Investigation on the source of surface inclusion defects in SPHC hot rolled coils by slag tracer method

Chengsong Liu[1, 2], Shufeng Yang[*1, 2], Jingshe Li[1, 2], Xiaojie Gao[1, 2], Linzhu Wang[1, 2]

(1. State Key Laboratory of Advanced Metallurgy, University of Science and Technology Beijing, Beijing 100083, China; 2. School of Metallurgical and Ecological Engineering, University of Science and Technology Beijing, Beijing 100083, China)
Corresponding author: Shufeng Yang. E-mail: yangshufeng@ustb.edu.cn

Abstract
Some surface inclusion defects were induced in SPHC hot rolled coils produced in Tang Steel, which affected the quality of steel product and economic benefit significantly. In this study, slag tracer experiments using rare earth elements were carried out in ladle refining and continuous casting processes to investigate the sources and types of surface inclusion defects. Results showed that mold slag entrapment was one of the main causes for inducing surface inclusion defects while refining slag, tundish covering slag and refractory coating of tundish contributed little. Two types of surface inclusion defect were found in the SPHC hot rolled coils which were divided by chemical composition: Al2O3-CaO type and SiO2-CaO type and caused by abnormal calcium treatment in ladle refining and mold slag entrapment, respectively. In addition, when Al2O3 content in the endogenous Al2O3-CaO inclusions in slabs decreased to <60 mass%, the possibility for inducing Al2O3-CaO type of surface inclusion defects in hot rolled coils decreased greatly. By employing the optimization of the composition of refining slag, calcium treatment process and mould flow field, significant improvement on the surface quality of hot rolled coils was verified effectively by industry trials.
Key words: SLAG TRACER METHOD, SURFACE INCLUSION DEFECT, NON-METALLIC INCLUSION, MOLD SLAG ENTRAPMENT

Quality and performance of steel products are significantly affected by non-metallic inclusions. With the development of science and technology, demands for high cleanliness of steel products become stronger and stronger in various fields such as aviation industry, traffic construction, ship engineering and so on. In order to minimize the size and number of inclusions, it is of great importance to investigate and understand their specific sources and formation mechanism in molten steel[1-2]. However, in the process of steel production, the type, quantity, size, morphology and physicochemical characteristics of inclusions in solidified slab are probably different with those in molten steel. One of the main reasons is that during the solidification of molten steel, not only the microstructure and properties of steel matrix but also the composition and characteristics of non-metallic inclusions in steel are probably affected by solid-state reaction between the steel matrix and inclusions [3-7]. It would lead to segregation of casting slab, modification of original inclusions and precipitation of new inclusions. Therefore, it is difficult to determine the specific sources and formation mechanism of inclusions just according to the analysis by SEM-

EDS on the morphology and composition of inclusions in casting slab after solidification.

Tracers such as rare earth elements are usually employed in the ladle, tundish or mould slags to investigate the role of slags in the formation of non-metallic inclusions in steel products. In previous work, many successful experiments were carried out for investigating the sources of inclusions by using slag tracers. F. Fuhr[8] and Min-Ho Song[9] used BaO as the tracer of ladle slag to study the influence of refining slag on the generation and compositions of non-metallic inclusions in final steel products. It was found that inclusions containing BaO were observed in some of the heats processed with the same ladle that previously had been used with traced slags which suggested that the slag layer that remained sticking to the ladle wall after finishing the heat may be a source of inclusions for the next heat. Research in POSCO [10] showed that by employing BaO and SrO as tracers for refining slag and tundish slag respectively, submerged entry nozzle (SEN) clogging was found to be caused by the deposition of solid-state non-metallic inclusions which were generated in secondary refining process. Moreover, by the experiments using La element to trace deoxidation inclusions[11], the relation between the submerged entry nozzle clogging and trace deoxidation inclusions has been investigated and revealed sufficiently.

Surface slag inclusion is a kind of common defect and general technology problem in hot rolled coils. In this study, the sources and types of surface inclusion defects in SPHC hot rolled coils produced in Tang Steel were investigated systematically by slag tracer method. Tracer experiments using rare earth elements were carried out in the process of ladle refining and continuous casting. Specific optimized process measures were proposed and verified by industry trials to effectively control and improve the surface quality of hot rolled coils.

1 Experimental Method

1.1 Production process

Production processes of SPHC hot rolled coil in Tang Steel are shown as follow: smelting in BOF → deoxidizing and alloying → bottom argon blowing in ladle → LF refining → slab continuous casting → heating in soaking pit → continuous rolling → coiling. Final chemical composition of SPHC hot rolled coil is shown in Table 1.

Table 1. Final chemical composition of SPHC hot rolled coil in Tang Steel

Composition	C	Si	Mn	P	S	Als	N
Content / mass%	≤0.06	≤0.03	0.15~0.40	≤0.022	≤0.015	0.030~0.060	≤0.005

Converter steelmaking: BF hot metal is main raw material for converter steelmaking in Tang Steel. Both of desulfurization rate and slag skimming rate of hot metal are more than 90% through hot metal pretreatment. During BOF tapping, lime for top slag forming is added into the ladle. After complete melting of the lime, aluminum matrix deoxidizer is added which immediately is followed by alloy material such as FeSi and FeMn for the improvement of the yielding rate.

LF refining: After the molten steel in ladle pouring into the tundish, residual slag in ladle is returned to subsequent ladles for LF refining to decrease the oxidizability of ladle slag. The FeO and MnO contents decrease to about 5%. White slag operation is employed to increase the Al_2O_3 content in slag to about 8% in the earlier period of LF refining which is beneficial to improve the cleanliness of molten steel. Bottom argon blowing through single hole in ladle refining is used for obtaining homogeneity of composition and temperature of molten steel as well as the promotion of the chemical reaction between molten steel and slag. In order to improve the casting properties of molten steel, calcium treatment is applied after deoxidation by aluminum. Ca-Al wire feeding rate and depth are 3.0~4.5 m/s and >1.6 m, respectively. The composition of Ca-Al wire is listed here: Al-20%~30%, Fe-40%~60%, Ca-20%~30% (mass fraction) and the addition amount is about 200m. Soft argon blowing time after the calcium treatment is about 8 min.

Continuous casting: For reducing the amount of roughing slag in tapping of LF refining, detection system is employed in the ladle nozzle. Submerged entry nozzle and mold slag are used in the continuous casting process. The depth of SEN varies approximately within 90 to 140 mm. Casting speed is set at 1.8 m/min.

In this tracer experiment, the section size of casting slab was 1270 mm×150 mm.

1.2 Tracer experiment method

Several plant trials were carried out to trace the sources of surface inclusion defects in hot rolled coils by tracer method using rare earth elements. On the one hand, the tracers added into slags should have some properties which are easy to be detected. On the other hand, the amount of tracers should be suitable without changing the properties of the slags and original slag-steel system. According to above related literatures and practical production [12-13], BaO, La_2O_3 and SrO were selected as the tracers added into refining slag, coating for tundish and tundish covering slag according to certain proportions which were 8%, 6% and 5%, respectively. Na and K elements were employed to be the tracers for mold slag. Specific adding methods for those tracers are described as follow.

1) LF Refining slag-BaO: after the slag forming process in the middle period of ladle refining, according to the evaluation of the amount of refining slag, 8 mass% of barium oxide was added into ladle from the observing hole.

2) Refractory coating for tundish-La_2O_3: MgO-based dry vibratable mix was fully mixed with lanthanum oxide before being coated onto the tundish wall. Therefore, the La_2O_3 tracer could be homogeneously distributed in dry vibratable mix after the ramming, baking and curing processes.

3) Tundish covering slag-SrO: In the initial pouring stage, tundish powder and SrO tracer were thrown onto the surface of molten steel through the holes in the cover plate of tundish. In order to guarantee the homogeneous distribution of SrO additions for covering the molten steel in tundish, SrO was added evenly from all of the holes.

In order to ensure a good mixing with the refining slag, barium oxide additions close to the end of the LF process were avoided. Mixer was employed for fully mixing of MgO-based dry vibratable mix and lanthanum oxides. After the casting conditions became steady, three similar industry trials were carried out for tracer experiment. Then, compositions of slag and inclusions were analyzed using electron probe micro analyzer (EPMA). In order to reduce analysis error, each value was obtained from the average of three or more analyses.

1.3 Sampling analysis

Slags including LF refining slag, tundish covering slag and mold slag were sampled. After crushing and screening, the compositions of slags were analyzed by chemical titration method. Molten steel in the process of LF refining and continuous casting was sampled by bucket sampler whose dimension is shown in Figure 1. Head of bucket sample with much slag entrapment was cut off. Samples of inner arc side of continuous casting slabs and surface inclusion defects in the hot rolled coils were also taken to analyze the compositions and morphology of inclusions in them by EPMA.

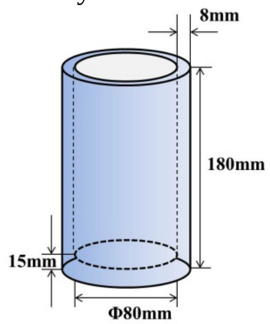


Figure 1. Schematic diagram of bucket sampler

2 Results and analysis

2.1 Compositions of slags and molten steel

Table 2, Table 3 and Table 4 show the main chemical composition of molten steel in the tapping of LF refining, chemical composition of ladle slag in each step of LF refining, tundish covering slag and mold slag. It could be found that the composition of molten steel was qualified within prescriptive range after the tracer experiment. In different steps of LF refining, 6.8~7.7 mass% of BaO content was detected in ladle slags. It was inferred that there was no significant chemical reaction between barium oxide and ladle slag and little loss of barium oxide was obtained. With the process of LF refining going on, the basicity and Ca/Al ratio of refining slag increased and decreased respectively which verified the effect of calcium treatment. Al_2O_3 content in the ladle slag rose up during the refining processes due to the absorption of aluminum deoxidation products by the ladle slag. In the tundish covering slag, SrO content was about 3.5%, while the contents of BaO and La_2O_3 were trace which meant good control of roughing slag in the tapping of ladle refining and low corrosion of refractories in tundish wal. High content of Na, K elements and trace of rare earth elements were found in mold slag. These phenomena suggested that ladle slag, tundish covering slag and refractories in tundish wall were probably non-dominating factors for the generation of surface inclusion defects in the SPHC hot rolled coils.

Table 2. Chemical composition of molten steel in the tapping of LF refining

Element	C	Si	Mn	P	S	Alt	Als	Ca
Content / mass%	0.05	0.02	0.17	0.011	0.007	0.039	0.036	0.0014

Table 3. Chemical composition of ladle slag in each step of LF refining

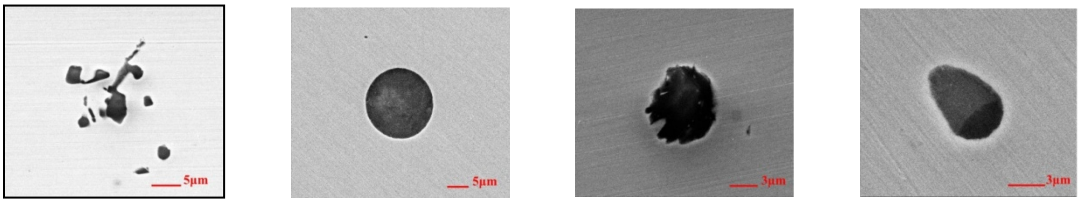
Step	Chemical composition mass%								
	SiO_2	Al_2O_3	MgO	CaO	FeO	MnO	BaO	R	Ca/Al
Tapping of BOF	4.7	32.8	8.7	43.6	1.8	0.8	7.7	9.28	1.33
After calcium treatment	3.6	34.1	9.4	44.4	1.0	0.1	6.8	12.3	1.30
Tapping of LF	3.5	34.9	9.3	44.3	0.5	0.1	6.8	12.7	1.27

Table 4. Chemical composition of tundish covering slag and mold slag

Type of slag	Chemical compositison mass%									
	SiO_2	Al_2O_3	MgO	CaO	FeO	MnO	BaO	La_2O_3	SrO	Na_2O	K_2O
Tundish covering slag	32.2	14.0	9.5	35.2	3.1	2.0	0.3	0.2	3.5	-	-
Mold slag	38.2	4.7	4.8	37.9	0.7	-	-	-	-	12.7	1.0

2.2 Analysis of micro inclusion in steel

Figure 2 shows the morphology and compositions of micro inclusions in molten steel in LF refining and continuous casting processes. As shown in Figure 2(a), after tapping of converter, a large amount of pure Al_2O_3 inclusions was observed. In Figure 2(b), after the calcium treatment, most of the inclusions consisted of Al_2O_3, CaO and MgO. Al_2O_3 contents in some inclusions were still very high (>70 mass%). By analysis of a large number of inclusions using SEM-EDS, it suggested that the modification of some inclusions was not fully completed and the inclusions with low-melting point such as $12CaO{\cdot}7Al_2O_3$ have not been obtained due to abnormal calcium treatment. As shown in Figure 2(c), during tapping of ladle refining, main type of inclusions in molten steel turned to Al_2O_3-MgO-CaS with slight decrease of Al_2O_3 content and increase of CaS content, which demonstrated desulphurization effect of calcium treatment. In tundish, compound inclusions consisting of Al_2O_3, CaS, CaO and MgO were obtained, and the morphology of some inclusions became layered. As shown in Figure 2(d), main component of part of the inclusion was Al_2O_3 (>60%), while that of the other part of the inclusion was CaS (>80%). Similar inclusions were also found in casting slab shown in Figure 2(e). Additionally, some Al_2O_3-CaO-MgO complex inclusions with spheroidal shape were observed in casting slab.



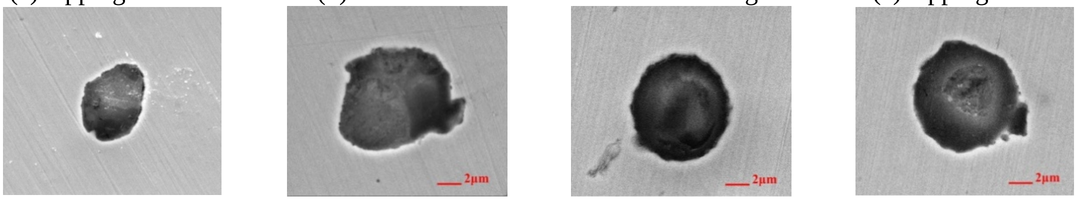
Al_2O_3: 100

Al_2O_3: 55.5 CaO: 34.46 MgO: 10.04

Al_2O_3: 74.32 CaO: 25.68

Al_2O_3: 68.94 CaS: 21.71 MgO: 9.35

(a) tapping of BOF (b) after calcium treatment in LF refining (c) tapping of LF



Upper part: Al_2O_3:4.86 CaO:5.78 CaS:89.36 Lower part: Al_2O_3:67.32 CaO:16.54 CaS:12.21

Upper part: MgO:8.92 Al_2O_3:59.01 CaO:26.25 Lower part: Al_2O_3:2.62 CaO:1.47 CaS:95.91

MgO:6.14 Al_2O_3:64.07 CaO:29.79

Core: Al_2O_3:2.77 CaO:4.95 CaS:92.29 Edge: Al_2O_3:55.41 CaO:25.63 CaS:13.13

(d) in tundish (e) in casting slab

Figure 2. Morphology and compositions of micro inclusions in steel (mass%) in LF refining and continuous casting process

2.2 Analysis of tracer elements in surface inclusion defects

24 surface inclusion defects were obtained from the SPHC hot rolled coils corresponding to the tracer experiments and analyzed by EPMA. In order to avoid misjudgment, when the analyzed content of one kind of tracer in the surface inclusions was over 2 mass%, it was defined that this tracer was involved in the surface inclusion defects and this sample was called “valid sample”. The statistical data for each type of tracer including Na, K, Ba, La and Sr were shown in Table 5 and Figure 3. It could be seen that the ratios of valid samples of Na, K, Ba, La and Sr elements in all samples were 30.43%, 0.65%, 1.09%, 0.65% and 5.65%, respectively.

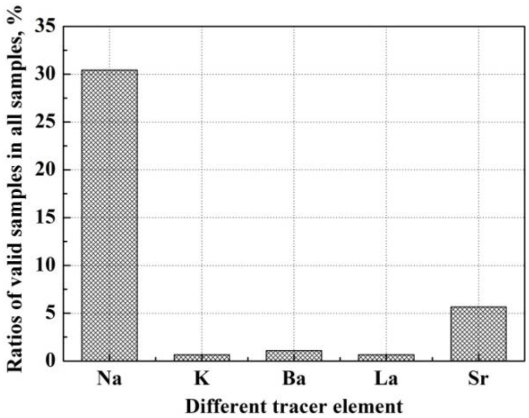


Figure 3. Ratios of valid samples of Na, K, Ba, La and Sr elements in all samples

Table 5. Statistical data for the tracer elements in surface inclusion defects

Serial number of surface inclusion defect	Quantity of all samples	Quantity of valid samples for tracers				
		Na	K	Ba	La	Sr
1#	10	-	-	-	-	-
2#	43	-	-	2	1	-
3#	32	12	-	-	-	1
4#	15	5	1	2	1	-
5#	14	2	-	-	-	-
6#	11	5	-	-	-	1
7#	10	1	-	-	-	-
8#	20	9	-	-	-	-
9#	30	9	-	-	-	1
10#	19	18	-	-	-	1
11#	10	-	-	-	-	-
12#	11	1	2	-	-	2
13#	10	-	-	-	-	-
14#	44	44	-	-	-	-
15#	34	12	-	-	-	-
16#	11	-	-	-	-	-
17#	57	-	-	1	1	-

18#	10	-	-	-	-	-
19#	35	3	-	-	-	-
20#	42	13	-	-	-	-
21#	10	-	-	-	-	-
22#	10	-	-	-	-	-
23#	14	1	-	-	-	3
24#	12	2	-	-	-	-

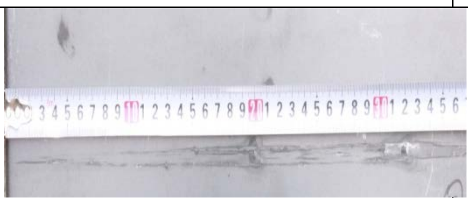
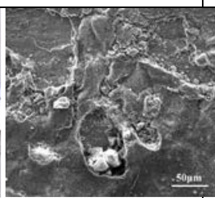
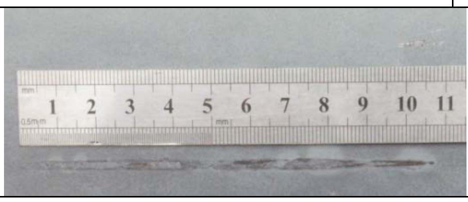
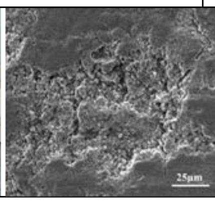
3 Analysis on the sources of surface inclusion defects

According to EPMA analysis, it could be found that there were mainly two types of surface inclusion defects in the SPHC hot rolled coils divided by chemical composition: CaO-SiO_2 type and Al_2O_3-CaO type, whose macro-morphology, micro-morphology and chemical compositions were shown in Table 6.

1) Analysis on the source of CaO-SiO_2 type defects

In the 24 surface inclusion defects, CaO-SiO_2 type defects accounted for about 45.8%. High Na_2O content (7%~12%) was found in the defects which suggested that it probably caused by the mold slag entrapment. Therefore, the chemical compositions of CaO-SiO_2 type defects and mold slag are compared and shown in Figure 4. It could be observed that in the Al_2O_3-CaO-SiO_2 ternary system, the composition of CaO-SiO_2 type defect was very similar with that of mold slag in use. It was inferred that large fluctuation of meniscus or inappropriate properties of mold slag caused the slag entrapment in mold during continuous casting and finally the surface inclusion defects were induced in hot rolled coils.

Table 6. Macro-morphology, micro-morphology and chemical compositions of Al_2O_3- SiO_2 type and Al_2O_3-CaO type surface inclusion defects / mass%

Type of defect	Macro-morphology	Micro-morphology	Main chemical compositions
CaO-SiO_2 type			Al_2O_3:7.36% SiO_2:26.66% CaO:39.79% MgO: 11.1% Na_2O: 9.18%
Al_2O_3-CaO type			Al_2O_3:67.75 % MgO:4.75% CaO:23.82%

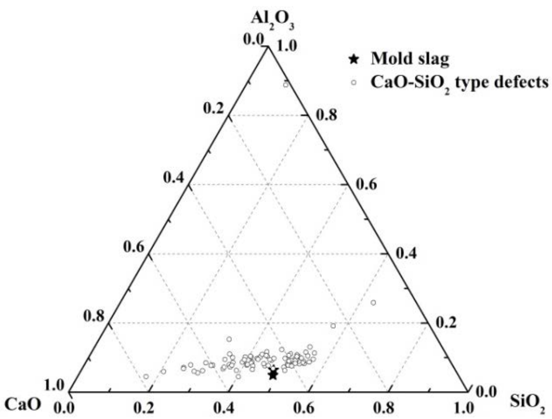


Figure 4. Comparison on compositions of CaO-SiO_2 type defects and mold slag

2) Analysis on the source of Al_2O_3-CaO type defects

In the Al_2O_3-CaO-MgO ternary system, change in the compositions of micro inclusions in molten steel during ladle refining and tundish processes is shown in Figure 5. It could be seen that after the calcium treatment, MgO content in the inclusions increased somewhat probably due to the corrosion of refractories in ladle wall. However, from ladle to tundish, MgO content of the inclusions in molten steel decreased slightly and most of the inclusions have not been covered by the low melting point area in this ternary system because of abnormal calcium treatment. Complex inclusions with high melting points were generated and the casting properties of molten steel would be affected.

Therefore, it could be inferred that Al_2O_3-CaO type inclusions in surface inclusion defects were probably caused by abnormal calcium treatment in ladle refining which resulted in generation and retention of Al_2O_3-CaO inclusions with high melting points in molten steel. Moreover, chemical compositions of Al_2O_3-CaO type inclusions in surface defects were similar with some of those micro inclusions in casting slab, in which the Al_2O_3 contents were >60 mass%, as shown in Figure 6. It further demonstrated that the cause for the Al_2O_3-CaO inclusions in surface defects was due to the aggregation and deposition of micro Al_2O_3-CaO inclusions with high melting points at the outlet of SEN. Al_2O_3-CaO type clogging was formed and probably droped into molten steel resulting in surface inclusion defects. When the Al_2O_3 content in the inclusions decreased to <60%, the generation rate for surface inclusion defects significantly decreased in hot rolled coils with the increase of removal rate of Al_2O_3-CaO type inclusions in molten steel.

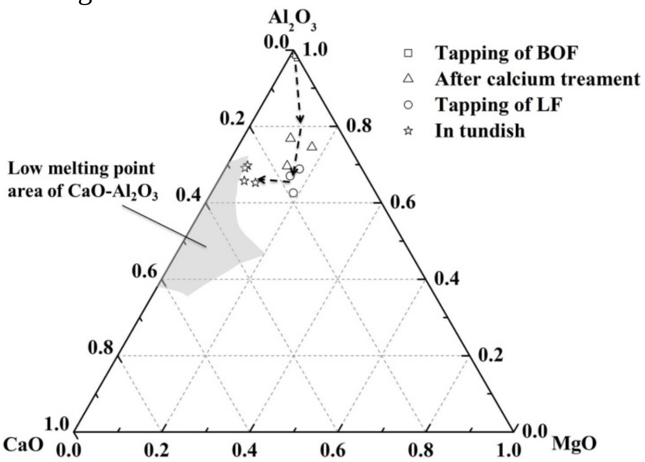


Figure 5. Change in the compositions of micro inclusions in molten steel in ladle refining and tundish

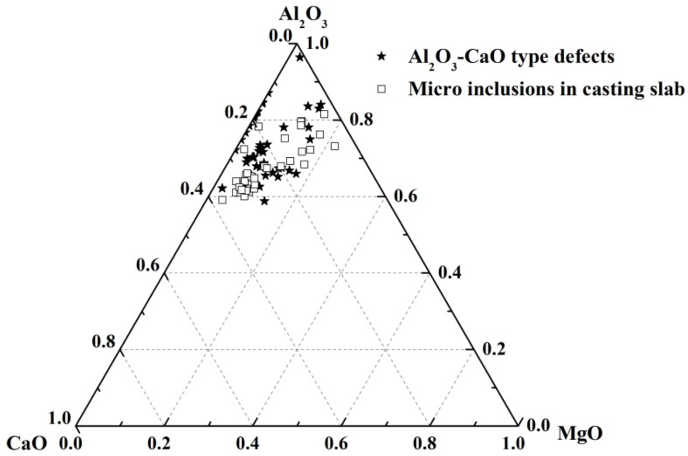


Figure 6. Comparison on the compositions of Al_2O_3-CaO type defects and some micro inclusions in casting slab (Al_2O_3 content<60 mass%)

In addition, in this study, as these surface inclusions were elongated during the hot rolling process, in order to estimate the equivalent diameters D_{eq} of the original spherical inclusions in casting slab, the length, width and thickness of CaO-SiO_2 type and Al_2O_3-CaO type surface inclusion defects in the hot rolled coils were measured. After rolling, the width of the defect was assumed to be the same with the diameter of the spherical inclusion. According to the analysis of length (L), width (D) and thickness (T) of the surface inclusion defects by EPMA, the diameters D_{eq} of original inclusions in casting slab before rolling were calculated by equation (1).

$$\frac{4}{3}\pi(\frac{D_{eq}}{2})^3 = L \times D_{eq} \times T \quad (1)$$

Figure 7 shows the correlation between the lengths of surface inclusion defects in hot rolled coils and the equivalent diameters of spherical inclusions in casting slab before rolling. It could be seen that basically the lengths of CaO-SiO_2 type defects distributed evenly in 20~150mm and the corresponding calculated diameters of large inclusions in slab were mainly in 2~10mm. Most of the lengths of Al_2O_3-CaO type defects distributed in 75~175mm and the corresponding calculated diameters of large inclusions in slab were in 5~8mm. A few of the lengths of Al_2O_3-CaO type defects reached 350~400mm with the corresponding diameters of large inclusions in 12~15mm.

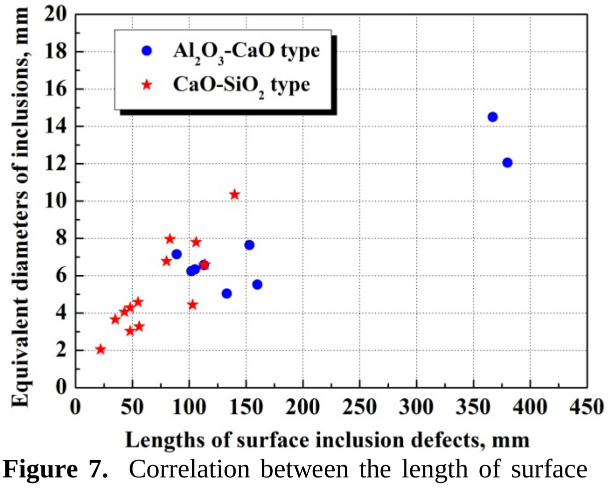


Figure 7. Correlation between the length of surface inclusion defect in hot rolled coils and the equivalent diameter of spherical inclusion in casting slab before rolling

4 Production process optimization of SPHC hot rolled coils

4.1 Optimization of process measures

Based on the results of tracer experiments and analysis on the sources of surface inclusion defects, some optimizations on process measures were proposed as follow: (1) CFD numerical simulation and nail dipping measurement[14-15] need to be employed to study and optimize the flow behavior of molten steel in mold for suppression on the entrapment of mold slag and promotion of inclusion removal in molten steel. (2) By thermodynamic calculation and high temperature experiment, the physical and chemical properties of refining slag in use should be studied and modified to improve the cleanliness of molten steel. (3) Calcium treatment in ladle refining need to be optimized for improving the yield rate of Ca-Al wire to control the composition of micro inclusions in molten steel into the low melting point area in Al_2O_3-CaO-MgO ternary system, specifically Al_2O_3 content should be < 60 mass%.

According to the analysis above, several technological measures involving mould flow field, physicochemical characteristics of refining slag and calcium treatment were optimized in this study.

(1) Using numerical simulation to investigate the mould flow field, the results showed that the optimum port angle and immersion depth of submerged nozzle were 18° and 140mm respectively which were different from the original parameters 12° and 120mm and beneficial to inhibit the mold level fluctuation. Subsequently nail dipping measurement was employed to measure the surface velocity and level fluctuation of liquid steel in mold. The surface velocity results calculated by simulation model were compared with those measured by nail dipping measurement which were shown in Fig. 8. It could be seen that most of experiment results evenly distributed along the velocity magnitude curve calculated by simulation model which verified the accuracy and reliability of the numerical simulation model. The exceptional experiment value which was lower than the calculated result was probably caused by the randomness and instantaneity of turbulence at the surface of molten steel in the mold.

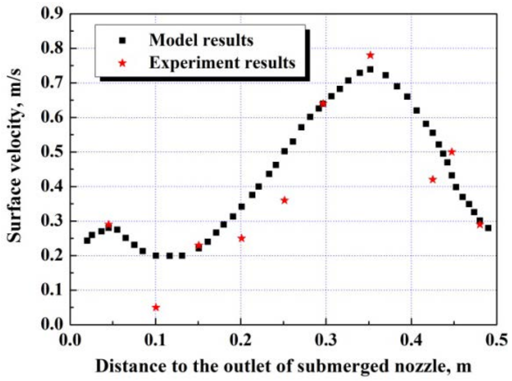


Figure 8. Surface velocity results calculated by simulation model and measured by nail dipping experiment

(2) Using practical production data, the calculation results in FactSage showed that the basicity of refining slag had negative correlation with the silicon content and dissolved oxygen in the molten steel. When the SiO_2 content in the refining slag was lower than 30 mass%, the main factor influencing the activity of Al_2O_3 was the CaO content. Iso-activity line of Al_2O_3 in ternary Al_2O_3-CaO-SiO_2 is shown in Fig. 9. In this figure, 1865K indicated the average temperature of molten steel before calcium treatment in LF refining.

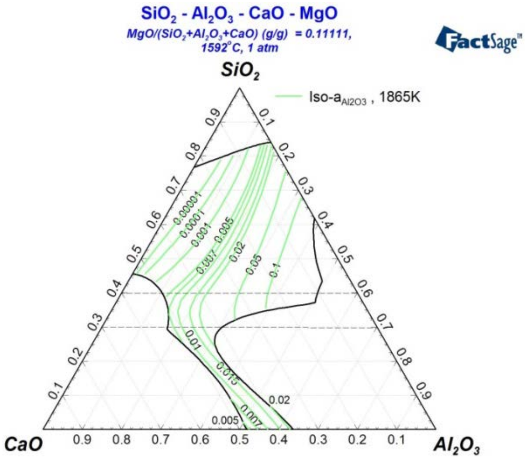


Figure 9. Iso-activity line of Al_2O_3 in ternary Al_2O_3-CaO-SiO_2 (MgO=10 mass%)

In order to melt the refining slag as soon as possible, avoid the increase of silicon content in molten steel, decrease the oxygen content and improve the adsorption capacity of slag for inclusions, the composition of refining slag before calcium treatment should be controlled in suitable region which were: MgO content was about 10%; SiO_2 content should be below 7%; CaO content should be between 42~48% and the basicity of the slag should be higher than 9; The CaO/Al_2O_3 should be between 1.0 and 1.5. Based on thermodynamic calculation and equilibrium experiment at high temperature, the composition of refining slag before calcium treatment was modified as shown in Table 7.

Table 7. Composition of refining slag before the calcium treatment before and after the optimization

Content /mass%	SiO_2	Al_2O_3	MgO	CaO	FeO	MnO	R	CaO/Al_2O_3
Before	5.88	34.4	8.45	52	0.54	0.058	8.84	1.51
after	1.56	40.07	9.94	47.78	0.66	0.058	12.9	1.20

(3) Research on Ca-Al wire feeding optimization in ladle was accomplished in the steelmaking plant at Tang Steel. Addition of Ca-Al wire increased from 200m to 300m to improve the effect of calcium treatment and decrease the Al_2O_3 content in Al_2O_3-CaO type inclusion.

4.2 Improvement on cleanliness of steel and surface quality of SPHC hot rolled coils

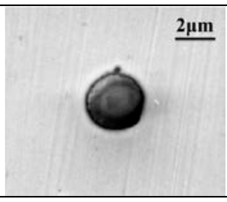
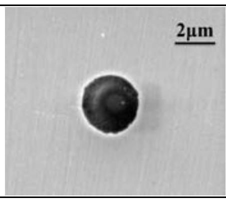
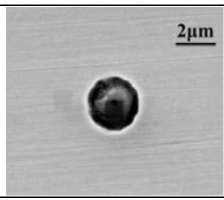
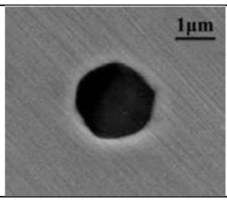
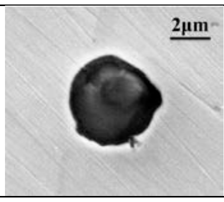
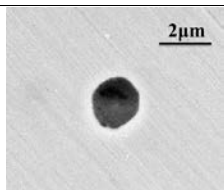
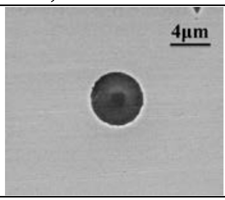
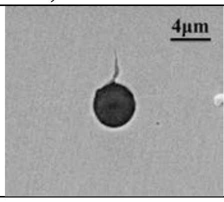
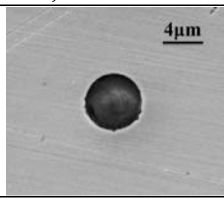
Several plant trials were carried out to verify the effect of optimization process measures. Steel samples were taken all along the process in the trials (without any tracer addition). Oxygen and nitrogen contents in the steel were analyzed and shown in Fig. 8. It could be seen that compared with that before the optimization, the oxygen contents in molten steel during the tapping of LF refining and slab after the optimization decreased from 24 ppm and 32 ppm to 21 ppm and 18 ppm respectively. Nitrogen content in the slab after the optimization was about 24 ppm which was similar to that before the optimization. In general, optimized process measures were benefit to the decrease of total gas contents in the slab and improvement on cleanliness of steel product.

Table 8. Total oxygen and nitrogen contents in steel in different process before and after the optimization of process measures

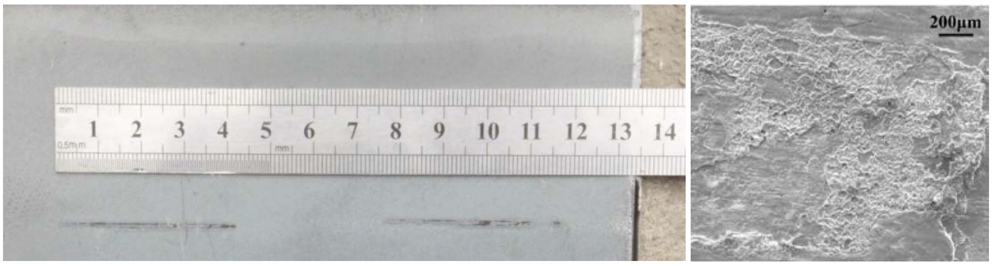
Procedure	Total oxygen content in steel / ppm		Nitrogen content in steel/ ppm	
	Before	After	Before	After
Tapping of BOF	61	45	14	19
Before the calcium treatment	96	52	24	18
After the calcium treatment	26	31	23	21
Tapping of LF	24	21	20	23
In tundish	42	25	23	25
In slab	32	18	24	24

Steel samples were polished and analyzed by means of EPMA involving the composition and morphology of micro inclusions in steel which were shown in Fig. 9. Results showed that before and after the optimization of process measures, the morphology of micro inclusions were similar. In the steel samples after the calcium treatment, micro-inclusions were mainly composed of Al_2O_3 and CaO. Due to the modification of composition of refining slag and increase of Ca-Al wire addition, Al_2O_3 content in the most of inclusions decreased to below 60 mass%. Additionally, adsorption capacity of refining slag was improved and benefited to removal of large inclusions. The average size of inclusions decreased to about 2 μm~4 μm. Most of inclusions in slab are mainly CaS-Al_2O_3-CaO by the analysis of EPMA and the ratio of spherical inclusions also increased according to the evaluation using image analysis software incorporated in the EPMA.

Table 9 . Morphology and composition of typical micro inclusions in steel during different process after the processing measurement optimization / mass%

After the calcium treatment			
	Al_2O_3: 54%, CaO: 42%, MgO: 3.2, CaS: 0.8%	Al_2O_3: 46%, CaO: 39%, MgO: 5.7, CaS: 9.3%	Al_2O_3: 54%, CaO: 46%,
During LF tapping			
	Al_2O_3: 55%, CaO: 34%, CaS: 11%	MgO: 12% Al_2O_3: 43%, CaO: 45%	Al_2O_3: 59%, CaO: 36%, CaS: 5%
In slab			
	MgO:8% Al_2O_3:53% CaO:39%	Al_2O_3:52% CaO:6% CaS:42%	Al_2O_3:51% CaO:49%

Defect samples in hot rolled coils produced by these plant trials were also taken for analysis. After the optimization of process measures, defect rate decreased from 31.86% to 6.24%. Macro and micro morphology of typical surface inclusion defects are shown in Fig. 10. EPMA analysis showed that most of the surface inclusion defects were composed of Al_2O_3-CaO while the CaO-SiO_2 type defects including Na_2O content basically disappeared. It was concluded that the optimization of the mould flow field inhibited mould slag entrapment and decreased surface inclusion defects in hot rolled coils effectively. Few Al_2O_3-CaO type of surface inclusion defects were probably caused by the secondary oxidation of molten steel in tundish.



(a) macro morphology **(b) micro morphology**

Figure 10. Macro and micro morphology of typical surface inclusion defect in hot rolled coil after the optimization of process measures

Generally the optimization of the refining slag composition, calcium treatment and mould flow field could effectively improve the modification and removal of inclusions in slab, decrease generation rate of surface inclusion defects in hot rolled coils. Good control and improvement on the surface quality of hot rolled coils have great technical and economic significance in the SPHC steel production of Tang Steel.

4 Conclusions

Based on the results of tracer experiments, ladle slag, tundish covering slag and refractories in tundish wall contributed little to the formation of surface inclusion defects in the SPHC hot rolled coils, while mold slag entrapment was one of the main causes for that.

Two types of surface inclusion defect were found in the SPHC hot rolled coils divided by chemical composition: Al_2O_3-CaO type and SiO_2-CaO type, which were caused by abnormal calcium treatment in ladle refining and mold slag entrapment, respectively.

When the Al_2O_3 content in the endogenous Al_2O_3-CaO inclusions in slabs was below 60 mass%, the possibility for inducing Al_2O_3-CaO type surface inclusion defects in hot rolled coils decreased greatly.

By employing the optimization of the composition of refining slag, calcium treatment process and mould flow field, significant improvement on the surface quality of hot rolled coils was verified effectively.

Acknowledgement

The authors would like to acknowledge the funding of the National Natural Science Foundation of China (Grant no. 51374021 and no. 51304016) and the Fundamental Research Funds for the Central Universities (Grant no. FRF-TP-14-114A2).

References

1. S. F. Yang, J. S. Li, X. Z. Gao, et al. Modification Kinetics of MgO-Al_2O_3 Inclusions by Ca-treatment. Metallurgical and Mining Industry, 2014, 6(2): 66-71.
2. S. F. Yang, J. S. Li, L. F. Zhang, et al. Evolution of Mgo·Al_2O_3 Based Inclusions in Alloy Steel During the Refining Process. Metallurgical and Mining Industry, 2010, 2(2): 87-92.
3. K. H. Kim, S. J. Kim, H. Shibata, et al. Reaction between MnO-SiO_2-FeO oxide and Fe-Mn-Si solid alloy during heat treatment. ISIJ International, 2014, 54(10): 2144-2153.
4. H. Shibata, K. Kimura, T. Tanaka, et al. Mechanism of change in chemical composition of oxide inclusions in Fe-Cr alloys deoxidized with Mn and Si by heat treatment at 1473K. ISIJ International, 2011, 51(12): 1944-1950.
5. H. Shibata, T. Tanaka, K. Kimura, et al. Composition change in oxide inclusions of stainless steel by heat treatment.

Ironmaking & Steelmaking, 2010, 37(7): 522-528.
6. W. Choi, H. Matsuura, F. Tsukihashi. Changing behavior of non-metallic inclusions in solid iron deoxidized by Al-Ti addition during heating at 1473K. ISIJ International, 2011, 51(12): 1951-1956.
7. M. Suzuki, R. Yamaguchi, K. Murakami, et al. Inclusion particle growth during solidification of stainless steel. ISIJ International, 2001, 41(3): 247-256.
8. F. Fuhr, G. Torga, F. Medina, et al. Application of slag tracers to investigate source of non-metallic inclusions. Ironmaking & Steelmaking, 2007, 34(6): 463-470.
9. M. H. Song, M. Nzotta, S. C. Du. Study of the formation of non-metallic inclusions by ladle glaze and the effect of slag on inclusion composition using tracer experiments. Steel Research International, 2009, 80(10): 753-760.
10. S. H. Nam, O. D. Kwon, D. W. Yang, et al. Improvement of steel cleanliness in ladle exchange period. 1995 Steelmaking Conference Proceedings, Nashville, 457-463.
11. T. H. English, D. J. Dyson, K. D. Walker, et al. Use of rare earth tracers in determination of inclusions sources and origins at British Steel, Scunthorpe Works. Ironmaking & Steelmaking, 1993, 20(2): 97-103.
12. H. Y. Tang, J. S. Li, L. Liu, et al. Research on non-metallic inclusions of high-pressure boiler steel. Steelmaking, 2008, 24(4): 26-29 (In Chinese).
13. A. M. Zhang, J. M. Li, X. L. Yang, et al. Study on behaviors of inclusion during slab casting. Iron and Steel, 2001, 36(11): 22-24 (In Chinese).
14. C. B. Ji, J. S. Li, H. Y. Tang, et al. Effect of EMBr on flow in slab continuous casting mold and evaluation using nail dipping measurement. Steel Research International, 2013, 84(3): 259-268.
15. R. Liu, B. G. Thomas, J. Sengupta, et al. Measurements of molten steel surface velocity and effect of stopper-rod movement on transient multiphase fluid flow in continuous casting. ISIJ International, 2014, 54(10): 2314-2323.