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Optimizing ladle-refining performance during treating special steel melts for aviation technology

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Abstract. The ever increasing demands of consumers towards superior quality steel products urged steelmakers to consider the implementation of various relatively new metallurgical technologies into the classical operations. The necessity to produce high quality steels led to the use of two stage steel production processes, one is the primary steelmaking and the other stage is the outside furnace treatment through which many metallurgical functions can be achieved like degassing, stirring and inclusion removal, Inclusion modification, desulphurization, deoxidation, decarburization, heating and alloying. In this research a trial was done to optimize the performance and usage of 30tons-ladle refining system during production of X65-pipeline steel as final product-Aluminium killed steel melts through controlling the mass flow contour using optimized modelling, optimum usage of Al₂O₃ and Sulphide modifiers and enhancing removal of non-metallic inclusions by altering their morphologies and hence floatation speed. Assessment of fine clusters of inclusions in the final steel product has been industrially correlated to the cleanliness of melt before refining, to the slag composition and to the parameters of materials flow rates as well as their effects on the mechanical properties of final X65-steel product. Scanning-EM+DX analyzing unit and metallurgical microscopes were used to emphasis qualitatively and quantitatively the characters of non-metallic inclusions.

1. Introduction

The cleanliness of different steels is governed by numerous standards; however, it differs according to the applications of the final product. Products having minor cross-sectional dimension, such as thin strip steel, where the presence of critically sized inclusions has increasing effects on product performance. Inclusion particle size distribution is frequently used to provide characteristic description of steel cleanliness. A comprehensive cleanliness evaluation also requires complementary information on non-metallic inclusions found such as quantity, spatial distribution, type, and morphology. With the growing demand for high quality materials, the cleanliness of different steel types has become very important [1-3]. High strength low alloy (HSLA) steels offer improved weldability, and superior mechanical and corrosion-resistant properties compared to mild or low carbon steel at a minor price premium Load-bearing HSLA steel found in structural applications is susceptible to fatigue failure initiated by non-metallic inclusions. [4-6]

The great challenge faced by steelmakers, at the present, is the lack of reliable cleanliness assessment methods to quickly evaluate the quality of steel products at low cost. The cleanliness assessments performed traditionally using metallurgical microscopy, chart comparison, and visual inspection of semi-finished products no longer gives adequate feedback in regard to micro-cleanliness. Image analysis is a powerful tool in assessing steel cleanliness with improved efficiency and accuracy. However, it has been reported[7-8] that



with automated image analysis using optical microscopy there exists a common problem of potential erroneous detection of defects arising from inadequate sample preparation.

The use of inert gas stirring is considered to be a reliable and widespread ladle metallurgy operation in steel production. Argon stirring has an important role in the control of impurities in steels that have been heavily deoxidized and micro-alloyed. In Al-deoxidized HSLA steels, the presence of the classic Al_2O_3 type inclusions is unfortunately unavoidable. [9-10] These impurities cause difficulties either with castability because of occasionally occurring nozzle clogging, or with the ability to meet the ever-stricter requirements for cleanliness. [11-12] There are some ways to avoid these ladle metallurgy problems. However; due to the need for very time-consuming material tests and analysis, these solutions require extremely precise production control (which is challenging even in the case of a well-equipped steel mill), and the technological conditions involved makes quality control difficult to handle.

2. Aim of Research

In this investigation a series of heats were evaluated to optimize the performance and usage of ladle refining system during production of HSLA-X65 steel as final product- Aluminium killed steel melts through controlling the mass flow contour using optimized modelling, optimum usage of Al_2O_3 and Sulphide modifiers and enhancing removal of non-metallic inclusions by altering their morphologies and hence floatation speed. Assessment of fine clusters of inclusions in the final steel product has been industrially emphasized to the cleanness of melt before refining.

3. Melting and Refining

Several heat Logs of HSLA steel grade X-65 has been taken into consideration for further analysis. All heats were melted in 100-130 tons basic-Oxygen steel furnace, deoxidized before tapping using 1000kg burnt lime-200kg Flourspar-900kg FeMn-80kg Al blocks. To ensure deepdeoxidation some deoxidants were also added during tapping to accomplish lowest oxygen activity of about 75ppm, then outside furnace degassed, micro-alloyed and CaSi-Al wire treated in ladle furnace (LF) technology to minimize and optimize count and morphology of non-metallic inclusions respectively. The overall technology flow diagram for steel melting, desulphurization and refining in (LF) is shown as in Figure (1). The average steel composition in final coils was as in Table1.

Table 1. Average values of chemical analysis for X-65 HSLA steel Coils.

Heats	C	Mn	Si	V	Nb	Al	S	P
x-65	0.2-0.25	1.5-1.6	0.5-0.6	0.08	0.09	0.04-0.06	0.007	0.015

3.1 Steel Deoxidation

After oxidation end enhancing melting using gaseous Oxygen, the amount of [o] in the steel melt increased to maximum equilibrium values. It is well known that the maximum solubility of oxygen in liquid iron at the eutectic of 1527°C is about 0.16% [3], while the oxygen solubility in solid iron, at temperature slightly below its melting point, approaches zero. Accordingly, and upon solidification, majority of dissolved oxygen will precipitate as FeO, SiO_2 and MnO inclusions. However the presence of alloying elements such as carbon can influence the dissolved oxygen content as clear from: $[\text{wt}\% \text{C}] \cdot [\text{wt}\% \text{O}] = \sim 0.0023$ [2] at 0.6% C maximum. This means that in order to prevent blowhole formation, porous cast product, or precipitation of FeO inclusions in sizeable quantities, liquid steel must be deoxidized prior to casting.

3.2 Deoxidation with Aluminium

Aluminium is one of the most effective deoxidizers used for steel deoxidation. In aluminium deoxidized steel, there are generally two species of deoxidation products: solid (FeO- Al_2O_3) spinel, and solid (Al_2O_3) corundum. Among the two deoxidation products, corundum is the dominant species found in steel. Corundum phase is characterized by having unique faceted shapes and relative smaller diameter as single particle and during deoxidation, follows dendritic growth. For steels deoxidized solely with aluminium, α - Al_2O_3 products are formed; clusters of these particles tend to remain as inclusions in steel. Corundum

inclusions, usually having the particle size of 1 to 5 μm , have a tendency to agglomerate upon colliding with one another in order to lower the overall contact area with molten steel and therefore effectively stabilize the entire unit by minimizing the surface energy.

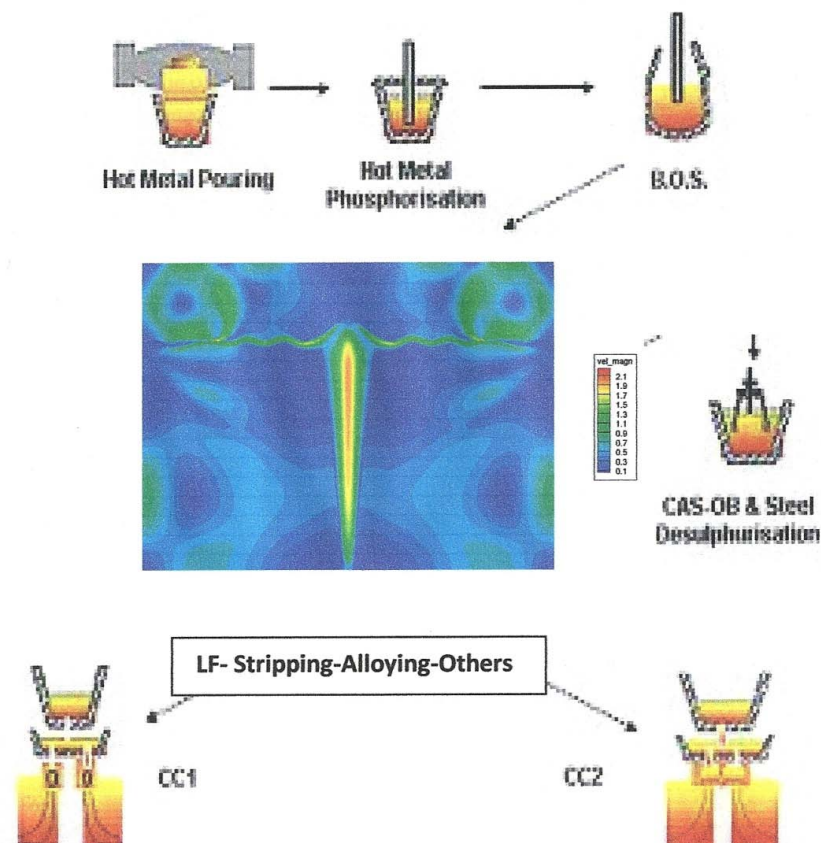


Figure 1. Melting and Refining technologies used in HSLA steel production.

4. Morphology of Nonmetallic Inclusions

After complete deoxidation of the molten steel (in furnace or in the tapping ladle) tremendous amounts of nonmetallic were detected, one of them the harmful Al_2O_3 clusters that deteriorate the continues casting operation through nozzle clogging and the mechanical and rolling conditions of the steel. Calcium has a strong affinity to oxygen and could potentially be utilized as steel deoxidizer and modifier for Al_2O_3 . The challenge, however, lies in the following properties of calcium: low boiling point (1439°C), limited solubility in steel (0.032% Ca at 1600°C), and high vapor pressure at 1600°C (1.81atm).[10] Due to these reasons, it is rather difficult to introduce calcium to molten steel in its metallic form, and it is usually added as various iron-containing Ca-Si alloys. The primary deoxidation products are therefore calcium silicates, which may also contain other oxides. When combinations of Ca and Al or Mn/Si deoxidation are carried out, the primary deoxidation products can be modified to oxides with lower activity and hence improve the removal of dissolved oxygen. By converting the solid alumina inclusions to liquid calcium aluminates, the extent of deoxidation can be improved from 8-10ppm O to 1ppm O in Al-killed steel (0.05% Al).[9] With a CaO: Al_2O_3 ratio of 12:7, calcium

treated Al_2O_3 can reach a melting point of 1360°C at the $\text{CaO}-\text{Al}_2\text{O}_3$ eutectic (Figure 2-7) and therefore exists in the liquid state at steelmaking temperatures. Moreover, there exist five modifications of calcium aluminates as indicated in Figure 2-7; $12\text{CaO}\cdot 7\text{Al}_2\text{O}_3$, $3\text{CaO}\cdot \text{Al}_2\text{O}_3$ and $\text{CaO}\cdot \text{Al}_2\text{O}_3$ are liquid, while $\text{CaO}\cdot 2\text{Al}_2\text{O}_3$ and $\text{CaO}\cdot 6\text{Al}_2\text{O}_3$ are solid at steelmaking temperatures. Instead of agglomerating, in alumina inclusions, liquid calcium aluminates will coalesce upon contact due to better wetting with liquid steel and will not easily attach onto refractory surfaces. Hence, solid deoxidation products can also be calcium treated so that the steel casting process is clogging-free.

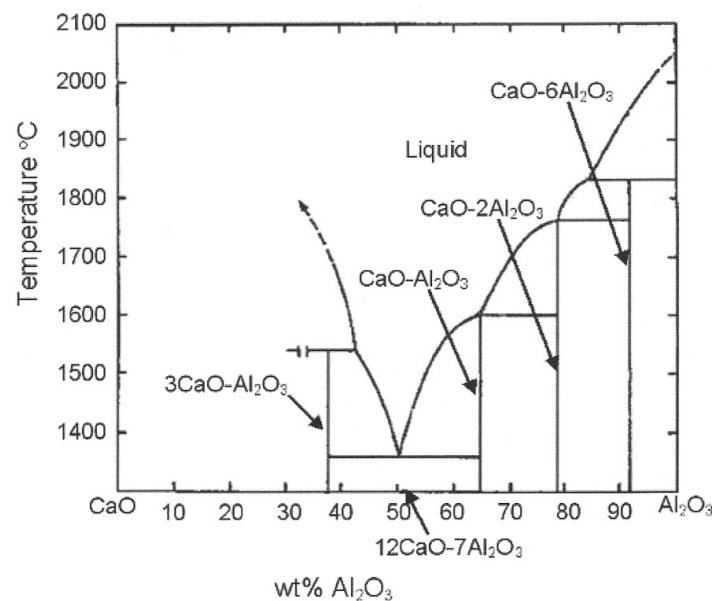


Figure 2. $\text{CaO}-\text{Al}_2\text{O}_3$ equilibrium phase diagram [10].

The modification of inclusions and removing them to the slag layer was accomplished in Ladle-Furnace (LF), in which cored calcium-aluminum wire feeding and melt stirring with Argon gas was used. The maximum gas flow rate was modeled to obtain the optimum turbulence between molten metal and slag layer as shown in Figure 3. It is evident that if an abrupt increase in gas flow rate was exercised, the molten metal can be spilled over the slag layer and then atmospheric oxidation is happened unfortunately again.

To achieve optimized inclusion count and morphology after Ca-cored wire feeding as shown in Figure 4, moderate bath stirring and the technological parameters projected in Table 2 must be used during melting and refining steel melts. The overall outcome revealed that optimal and satisfactory Al_2O_3 inclusion removal from the molten steel could be accomplished in ladle-Furnace at $[\text{O}]$ and $[\text{Al}]$ levels reached 2-3ppm and 0.05-0.06% respectively. This was only possible at the technological values given in table 2.

However the overall modifications occurred during Ca-treatment of liquid steel in LF can be summarized as in Figure 5, where Calcium Aluminates centered the inclusions and CaS precipitated as a rim around the inclusions. Meanwhile, MnS precipitated as scattered globules and cuboids but reversion of $[\text{Mn}]$ and $[\text{Si}]$ can also happened. The floatation of even $\text{Ca}-\text{Al}_2\text{O}_3$ minute inclusions (1-5 microns) to the slag layer was possible leaving behind a clean steel melts. The assurance of intimate final refining slag analyses can give answer for what is the meaning of Optimized Ladle-Furnace treatment. Table 3 shows the overall analyses of the slag after LF processing.

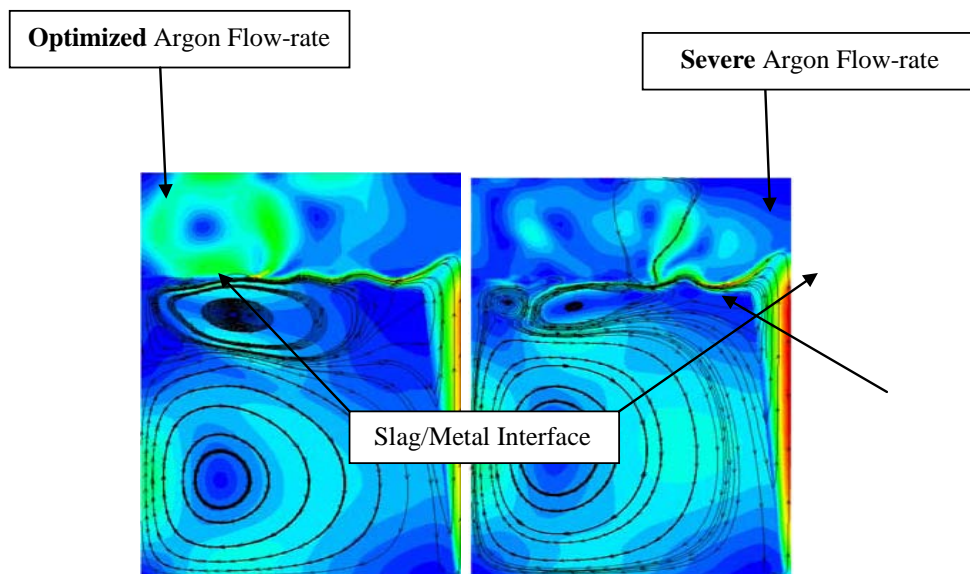


Figure 3.Influence of Argon Flow rate on Metal/Slag Interface.

Table 2.Technological parameters of experimental heats produced at mild stirring.

Total Al kg	CaSi-Al wire kg	Argon used m ³ x100	Blowing time min	[O],.ppm	[Al], %
210	55	31	15	2.4	0.055
245	53	81	25	4.7	0.063
230	57	72	27	4.0	0.057
225	55	67	15	3.6	0.062
210	21	54	19	3.2	0.060
280	34	58	35	3.5	0.061
230	12	53	21	3.0	0.057
210	16	45	21	2.6	0.052
265	52	50	25	3.1	0.053

Argon flow rate Nm ³ /min	1000	2000	3000	4000
Depth of molten steel [m]	2.85	2.85	2.85	3.12
Bulge height [m]	0.21	0.24	0.3	0.39
velocity [m/s]	2.0	2.1	2.2	2.4

5. Optimized Argon Stirring

The removal of minute inclusions depends to a great extent not only on the intensity of Argon stirring but also on the time of blowing, meanwhile care must be taken on the molten steel temperature, not to cool down after long time gas stirring. The dependency between floatation of nonmetallic inclusions (1-50 microns) and the parameters of argon stirring in LF can be controlled according to the turbidity occurred during gas purging and the time required for such operation. However, and according to our gained experience to reduce melt temperature about 4000 Nm³/min of Argon must be blown, to homogenize steel bath about 3000 Nm³/min, to enhance desulfurization process 2000 Nm³/min, and about 1500-2000 Nm³/min can be used for removing impurities.

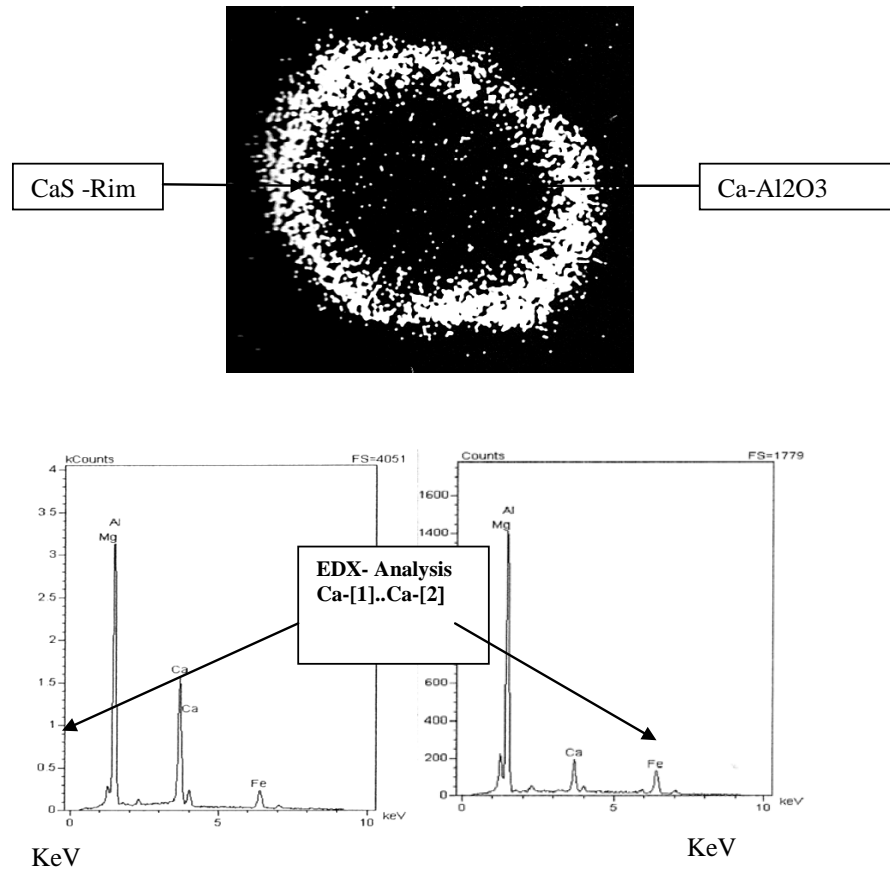


Figure 4. Morphology of modified Al₂O₃ inclusion and its EDX-composition ratios.

The effect of low to high gas flow rate at a given time of purging (15minutes) was investigated through gathering the changes occurred in the final liquid slag and metal analyses such as for (Al₂O₃), (FeO), (SiO₂), [O] and [Al] after treatment in LF. The results can be easily followed as in Figure 6. It is evident that a moderate decrease in (FeO)% along moderate argon blowing (500-1500 Nm³/min), reaching minimal content (4%) at 2000-2500 Nm³/min. However, (FeO) content is drastically increased at higher stirring rates. This is due to the increased bulge height causing direct contact between molten steel and atmospheric air. It is also evident that at increased stirring intensity the activity of oxygen [O] in molten steel and content of Alumina (Al₂O₃) are increased to reach (6-10ppm) and (40-60%) respectively. The optimum LF working zone at satisfactory concentrations of (FeO), (Al₂O₃) and [O] can be obtained at stirring intensity between 2000-2500Nm³/min.

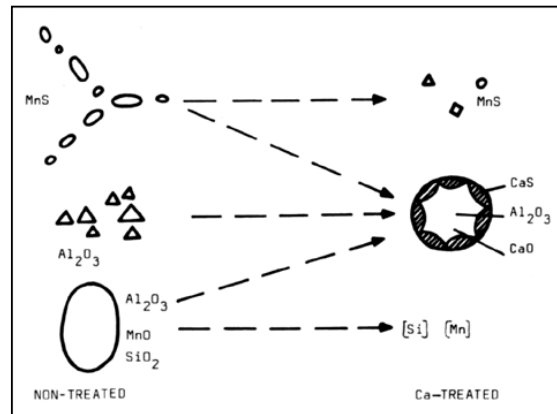


Figure 5. Modification of composition and morphology of inclusions.

Table 3. Overall analyses of slags after LF process Al-Wire Feed 50-60kg + Argon Stirring.

(CaO) %	(FeO) %	(Al ₂ O ₃) %	B	(CaO) %	(FeO) %	(Al ₂ O ₃) %	B
57.78	2.4	25.81	7.23	55.9	2.8	24.8	7.2
58.22	2.24	28.22	13.04	60.8	2.3	25	7.3
56.30	3.45	25.04	6.59	61.3	4.5	24	11.4
62.30	3.78	24.10	12.35	61.4	4.1	25	13
61.17	4.46	23.19	11.53	63.5	3.2	24.3	12.7

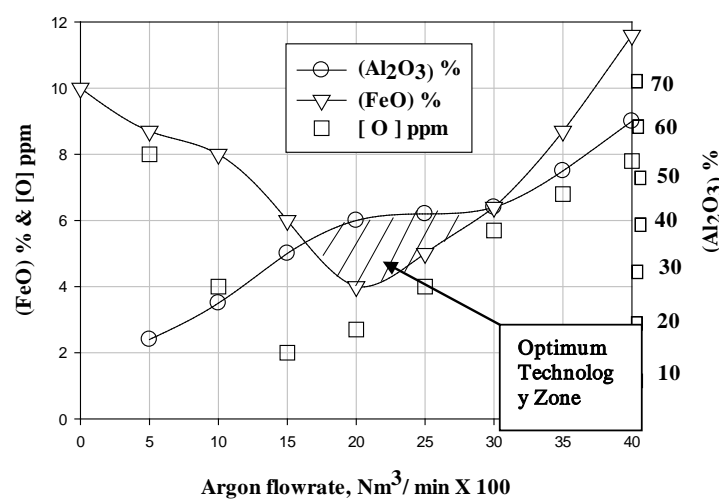


Figure 6. Stirring intensity versus (FeO), (Al₂O₃) slag contents and [O] activity in steel.

6. Conclusion

- 1) Optimized Ladle-Furnace treatment was accomplished not only through adequate bottom stirring modeling but also through industrial implementation and data base establishment.
- 2) The maximum gas flow rate was modeled to obtain the optimum turbulence between molten metal and slag. At abrupt increase in gas flow rate, the molten metal can be spilled over the slag layer and then atmospheric oxidation is happened unfortunately again.
- 3) Optimal and satisfactory Al₂O₃ inclusion removal from the molten steel could be accomplished in ladle-Furnace at [O] and [Al] levels reached 2-3ppm and 0.05-0.06% respectively.
- 4) floatation of nonmetallic inclusions (10-50 microns) depends on the projected parameters of argon stirring in LF.
- 5) To achieve optimized inclusion count and morphology after Ca-cored wire feed, moderate bath stirring and the optimum technological parameters zone has to be followed.

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