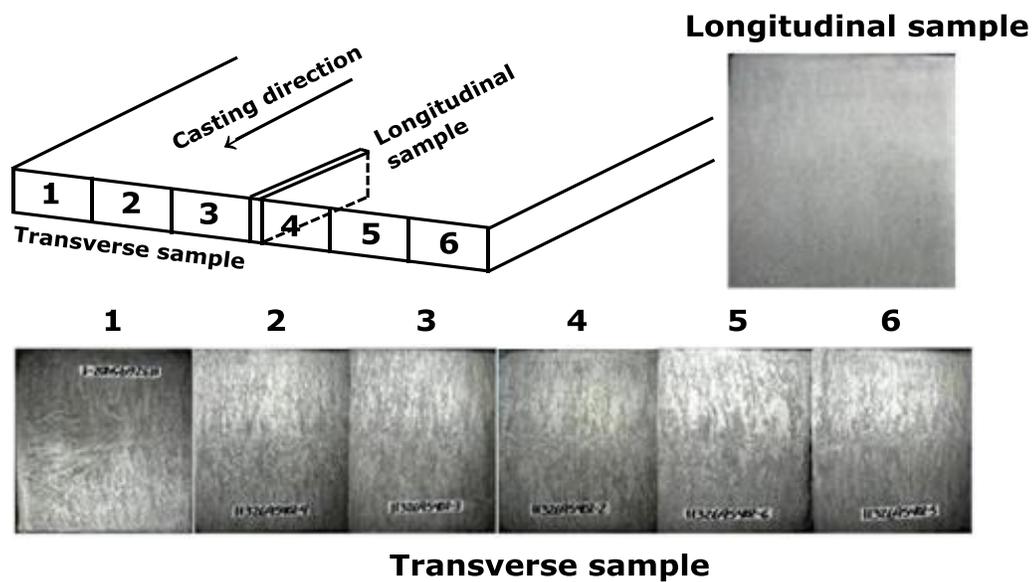


Figure 5. Macrograph of full width slab sample after etching, revealing surface to center columnar cast structure with no indication of centerline porosity or segregation.

| C     | Si    | Mn    | P     | S      |
|-------|-------|-------|-------|--------|
| 0.057 | 0.240 | 1.320 | 0.010 | 0.0005 |
| 0.056 | 0.250 | 1.310 | 0.010 | 0.0005 |
| 0.057 | 0.250 | 1.300 | 0.010 | 0.0006 |
| 0.058 | 0.256 | 1.290 | 0.010 | 0.0005 |
| 0.056 | 0.246 | 1.290 | 0.009 | 0.0004 |
| 0.055 | 0.249 | 1.330 | 0.010 | 0.0006 |
| 0.053 | 0.244 | 1.320 | 0.009 | 0.0007 |
| 0.054 | 0.248 | 1.320 | 0.009 | 0.0009 |
| 0.057 | 0.253 | 1.300 | 0.009 | 0.0008 |
| 0.056 | 0.254 | 1.300 | 0.009 | 0.0008 |

Figure 6. Chemical analysis of samples taken at various locations along through thickness section of macroetched X65 grade slab, wt.%.

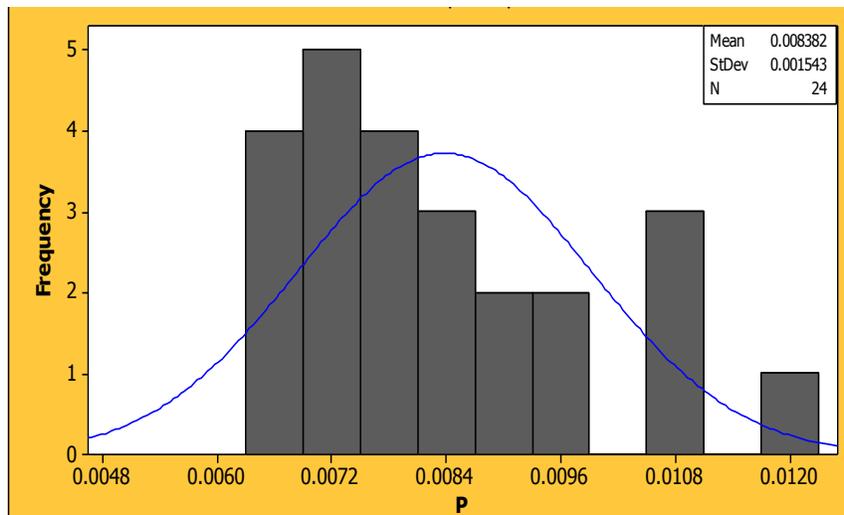
## Chemistry Control

Figure 7 shows the level of P, S, N and H control that can be achieved in X65 HIC grade heats. Most of the heats have very low levels of S (0.001 wt.% and less) and P (0.012 wt.% and less) and gas contents which are important in guaranteeing HIC resistance and toughness of linepipe steels. The low levels of S and P also guarantee a clean centerline condition in the cast slabs. Use of 100% DRI and ladle metallurgy process controls are facilitators in achieving such levels of purity in the sour service slabs.

## Processing of Slabs at Customers Mill

Slabs are processed at customer mills for making 6.25 mm thick HR coils and thick gauge plates up to 20 mm. The hot rolled coils and plates are further processed into ERW pipes and LSAW pipes respectively. Details of the plate and pipe processing are the subject of a separate publication but noteworthy results can be mentioned as follows:

- The plate centerline microstructure mirrored what is found at the slab centerline and did not indicate the presence of any significant chemical segregation, as seen in Figure 8;
- Plate microstructure was also examined for cleanliness in terms of non-metallic inclusions and exhibited excellent inclusion shape and size control. A typical microstructure of a hot rolled product from sour service slab is presented in Figure 9 showing globular oxy-sulfides and fine oxide inclusions. Sulfide inclusions are mainly fine globular (Ca,Mn)S type and hence remained undeformable during hot rolling.



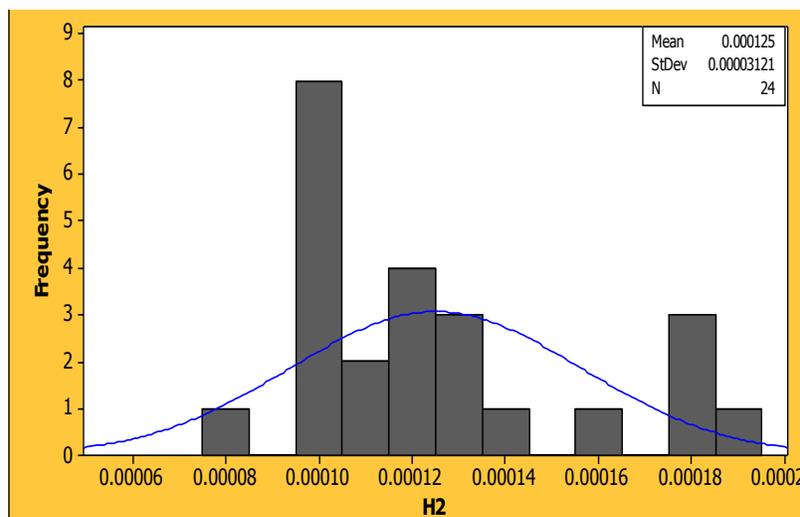
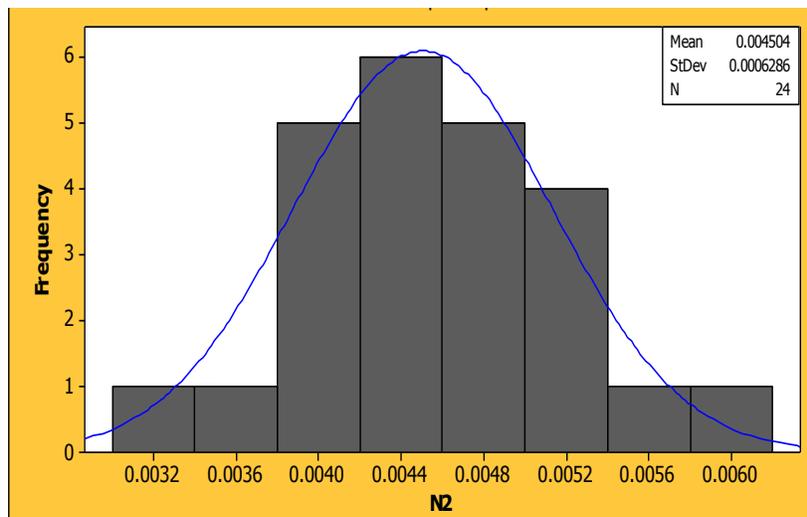
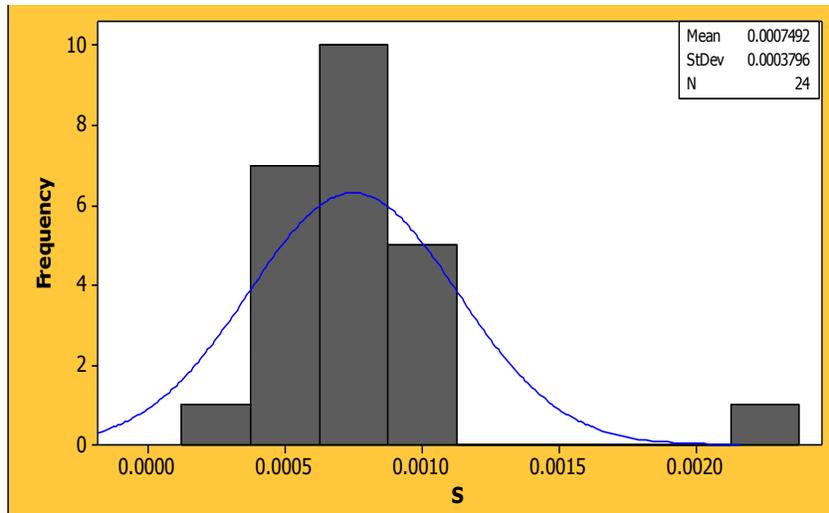


Figure 7. Chemistry controls (P, S, N, H) achieved in some of the X65 grade HIC heats cast at AMLC against a particular commercial order.

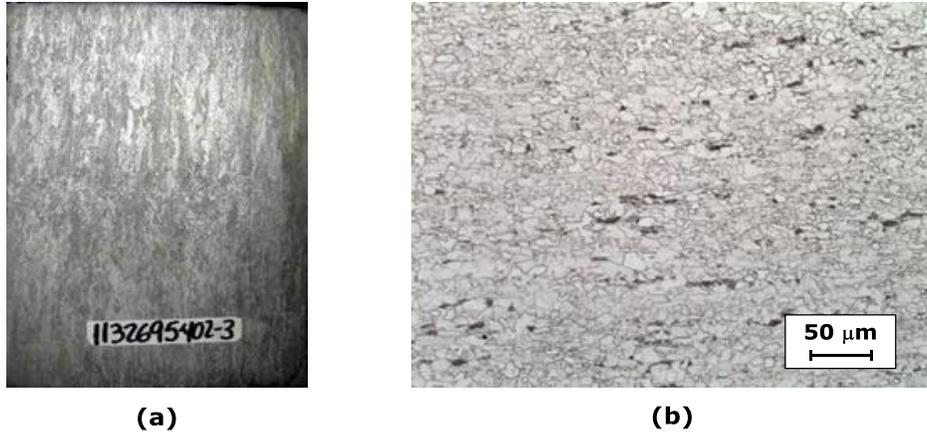


Figure 8. Correspondence of API X65 HIC grade; (a) slab centerline and (b) final pipe centerline microstructure revealing no centerline segregation.

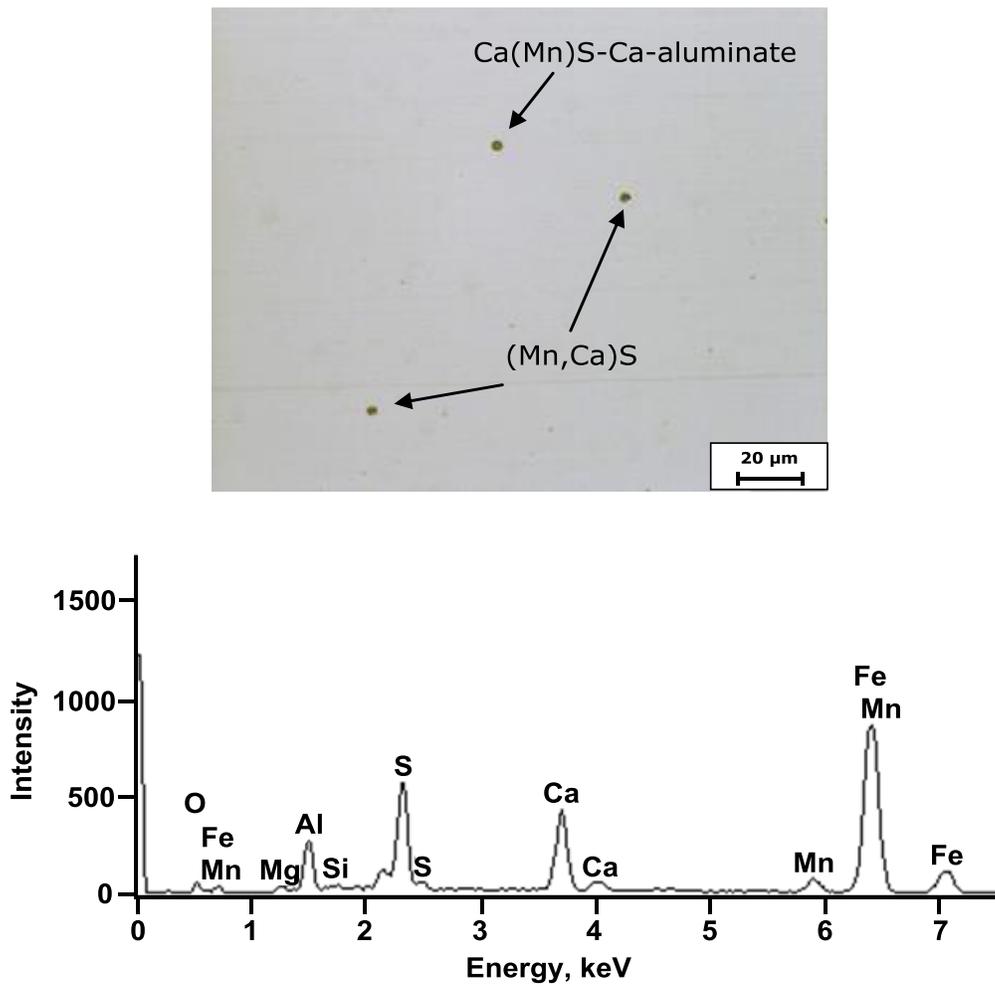


Figure 9. Typical inclusion contents in linepipe grade heats. Sulfides are effectively Ca-treated for shape control.

## HIC and SSCC Test Results

HIC tests in Solution A as per NACE standard (TM 0284-2003) [11] were performed on API X52 and X65 grade pipes made from HR coils and plates and indicated 0% CLR, CTR and CSR in all samples. SSCC tests (TM 0177-1996) [12] were also successful in both thin gauge and thick gauge pipes up to 20 mm wall thickness.

The successful performance of pipes in HIC and SSCC tests suggests effective process and quality controls at AMLC for the production of slabs for sour service applications requiring excellent resistance against hydrogen induced cracking.

## **Conclusions**

AMLC Mexico has implemented key technological improvements in its steelmaking unit for the production of sour service slabs for linepipe and offshore applications. Through effective process and quality control measures together with an inhouse developed controlled cooling system for hot slabs, significant tonnages of linepipe quality slabs have been produced which demonstrated excellent HIC resistance in the final product after processing on Customers mills. Deep macro etching and ultrasonic testing of slabs revealed excellent internal quality suitable for HIC resistant coil or plate production. Trial slabs of various linepipe grades were processed on customer's mills for making HR coils, plates and pipes. API X65 grade plates processed up to 20 mm thickness revealed no internal cracks after hot rolling during online ultrasonic testing. HIC testing of 6 mm thick coils, 20 mm thick plate and pipe samples tested in Solution A as per NACE TM 0284 revealed 0% CLR, CTR and CSR, which further validated internal soundness and excellent centerline qualities of AMLC slabs and the effectiveness of process and quality controls for the production of sour service slabs.

## **Acknowledgements**

The authors sincerely acknowledge invaluable support and continuous encouragement of Jose Fernandez, COO, and Santiago Neaves, CTO, AMLC during the entire development process and for permission to publish this article. Outstanding technical support from AM USA Global R&D is also greatly appreciated. Technical discussion with Murali Manohar, Dan Kruse, AM Global R&D, E Chicago has been immensely helpful.

## **References**

1. C.G. Interrante, "Basic Aspects of the Problems of Hydrogen in Steel," *Current Solutions to Hydrogen Problems in Steel*, ed. C.G. Interrante and G.M. Pressouyre, (Materials Park, OH: American Society for Metals, 1982), 3.
2. Sour Gas Resistant Pipe Steel, Niobium Information, 18, 2001.
3. G.M. Pressouyre, "Hydrogen Problems in Steels," *Proceedings of the First International Conference*, ed. C.G. Interrante and G.M. Pressouyre, (Materials Park, OH: American Society for Metals, 1982), 18.

4. Y. Tomita et al., "Hydrogen Problems in Steels," *Proceedings of the First International Conference*, ed. C.G. Interrante and G.M. Pressouyre, (Materials Park, OH: American Society for Metals, 1982), 63.
5. A. Ghosh, "Segregation in Cast Products," *Sadhana*, 26 (2001), 5-24.
6. D.G. Stalheim and B. Hoh, "Guidelines for Production of API Pipelines Steels Suitable for Hydrogen Induced Cracking (HIC) Service Applications," *Proceedings of IPC 2010, 8<sup>th</sup> International Pipeline Conference*, (September-October 2010), Alberta, Canada.
7. H.T. Tsai and R. Torres, "Process Improvements at ISMAT Mexicana to Supply Quality Slabs for the World Market - Third International Conference on Continuous Casting of Steel in Developing Countries, Beijing," *Journal of Iron and Steel Research International*, 39 (2004), 54.
8. J. Nieto et al., "Development of Technology for the Production of HIC Resistant Slabs for Sour Service Applications at ArcelorMittal Lazaro Cardenas, Mexico," *Proceedings of 4<sup>th</sup> Congress of National Steelmaking Conference*, Association for Iron and Steel Technology (AIST) Mexico, Monterrey, October (2010).
9. D.M. Humes, "Dynamic Soft Reduction of Continuously Cast Slabs," *Iron and Steel Technology*, July (2008), 29.
10. Mannesmann Rating System for Internal Defects in CC Slabs: April (2001), PTS, Germany.
11. NACE Standard TM 0284-03, 2003.
12. NACE Standard TM 0177-96, 1996.