Optimization of High Purity Compacted CaSi 30/70 Injection Treatment in Low C Al-Si Killed steels and cleanliness improvement observed compared to conventional cored wire CaSi 30/60 (CCW CaSi 30/60)

Fernando Velázquez Guzmán

John Emerick

Abstract

This work describes the optimization process of High Purity and Compacted (HPC) CaSi 30/70 injection treatment process in low C Aluminum-Si killed steels. A total of 55 heats were treated with HPC CaSi 30/70 wire made with a 1 mm wall thickness sheet (welded seam) and filled with 230 grams of CaSi per meter (0.155 lb/ft). The yield was evaluated in terms of % and ppm Ca. The parameters optimized were injection velocity and kg Ca/ton addition rate. A Ca yield value of 33.7% with 20 ppm Ca in the ladle after treatment and an average consumption of 9.2 kg Ca per heat was documented for these 55 heats. The single best injection velocity and consumption was 122 m/min and 7.4 kg Ca/heat, giving a maximum yield of 43.4% with 20 PPM Ca.

Additionally, inclusion analysis was performed of the heats submitted to HPC CaSi 30/70 with the injection parameters closest to the optimum values. Inclusions were characterized in terms of chemical composition, morphology, density of inclusions (number of inclusions per mm2) and inclusion area fraction (IAF) by Scanning Electron Microscope (SEM/EDX) and Image Analyzer. The inclusion analysis was evaluated before and after the HPC CaSi 30/70 treatment. The inclusions before injection were characterized by Al_2O_3 -Si₂O-MnO inclusions of globular shape and size larger than 20 μ m. After the cored wire injection, the inclusions present were mainly of Al_2O_3 -CaO with MgO content lower than 10% wt, with globular morphology and smaller than 10 μ m in size.

Key words: Yield, ppm Ca, injection velocity, inclusion state, inclusion modification.

Introduction

Currently in the steel industry a great variety of steel grades are deoxidized not only with Al, but also with Si and Mn in order to optimize the deoxidation reactions that take place during tapping. The strict control of these elements is essential in the secondary metallurgy process. Conventional cored wire powders such as conventional CaSi, used in these steel grades without a restricted Si content, have performed well in the modification of solid inclusions. Nevertheless, the large amount of material injected due to reduced yield has influenced the high cost of their use. For this reason steel producers are interested in alternatives that could satisfy their castability and productivity requirements more economically. The optimization of the parameters used during the injection of this HPC CaSi 30/70 welded seam cored wire with a 1 mm wall thickness sheet has proven to allow a more economic CaSi injection process with a better accuracy in the Si chemical analysis, having also an improved inclusion modification mainly obtained due to the deeper penetration reached by this more rigid wire. In addition S.E.V. (Statistics of extreme variation) analysis has revealed an improvement in steel cleanliness levels as compared to conventional cored CaSi 30/60 wire.

Development

A total of 55 heats were treated with HPC CaSi 30/70 wire. The injection parameters were defined according to the weight of the heat and the Ca chemical analysis specification (ppm Ca) at the end of the treatment in the ladle furnace. The average weight of the heats was 152 ton and initially 152 m of HPC CaSi 30/70 (10.5 kg Ca) at a velocity of 137 m/min were injected aiming for a ppm Ca target of 20. These parameters (injection velocity and kg Ca/ton) were modified according to the results obtained. A sample was taken from the ladle furnace after 3-5 minutes of the injection treatment for chemical analysis. Other process variables were monitored in order to evaluate their impact on the treatment yield, such as levels of Al and S dissolved. Furthermore, samples of heats HPC CaSi 30/70 treated were analyzed by Scanning Electron Microscopy and Image Analysis to evaluate the inclusions.

Results

Trials at Steel Shop

Results obtained after HPC CaSi 30/70 injection treatment are presented in Table I. An average yield of 33.7% was accomplished with a standard deviation of 8.1% having as result 20 ppm Ca with 0.060 kg Ca/ton, and all heats treated were within the Ca ppm range specified (10-20 ppm Ca).

RESULTS											
Injected meters	Injected meters TLS		Std Deviation	Average ppm Ca	kg Ca per heat	kg Ca per ton					
133	152	33.7	8.1	20	9.2	0.060					

Table I Average results after HPC CaSi 30/70 injection treatment of 55 heats during trials atSteel Shop.

Effect of the injection velocity and kg Ca / heat addition rate in the efficiency behavior.

The evidence of the optimized parameters for the HPC CaSi 30/70 injection process in the yield is plotted in Figure 1. At the beginning of the tests, the wire was injected at 137 m/min to evaluate the penetration in the liquid bath. Five heats were injected at 137 m/min and 152 meters (10.5 kg Ca per heat) having an average yield of 25.3%. Subsequently the yield was improved reducing the consumption to 9.4 and 8.4 kg Ca per heat (137 and 122 m of wire length respectively). With the objective of evaluating the increase in the yield, the injection velocity was increased to 152 m/min. With this raise in velocity, a slight improvement in the yield occurred reaching levels of 28.5% and 30% with 10.5 and 9.4 kg Ca respectively. Nevertheless, steel splashing at this injection velocity was also higher. The next step consisted of reducing the injection velocity to 122 m/min to determine an adequate wire penetration that guarantees acceptable splashing levels and further increases in the yield obtained. The efficiencies obtained with the injection velocity (122 m/min), the HPC CaSi 30/70 wire goes deeper in the liquid bath before being melted. This causes the Ca to be liberated and react in a high ferrostatic pressure zone, raising the residence time in the steel and resulting in an adequate dissolution and an efficient inclusion modification.



Figure 1. Effect of the injection velocity and Ca consumption in yield treatment.

Al and S content before injection treatment.

In Figure 2 contents of Al and S in liquid steel before cored wire injection are presented. It was observed that most of the heats treated with HPC CaSi 30/70 (85% of the heats) were thoroughly deoxidized and desulphurized. This is evidenced by the S content before the Ca treatment below 0.0080% wt and Al levels which were within 0.025-0.045 %wt. With these adequate levels of Al and low S within the steel it can be guaranteed that the injected Ca would not act as desulphurizer or deoxidant.



Figure 2. Content of Al and S in the liquid bath before HPC CaSi 30/70 injection treatment.

Effect of the injection velocity in Ca recovery

The effect of the wire injection velocity on the Ca recovery in heats treated with HPC CaSi 30/70, in the system Fe-Al-Ca-S-O at 1600 °C is shown in Figure 3. As it can be observed on the right side of figure 3, Al dissolved content after injection treatment ranges in 0.030-0.050 % wt (300-500 ppm Al). At these Al levels, the content of Ca required to promote an adequate inclusion modification must be within 10-25 ppm Ca. With these Ca recoveries, a satisfactory inclusion modification has been reached having mainly liquid calcium aluminates (45-55% CaO). All heats treated with HPC CaSi 30/70 had ppm Ca within the specified range and as the injection velocity was reduced from 152 to 137 and 122 m/min, the ppm Ca recovered increased. It is important to mention that at the optimum injection velocity 122 m/min most of the heats HPC CaSi 30/70 treated recovered Ca contents higher than 20 ppm (above the superior limit specified) showing an important tendency of the inclusions to be enriched with CaO without being saturated with this oxide. In the left side of Figure 3 it can be observed the same tendency of increasing the Ca recovery with an injection velocity reduction. In this part of the Fe-Al-Ca-S-O diagram, it can be noted that for steel grades Si killed, with Ca recoveries higher than 20 ppm, the S content in the metal after the injection was lower than 0.0080% wt (80 ppm) for most of the heats injected with HPC CaSi 30/70, thus conditions for CaS formation were not met.



Figure 3. Effect of the injection velocity in ppm Ca recovered.

Evaluation of the inclusions in the samples

Metallographic preparation and Scanning Electron Microscopy Analyses

Samples were obtained at the ladle furnace before and after the HPC CaSi 30/70 treatment as well as at the caster in three heats named A, B and C, which were treated at optimum Ca recovery injection parameters (7.4 kg Ca at 122 m/min) to characterize their inclusions. Table 2 showns the chemical composition and PPM Ca recovered of these heats. These samples were cut and metallographically prepared for optic microscope analysis. The quantification of number and area fraction of the inclusions was performed on an Image Analyzer coupled to an Olympus Vanox Mod. AHMT3 optic microscope. All microscopy was done on mirror polished surfaces. A total of 50 fields were evaluated with a magnification of 200 X. Figure 4 illustrates the sample preparation. The samples were then processed on a PHILIPS XL30ESEM scanning electron microscope equipped with an EDX micro-analyzer for chemical analysis. The voltage and time of analysis was 20 kV and 30 s respectively.

HEAT	TLS				Chemical Composition after				% Recov LF	
		primary	primary kg	real	%	%Si	% \$	% AI	% (2	16
		(m)	Ca	(m/min)	700	7051	70 3		70 Ca	
А	152	107	7.4	122	0.12	0.184	0.0066	0.045	0.0022	45.4
В	152	107	7.4	122	0.17	0.180	0.0068	0.037	0.0014	28.9
С	152	107	7.4	122	0.17	0.179	0.0073	0.039	0.0020	41.3





Figure 4. Sample preparation for inclusion cleanliness evaluation.

Inclusion chemical composition evolution and morphology

The chemistry of the analyzed inclusions in these optimally processed heats is presented in the ternary systems $Al_2O_3 - SiO_2 - MnO$ (before treatment) in Figures 5 and $Al_2O_3 - CaO - MgO$ after treatment in ladle Furnace in Figure 6. Before the Ca treatment the inclusions were mostly manganese-silico-aluminates with variable contents of alumina (40-100 % wt Al_2O_3). The morphology of such particles was mainly globular and their size was greater than 20 µm. As these inclusions become richer in SiO₂ and MnO, their melting points decrease becoming liquid at a temperature of 1600 °C (see Figure 5). These particles coalesce and form bigger inclusions enabling their flotation towards the slag. Those inclusions rich in Al_2O_3 which remain until CaSi injection are modified to liquid calcium aluminates.



Figure 5. Chemistry of analyzed inclusions in the three optimally processed heats in the ternary system $Al_2O_3 - SiO_2 - MnO$ before Ca treatment.

The chemical composition of the inclusions analyzed after Ca treatment with HPC CaSi 30/70 in ladle furnace is presented in the ternary system Al2O3 - CaO – MgO in Figure 6. The inclusions of these heats were mostly Al_2O_3 -CaO (45-55 %wt) whose melting point is lower than steel making temperature (1590 °C). These inclusions were globular morphology and were bigger than 10 µm. It can be observed in this diagram, the inclusions were fully modified with an injection velocity of 122 m/min. At this velocity, the HPC CaSi 30/70 was released deep enough in the steel to overcome the ferrostatic pressure and reach deeper toward the ladle furnace bottom, such that it has more time to react in the steel before reaching the surface . This type of inclusion with lower surface tension, floats towards the slag improving the steel cleanliness.



Figure 6. Chemistry of the analyzed inclusions in the three optimally processed heats in the ternary system Al2O3 - CaO - MgO.

Figure 7 shows the inclusion chemical composition of two heats before Ca treatment with conventional cored CaSi 30/60 in the ladle furnace and similarly analyzed to compare their inclusion chemical composition and morphology.



Figure 7. Chemistry of analyzed inclusions before Conventional Cored CaSi 30/60 treatment in two treated heats in ladle furnace.

In figure 8 the composition of these inclusions analyzed is presented in the ternary system Al2O3 - CaO – MgO. The inclusions of these heats were mostly Al_2O_3 -MgO (20-50 %wt) whose melting point is much higher than steelmaking temperature (1590 °C). These inclusions were irregular morphology. We can see that after the injection of CCW CaSi 30/60 the inclusions were only semi-modified toward the liquid calcium aluminate area of the ternary.



Figure 8. Chemistry of analyzed inclusions after Conventional Cored CaSi 30/60 treatment in two treated heats in ladle furnace.

Evaluation of cleanliness steel treated with HCP CaSi 30/70 by SEV method

Samples obtained at the caster of treated heats with HCP CaSi 30/70 and CCW CaSi 30/60 were evaluated. The level of steel cleanliness was expressed in terms of maximum inclusion size. This level of cleanliness was evaluated by the statistics of extreme values method (SEV) developed by Murakami et al.¹ In Figure 9 are plotted in ascending order in the horizontal axis the probability function [-Ln (-Ln (H))]) and on the vertical axis is plotted square root of the inclusion area measurement (ν) Shi et al ² used this method to compare different levels of cleanliness of steel by the slope of the linear relationship between the probability function [-Ln (-Ln (H))]) and the square root of the area of inclusion (inclusion size). In this paper we used this tool to discriminate between treated heats with HCP CaSi 30/70 and treated heats with CCW CaSi 30/60. The slope a in the case of heats treated with HCP CaSi 30/70 is smaller than those heats treated with CCW CaSi. A smaller slope will give a smaller calculated size of the "maximum inclusion in the same volume of steel"



Figure 9. SEV Chart for treated heats with HCP CaSi 30/70 and CCW CaSi 30/60.

Conclusions

Injection parameters and yield

From results obtained of the HPC CaSi 30/70 injection trials the following conclusions are presented:

- 1. An average yield of 33.7% was reached in the ladle with the HPC CaSi 30/70 injection treatment.
- 2. The average consumption to obtain such yield was 0.060 kg Ca/ton, that is, 9.2 kg Ca/heat.
- 3. Optimum injection velocity was defined as 122 m/min.
- 4. Best yields (43.4 %) were reached in the ladle by injecting 7.4 kg Ca/heat with an average Ca of 20 ppm.

Steel cleanliness

Regarding the steel cleanliness met with the HPC CaSi 30/70 injection treatment we can conclude the following.

- 1. An immediate modification of inclusions after treatment was met.
- 2. This successful inclusion modification is favored by the strict control in Al and S levels before Ca treatment.
- 3. Best inclusion levels are associated with an early modification of the inclusions (just after the HPC CaSi 30/70 injection), promoting the flotation due to the higher time available for this effect. With more flotation time a better quality finished product results. There are reduced rejects resulting from defects that are exposed during the lamination process due to the reduction of inclusions.
- 4. Inclusion levels via S.E.V. analysis are reduced with HPC 30/70 CaSi when compared to conventional cored 30/60 CaSi. Particularly for the long products SBQ makers, bearing steel producers and specialty steel producers this improvement will yield higher quality end products in your customer's operations.

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