

42nd Steelmaking Seminar - International

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INTERACTION BETWEEN MOLTEN STEEL AND MgO-BASED TUNDISH LINING: A POTENTIAL SOURCE OF NON-METALLIC INCLUSIONS¹

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Abstract

Interactions between the molten steel and the refractory linings are of fundamental importance for the steelmaking industry. During the casting process the steel cleanliness can get worse because of the oxygen pick up of the liquid steel in contact with the atmosphere and the reoxidation of the molten steel caused by top slag and the refractory materials with high oxygen potential. In the industrial trials, three tundish refractory linings (pre-formed boards and two kinds of gunning masses, all MgO based refractories) were used in order to evaluate the interactions among the refractories and the molten steel. Due to small dimension of the tundish (5 ton) used in the continuous casting at Villares Metals SA, after casting, the chilled steel was cut in order to show us in a single section the stopper rod, submerged entry nozzle (SEN), MgO ramming and the work refractory lining. Cross sections of samples from steel/refractory interface were investigated by means electron probe microanalysis (EPMA). Three major aspects were observed at the steel/refractory interface: (1) steel infiltration into the tundish refractory lining, mainly in the gunning masses; (2) a steel oxidized layer formed at the steel/refractory lining interface and; (3) many particles were found in the steel phase, near to steel/refractory interface, being a potential source of non-metallic inclusions.

Key words: Steel; Refractory; Interaction; Inclusion.

INTERAÇÃO ENTRE O METAL LÍQUIDO E O REVESTIMENTO REFRATÁRIO (MgO) DO DISTRIBUIDOR: UMA POTENCIAL FONTE DE INCLUSÕES

Resumo

As interações entre o aço líquido e os revestimentos refratários são de grande importância para a indústria siderúrgica. Durante o processo de lingotamento contínuo a limpeza do aço líquido pode ser prejudicada devido à reoxidação em virtude do contato com a atmosfera, escória e refratários com elevado potencial de oxigênio. No presente trabalho, três revestimentos refratários baseados em MgO foram utilizados durante o lingotamento contínuo do aço SAE HNV 3, visando avaliar a interação entre o aço líquido e os revestimentos (duas massas projetadas de composições diferentes e placas pré-formadas). Devido à pequena dimensão do distribuidor (5 t) utilizado na Villares Metals S/A, após o lingotamento, o aço remanescente, tampão, válvula submersa, linha de reparo e de trabalho foram cortados de tal modo para serem vistos numa única seção. Amostras da interface aço/refratário foram analisadas utilizando-se uma microssonda eletrônica. Três principais aspectos foram observados na interface aço/refratário: (1) infiltração de aço no refratário do distribuidor, principalmente nas massas projetadas; (2) formação de uma camada de óxido na interface aço/refratário constituída pelos principais elementos do aço e; (3) foram encontradas muitas partículas resultantes da interação, próximas à interface aço/refratário, sendo uma potencial fonte de inclusões.

Palavras-chave: Aço; Refratário; Interação; Inclusão

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1 INTRODUCTION

The increasing demands to obtain products with greater cleanliness drive the continuous improvement of steelmaking practices to ensure low and consistent level of non-metallic inclusions in the cast material. Non-metallic inclusions can be classified in two types depending on the generation sources: endogenous and exogenous. Endogenous inclusions in steel are mainly formed in deoxidation process, while exogenous inclusions are caused by the reoxidation of molten steel, slag entrapment and breakdown of refractories materials. In order to take actions that can minimize the presence of inclusions, it is important to have a proper understanding of their sources and mechanisms of generation. (1-10)

In the production of high-quality steels, it is essential that molten steel is not oxidized during casting. The refractories applied to the continuous casting consist of air seal pipe, tundish lining, coating refractories, insulating board, nozzle stopper, sliding nozzle and submerged nozzle. These refractories have a large influence on the casting operation and steel quality; therefore high grade refractories should be applied. (11) Tundish refractory lining can be a source of reoxidation for deoxidized steels. Depending on the steel grade, these reactions can lead to the formation of alumina and spinel particles, which influence the steel cleanliness and castability. (12-14)

The experimental work presented here, which consists in industrial trials, concerns about the interaction between molten steel and MgO-based tundish lining. In order to find a relationship between the characteristics of the macroinclusions found out in the rolled bars and the products generated at tundish refractory lining-steel interface, three tundish skulls were cut and the steel-tundish lining interfaces were analyzed by means electron probe microanalysis (EPMA).

1.1 Metal-refractory Reactions

According to Lehmann, Boher and Kaerle, (15) the liquid steel reoxidation by refractories products increases when the MgO content of the refractories decreases. The authors showed that the main reaction causing this reoxidation is the reduction of the iron oxide, which content increases with the silica content of the refractories, i.e. with their olivine proportion. In addition, it was shown that the water content is an important aspect. Refractories fired at 1200 °C before the contact with liquid metal provide less reoxidation than refractories simply dried at 180 °C. Other experiments were performed by the same authors (15) in order to study the magnesium transfer from magnesia refractories (gunning mass) to the molten steel. The experiments consisted in the immersion of an alumina plate in the molten steel (Al and Si-Mn deoxidized steels) and in the measurement by image analysis of the degree of transformation of alumina crystal to spinel at surface of the plate. Lehmann, Boher and Kaerle⁽¹⁵⁾ concluded that the magnesium transfer to liquid steel increases with the refractory MgO content, and can lead to the formation of spinels, which can contribute to deposits on alumina casting refractories. According to authors. (15) the reoxidation products lead to the formation, at the interface between steel and refractory, of a partly solid reaction layer. The composition of this reaction layer, as well as its physical state (i.e. its percentage of liquid phase), varies from one refractory to the other. Magnesia is present in the solid reaction layer only for the refractories with MgO content larger than 75%. The authors conclude that the tundish refractories choice must be a compromise: refractories with MgO content larger than



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75% will benefit for overall cleanliness by minimization of reoxidation, whereas the amount of harmful spinel would be much higher.

In other experimental work, Lehmann, Boher and Kaerle⁽¹⁶⁾ studied the reaction between MgO-based tundish refractory (gunning mass) and an Al-killed steel with low silicon content. The main reactions taking place between the liquid metal and the refractory are the reduction, by aluminium dissolved in the metal, of the most reducible oxides (iron oxide and silica) contained in the refractory. These reactions result in the formation of two reactions layers: a first layer covering the refractory, made of very many spinel crystals and a liquid silicate phase, and a second layer covering the first one, made of calcium aluminates. These two layers contain metallic droplets especially numerous in the calcium aluminate layer. This study has shown the importance of on the one hand, the iron oxide content of the refractory on the extent of reoxidation and, on the other hand, the liquid silicate phases in the transport mechanisms of the reactive agents towards the metal.

Simões and Janssen⁽¹⁷⁾ studied the influence of tundish lining composition on steel cleanliness. After casting, some tundish lining samples were taken from steel-lining interface and analyzed by means scanning electron microscopy and energy dispersive spectroscopy (SEM-EDS). According to the authors, there is a strong steel reoxidation at steel-tundish lining interface, which can produce very large inclusions during casting process, sometimes, too late for flotation, because of the origin of the inclusion (near to submerged entry nozzle - SEN) and unsuitable tundish design or liquid metal flow. Besides the influence of the air that infiltrates through refractory lining porosity and how this may affect the generated inclusions, the silica in tundish lining was the main identified parameter in the formation of liquid slag type phase, which combines with steel reoxidation products and other lining components. The authors conclude that only low silica linings should be used in direct contact with liquid steel, mainly in the production of Al killed steels.

Bannenberg and Lachmund⁽¹⁸⁾ also investigated the reactions between tundish lining and their influence on steel cleanliness. The plant trials as well as the laboratory tests demonstrate the transformation of the magnesium silicate (2MgO.SiO₂) to spinel (MgO.Al₂O₃) at the refractory-steel interface.

2 EXPERIMENTAL

2.1 Plant Description

The range of products produced at Villares Metals S.A. covers tool steel, high speed steel, stainless steel, valve steel, engineering steel and nickel based alloys. The steelmaking shop has two electric arc furnaces (35 and 25 t) powered by two 20/24 MVA transformers. Secondary refining facilities consist of two ladle furnaces powered by two 4 MVA transformers and two vacuum degassing/vacuum oxygen decarburizing (VD/VOD) units. The special melting practice comprises two vacuum induction (VIM) units, three electro slag re-melting (ESR) units and one vacuum arc re-melting equipment (VAR). In addition, ingot and continuous casting, hydraulic forging presses (up to 5000 ton), rolling and roughing mills, heat treatment, and finishing and inspection lines complete our facilities. Table 1 shows the main characteristics of the continuous casting machine at Villares Metals S.A.



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Table 1. Main characteristics of the continuous casting machine

	<u> </u>
Ladle capacity	25 t
Tundish capacity	5
Machine radius	10 m
Number of strands	1
Cast section	145 x 145 mm
Mold type	Curved, 750 mm
Oscillation type	Electromechanical
Stirring	M + S-EMS
Secondary cooling	Air Mist
Containment	2 rows foot rolls + 4 rolls containment

2.2 Plant Trials

Three heats were performed in order to investigate the interaction between tundish refractory lining and molten steel. Tables 2 and 3 show the chemical composition of the steel (SAE HNV 3) and tundish refractory linings used in the experiments, respectively. The gunning masses No. 1 and 2 come from different suppliers.

The gunning mass is a MgO-based refractory applied with a trowel by projecting it on cold tundishes. The gunning mass consists in two main phases: magnesia and magnesium silicates. Magnesia grains are assemblages of pure magnesia crystals bonded by a mixture of various secondary phases whose main one has a composition very close to monticellite (CaO.MgO.SiO₂). According to suppliers of the gunning masses, the iron oxide content shown in table 3 comes from the olivine 2(MgO,FeO).SiO₂. To these main phases are added mineral fibers, cellulose and sodium silicate (binder). Before casting, the tundish lined with the gunning mass is dried and pre-heated up to 1200°C. The pre-formed MgO boards come from the same supplier of the gunning mass No. 1. In fact, the same raw material for the gunning mass No. 1 is used to produce the pre-formed MgO boards, except the cellulose.

After casting, the billets were rolled in order to produce bars with \varnothing 25 mm. Further, these bars were inspected by means ultrasonic testing in order to verify the presence of macroinclusions.

Table 2. Chemical analysis of the molten steel (wt%)

		1110 111011011 01001 (11170		
С	Si	Mn	Cr	Ni
0.45	3.1	0.4	8.5	0.25

Table 3. Chemical analysis of the gunning masses and MgO pre-formed boards (wt%).

Refractory	MgO	SiO ₂	Fe_2O_3	AI_2O_3	CaO	Na ₂ O	<i>K</i> ₂ O
Gunning No. 1	93.0	2.19	1.2	0.72	1.59	0.28	0.65
Gunning No. 2	84.6	3.64	5.0	1.13	5.04	0.47	0.12
MgO boards	91.5	3.0	2.0	1.5	0.5	na	na

na = not analyzed.

2.3 Samples Preparation

Due to small dimension of the tundish, after casting, the chilled steel was removed from the tundish. Further, the tundish skull was cut according to Figure 1, where is possible to see in a single section the stopper rod, chilled steel, submerged entry nozzle (SEN), MgO-based refractory lining (gunning mass and pre-formed



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board), MgO ramming and the region which was analyzed by means electron probe microanalysis (EPMA).

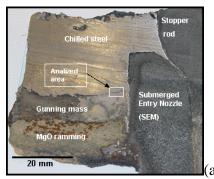




Figure 1. Typical cross section of the tundish skull showing the stopper rod, chilled steel, submerged entry nozzle (SEN), MgO ramming and work refractory lining. (a) gunning mass No. 1; (b) pre-formed MgO board.

3 RESULTS AND DISCUSSION

Three major aspects were observed at the steel/refractory interface: (1) steel infiltration into the tundish refractory lining, mainly in the gunning masses; (2) a steel oxidized layer formed at the steel/refractory lining interface and; (3) many particles were found in the steel phase, near to steel/refractory interface, being a potential source of non-metallic inclusions if they are not removed by flotation.

The pre-formed MgO boards were less prone to steel infiltration than the gunning materials (Figure 2). The main difference between them is related to production and application, which influences the porosity of the tundish lining materials. Two aspects contribute to greater porosity in the gunning mass, the first one is its use (sprayed on cold tundish) and the second aspect is related to water content used for spraying, which initially fills the pores and further is removed during heating. On the other hand, MgO boards are pre-formed materials and there is no concern about water content. However, in terms of operational practice, the deskulling of the gunning mass is easier than the pre-formed MgO boards.

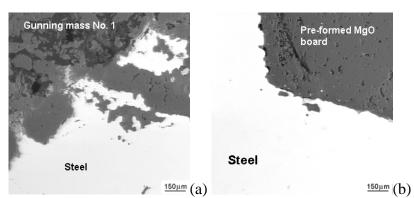


Figure 2. Steel/refractory interface. (a) gunning mass No. 1 and; (b) pre-formed MgO board.

As can be seen in Figure 3, many particles are detaching from tundish refractory lining (gunning mass No. 1 and No. 2), most of them consisting in silicate based phases and MgO crystals involved by CaO.MgO.SiO₂ phase. When present, silicate based liquid phases from refractory lining are capable of incorporating and dissolving all the oxides that come from the steel oxidized layer. This results in the formation of liquid complex slag type phases that mainly include Al₂O₃, CaO, FeO,



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MgO, MnO, Na $_2$ O and SiO $_2$, as well as other minor or less frequent components. Once detached from the tundish lining and when mixed and in contact with the bulk of the steel which was not reoxidized, these non-metallic inclusions rich in highly reducible oxides will progressively react with the killing elements present in the steel and their composition will evolve. $^{(17)}$

For the gunning materials, at steel/refractory interface, it is possible to see spinel particles, which are not a primary component of gunning mass, but a reaction product originated from interaction of refractory material and the aluminium from the molten steel (see figure 3b and table 4). According to Bannenberg and Lachmund, for MgO-lining materials with low silica content is necessary to take into account the reaction (1). Considering the MgO-Al₂O₃-SiO₂ system, for lining materials with silica content less than 55% in the binary system MgO-SiO₂, the transformation of magnesium silicate in spinel (MgAl₂O₄) takes place in the solid state because the liquidus temperatures of these phase areas are clearly above 1700 °C. This reaction does at first not impair the steel cleanliness because there is a substitution of silica by alumina in the lining material. However, at the steel/gunning mass interface is formed a spinel layer, which can spall and impair the steel cleanliness.

$$3(2MgO.SiO_2) + 4AI = 2(MgO.AI_2O_3) + 4(MgO) + 3Si$$
 (1)

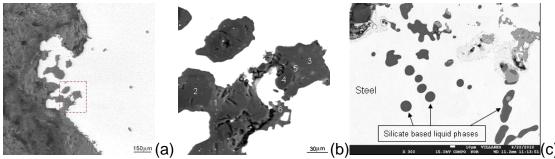


Figure 3. Backscattered electron images (SEM) from steel/refractory interface. (a) and (b) gunning mass No. 1; (c) gunning mass No. 2.

Table 4 – Microanalysis (EDS) from regions shown in Figure 3b

Regions/wt%	MgO	AI_2O_3	Ca0	SiO ₂
1	54.83		2.96	42.21
2	100.00			
3	29.52	70.48		
4	99.63	0.37		
5	31.50		28.66	39.84
6	29.31	70.69		

Figure 4 shows a sequence of pictures in which is possible to see the refractory lining/steel interface and a particle presenting three phases, i.e., spinel (MgAl₂O₄), magnesium silicate and metallic droplets. The compositions of these three phases are shown in Table 5. It is totally clear the transformation of magnesium silicate (forsterite) in the spinel phase at steel/refractory interface according to reaction (1). In addition, note that the Si content in the metallic droplet exceeds the nominal composition of the molten steel, in other words, part of the Si generated by reaction (1) is transferred to metallic droplet. Another interesting characteristic of the metallic droplets is the presence of Ni in its composition. The Ni content in the metallic droplet can reach up to 20% and is totally outside the molten steel



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composition (Table 2). The source of Ni comes from the addition of spent refractory from stainless steelmaking to raw material of the gunning mass.

When the spinel phase is present, three layers near to steel/refractory interface were indentified. The first one consists in spinel and metallic droplets followed by a mixture of magnesium silicate and metallic droplets. The third layer consists in periclase and metallic droplets. The layers and their compositions are shown in Figure 5 and Table 6, respectively. In fact, there are three phenomena taking place at refractory lining/steel interface: (1) a steel infiltration in the refractory lining; (2) the magnesium silicate reduction according to reaction (1) and; (3) the simultaneous diffusion of silicon to metallic droplets and/or bulk steel.

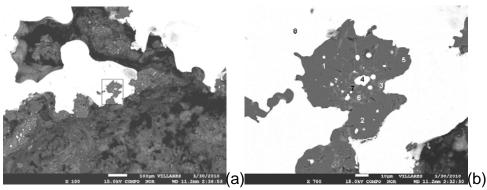


Figure 4. Backscattered electron images. Note the particle which is detaching from steel/refractory interface (gunning mass No. 1).

Table 5. Microanalysis (EDS) from regions shown in Figure 4b

Table 3. Microanalysis (ED3) from regions shown in Figure 46									
Region/wt%	MgO	AI_2O_3	Ca0	SiO ₂	Na₂O	Fe	Cr	Si	Ni
1	29.39	70.61							
2	55.05	0.45	2.61	41.89					
3	20.22	38.27	19.09	21.60	0.82				
4						75.01	7.62	5.10	12.27
5	29.38	70.62							
6	18.91	33.98	20.90	25.62	0.59				
7	13.49	20.81	30.51	34.70	1.09				
8						87.95	8.77	3.28	

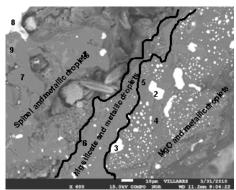


Figure 5. Backscattered electron image (SEM) from steel/refractory interface (gunning mass No. 1).



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Table 6. Microanalysis (EDS) from regions indicated in Figure 5

Regions/wt%	MgO	AI_2O_3	CaO	SiO_2	Fe	Cr	Si	Ni
1	29.50	70.50						
2					71.15	7.04	1.16	20.65
3					69.72	6.56	1.95	21.77
4	100.00							
5	50.95	10.66	2.18	36.21				
6	55.54	0.92	1.68	41.86				
7	29.53	70.47						
8					88.29	8.25	3.46	
9	11.36	8.99	37.71	41.94				

The formation of a steel oxidized layer was observed at the steel/refractory interface for all heats. The mechanism proposed in the literature (17,19) for this steel oxidized layer formation is shown in figure 6. The formation of the steel oxidized layer, which consists in Fe, Cr and Si oxides, is believed to result from the presence of a large difference in oxygen potential between the refractory lining and the steel. In fact, the refractory can be considered as a potential oxygen provider in which the air trapped in the pores and the presence of easily reducible oxides from gunning mass, particularly Na-silicate and FeO, may provide the sources of oxygen.

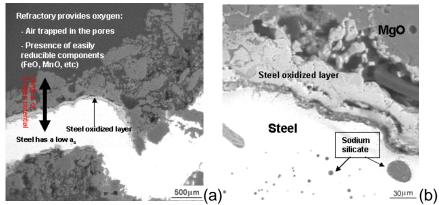


Figure 6. Backscattered electron images (SEM) from steel/refractory interface showing the steel oxidized layer (gunning mass No. 1).

The interaction between the molten steel and gunning mass gives arise to inclusion shown in figure 7, which consists in a spinel particle associated to a calcium silicate phase containing sodium. The same characteristics can be seen in the Figure 8, where a spinel particle (MgO.Al $_2$ O $_3$) associated to a calcium silicate phase containing sodium is still attached to steel oxidized layer. In fact, the microscopic observation has revealed that the steel-refractory interface is always in motion because there are regions with low melting points that are involving the MgO grains (Figure 9). This movement leads to steel infiltration and to the breakdown and/or engulfment of the steel oxidized layer, as well as releases particles from the tundish refractory lining to liquid steel (Figure 10).

In areas of fast steel flow the particles shown in Figures 3 (region 3), 8 and 10b can be carried away into the steel stream. When this happens in the vicinity of tundish nozzle, the high speed steel flow and short residence time prevent the flotation of these particles in the tundish, giving arise to non-metallic inclusion shown in Figure 7.



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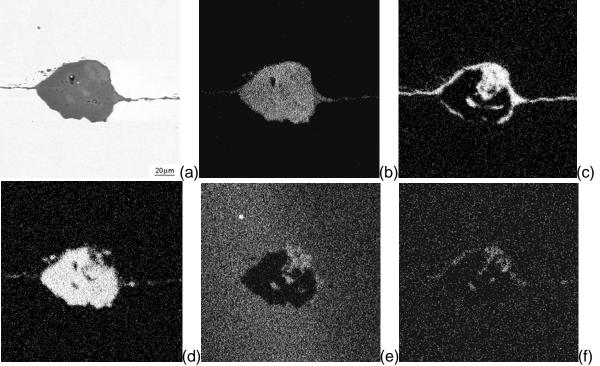


Figure 7. Analysis of a typical non-metallic inclusion which comes from the refractory lining (gunning mass No. 1). (a) backscattered electron image (SEM); (b) Al; (c) Ca; (d) Mg; (e) Si and; (f) Na are X-ray images (WDS).

It is clear that the interaction between molten steel and tundish refractory lining is inherent to the process, mainly in cases where the casting time reaches 120-130 minutes for each heat. In general the size of the particles released from the tundish lining is bigger than 50 μ m, which could be easily removed by flotation if the fluid flow in the tundish is suitable for that. Any attempt to increase the residence time of the molten steel for inclusion flotation, redesign of the flow control devices and the position of the submerged entry nozzle (if it is very close to side wall of the tundish), can contribute to minimize the presence of non-metallic inclusions (final product) that come from the interaction between liquid steel and tundish lining.



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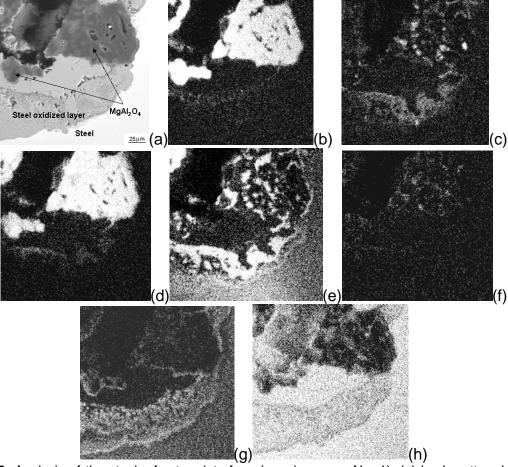


Figure 8. Analysis of the steel-refractory interface (gunning mass No. 1). (a) backscattered electron image (SEM); (b) AI; (c) Ca; (d) Mg; (e) Si; (f) Na; (g) Cr and; (h) Fe are X-ray images (WDS).

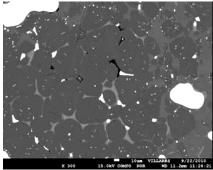


Figure 9. Backscattered electron image (SEM) showing steel infiltration and the MgO grains involved by a phase with low melting point (gunning mass No. 2).

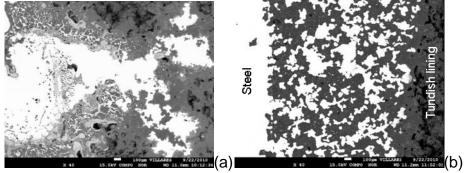


Figure 10. Backscattered electron images. (a) steel oxidized layer engulfed into the tundish refractory lining (steel/gunning interface) and; (b) particles released from tundish refractory lining (gunning No. 2).



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4 CONCLUSIONS

The pre-formed MgO boards were less prone to steel infiltration than gunning materials.

The formation of a steel oxidized layer at steel/refractory interface was observed in all industrial trials. This layer consists in Fe, Cr and Si oxides and is believed to result from the presence of a large difference in oxygen potential between refractory lining and the steel.

The microscopic observation has revealed that the steel/refractory interface is always in motion because there are regions with low melting points which are involving the MgO grains. This movement leads to steel infiltration and to the breakdown and/or engulfment of the steel oxidized layer, as well as releases particles from the tundish lining to liquid steel.

The formation of spinel layer (MgO.Al₂O₃) at steel/refractory interface takes place according to reaction: $3(2MgO.SiO_2) + 4\underline{Al} = 2(MgO.Al_2O_3) + 4MgO + 3\underline{Si}$

This reaction does at first not impair the steel cleanliness because there is a substitution of silica by alumina in the refractory lining. The problem involving this reaction is the spalling of the spinel layer which can influence negatively the steel cleanliness.

When the spinel phase is present, from steel/refractory interface three layers were indentified. The first one consists in spinel and metallic droplets followed by a mixture of magnesium silicate and metallic droplets. The third layer consists in periclase and metallic droplets.

There are several metallic droplets in the refractory lining near to steel/refractory interface. Most of them have high contents of Si, Cr and Ni. Besides the steel infiltration in the refractory lining, the other sources of these elements can be: (1) the silicon comes from the reaction described above, which diffuses to metallic droplet and; (2) the addition of spent refractory from stainless (Cr and Ni) steelmaking to the raw material of the gunning mass.

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