

# AVALIAÇÃO DA PERFORMANCE DE PÓS FLUXANTES PARA AÇOS ULTRA BAIXO CARBONO\*

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#### Resumo

Pós fluxantes desempenham um papel significativo na qualidade do aco durante o processo de lingotamento contínuo. Os pós fluxantes são escórias sintéticas usadas para lubrificar o molde durante o lingotamento contínuo do aço. Para que isso ocorra, o fluxante deve apresentar as seguintes funcões: isolamento guímico e térmico do aco, absorcão de inclusões não metálicas do aço, lubrificação do molde e controle da transferência de calor entre o aço e o molde. O desempenho de cada função está relacionado às propriedades químicas e físicas do pó fluxante, que são dependentes de sua composição química e sua composição mineral, devido a sua relação com a taxa de fusão do material. A fim de garantir uma produção eficiente e de alta qualidade durante o lingotamento de um aco ultra baixo carbono, os pós fluxantes A e B foram investigados de acordo com diferentes propriedades físico-químicas. Diversos experimentos laboratoriais foram realizados para comparar o desempenho dos materiais A e B, visando à diminuição de captura da escória pelo aço no molde: avaliação do comportamento de fusão e fluidez, composição química e viscosidade. As temperaturas de processo foram avaliadas através de testes estatísticos, visando caracterizar o fluxo de calor e o trabalho de fricção durante lingotamento. Os testes industriais preliminares, realizados na Ternium Brasil, foram conduzidos com o objetivo de analisar o comportamento do pó fluxante durante a operação de lingotamento contínuo em relação a alguns aspectos operacionais e fornecer informações sobre sua influência na qualidade superficial do aço. Os resultados mostraram que, aumentando a viscosidade do fluxante, o consumo de pó tornou-se menor, levando a um maior trabalho de fricção e menor fluxo de calor no molde. No entanto, o comportamento da camada de escória mostrou que, apesar do aumento da força de atrito no molde, a espessura da escória foi adequada e não houve relatos de defeitos superficiais durante o uso do material fluxante de maior viscosidade.

Palavras-chave: Pó fluxante; Lingotamento Continuo; Qualidade de Placas; Viscosidade.

# EVALUATION OF PERFORMANCE OF MOULD POWDERS FOR ULTRA LOW CARBON STEELS

#### Abstract

Mould flux plays a significant role in quality during the continuous casting process. Mould powders are synthetic slags used to lubricate the mould during continuous casting of steel. For this to occur, the mould powder has the following functions: chemical and thermal insulation of steel, absorption of non-metallic inclusions from steel, lubrication of the mould and control of heat transfer between the steel and the mould. The performance of each function is related to the chemical and physical properties of the mould powder, which are functions of its chemical composition, and besides its chemical composition also its mineral composition should also be considered, as it affects its melting rate. In order to ensure efficient and high quality production during an ultra-low carbon casting, mould fluxes A and B were investigated according different physico-chemical properties. Several laboratories experiments were carried out to compare the performance of mould fluxes A and B targeting to avoid slag entrapment: evaluation of melting and fluidity behavior, chemical composition, viscosity and temperatures and statistical tests were conducted in order to characterize heat flux and frictional work during casting. The preliminary industrial trials, performed at Ternium Brasil were accomplished aiming to analyse its behavior during continuous casting operation



regarding some operational aspects and to provide information about its influence on the superficial quality of the steel. The results showed that by increasing mould fluxes viscosity, the mould powder consumption became lower leading to higher frictional work and lower heat flux in the mould. However, the slag layer behavior showed that despite the increased frictional force in the mould, the slag thickness was adequate and there were no reports of surface defects during use of the higher viscosity mould flux.

Keywords: Mould powders; continuous casting; slab Quality; viscosity.

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## **1 INTRODUCTION**

Mould powders are mineral mixtures that, in contact with liquid steel, must melt and generate a liquid slag used to lubricate the mould during the continuous casting, and thus generate a better finishing and surface quality of the casted steels <sup>(1)</sup>.



**Figure 1** – Photo of Mould Powders (Source: Imerys Steelcasting do Brasil)

Mould powders perform several functions that are important for the stability of the continuous casting process (2). The duties of the mould powders are to provide (i) strand lubrication through the mould, (ii) uniform heat transfer between steel shell and mould, (iii) protection of the molten steel against oxidation, (iv) absorption of non-metallic inclusions and (v) thermal insulation of molten steel. In addition, it is also clarified that mould powders play an important role on affecting surface quality of Steel (3). All these functions are essential to the good work of casting process, in which lubrication and heat transfer are the most important ones (4). Typically the mould powders are composed by several mineralogical components and carbon – oxides, fluoride and carbonates – and their composition varies according to steel grade and operational parameters.

The main components of mould powders are CaO-SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-Na<sub>2</sub>O-CaF<sub>2</sub> (3).



Figure 2 – Schematic drawing of the various slag layers formed in the mould <sup>(5)</sup>

Regarding the production cost, casting speed should be as fast as possible to achieve high productivity, decreasing the steel cost per tons. On the other hand, high casting speed can cause unstable process conditions in the mould and consequently slab quality defects.

The requirements of steel user have diversified, and properties as better workability, higher strength, stricter surface control, and low levels of defects came to demand.

Minimizing inclusions in the mould is one of the most important challenges in the continuous casting process. Inclusions deteriorate the mechanical properties of steel under tension, bending, press forming, and other types of working and cause surface defects. (6).



The in-mould entrapment of slag, oxides and gas bubbles remains a serious problem, especially as casting speeds are raised to increase productivity. Mould slag, endogenous non-metallic inclusions (e.g.  $Al_2O_3$ ) and gas bubbles get trapped by the newly formed shell in the meniscus region. Slag entrapment has become much more important in recent years with the use of increased casting speeds to meet productivity demands. The velocity and turbulence of the metal flow in the mould is a key factor in all three forms of entrapment (7).

Mould slag (or mould flux) entrapment is characterized by mould powder being drawn into the molten steel pool inside a continuous casting mould. Mould slag entrainment can cause both surface and internal defects in the final product if the entrained droplets become trapped in the solidifying metal, which makes it a significant problem in the production of clean steel (8).

Surface quality of continuously cast is strongly influenced by the interfacial tension between steel and mould flux slag. The meniscus shape and the inclusion entrapment are directly determined by interfacial tension (9).

Figure 3 shows how and why slag entrapment can happen:



Figure 3 – Slag Entrapment (10)

The surface tension of molten slag is one of the most important parameters for controlling the various interfacial phenomena in the refining or continuous-casting process of steelmaking. However, it is not often possible to find appropriate data on the surface tension of molten slag targeted for specific purposes, because molten slag is usually a multi-component oxide, sulfide, and fluoride system, where the effects of these components on the surface tension is very complicated, and not all the data (particularly over a wide temperature and composition range) have been reported.

One of the main problems found in steels produced by continuous casting is the presence of non-metallic inclusions, which are often derived from the drag of slag to the liquid steel in the mould during the casting process (11).

This drag is directly related to the level of turbulence that develops at the metal-slag interface.

The main factors responsible for this turbulence are (11):

## 1.1. Depth of the submerged entry nozzle (SEN)

Low valve depths cause instability in the meniscus with the entry of liquid steel into the mould. This instability increases turbulence, which can lead to the drag of slag to steel.





Figure 4 – Schematic drawing of the various slag layers formed in the mould <sup>(11)</sup>

Figure 4 illustrates the drag occurring due to low immersion depths of the submerged nozzle. It can be observed that the liquid steel, when it leaves the submerged nozzle, shears the slag layer nearby, causing drops of slag to be drawn.

## 1.2. Submerged Nozzle Geometry:

The direction of the angles of the submerged nozzle outlet is directly connected to the drag. Angles in the downward direction (negative angles) increase the flow intensity in this direction, decreasing the recirculation of the steel at the top of the mould and, consequently, reducing the turbulence at the steel-slag interface, reducing the possibility of occurrence drag.

## 1.3. Argon Injection:

The flow of injected argon must be controlled, because there is a critical flow above, which the gas bubbles cause turbulence at the steel-slag interface, which can lead to drag.

Figure 5 shows a typical sequence of an argon bubble resting at the metal-slag interface at 1450°C. This picture shows the metal interface deforming and the metal layer entrapped between the bubble and the slag layer (12).





Figure 5 – Argon bubble resting a slag-metal interface <sup>(12)</sup>

## 1.4. Casting Speed:

High casting speeds cause an increase in surface turbulence in the mould, where this increase of turbulence causes vortexes to emerge near the submerged nozzle, which can result in drag.

1.5. Variation of Steel Level in the Mould:

Reductions of the steel level in the mould can cause vortexes close to the submerged nozzle, which can result in the slag layer being detached to the liquid steel.

1.6. Physical Properties of Mould Powders:

The properties of the mould powders that affect slag entrapment are the viscosity and interfacial tension between steel and slag. Regard to viscosity, it is desirable that would be high enough to reduce surface turbulence and prevent slag drag. However, the property that is most effective in reducing the drag of slag is the interfacial tension between steel-slag. As higher the interfacial tension, as lower the chances of dragging.

In relation to the mould powder, it must be formulated in order to possess certain levels of elements that contribute to increase this interfacial tension.

In 1999, Stollberg Germany together with the University of Freiberg made a study on the effect of the chemical composition of the flux powders on the interfacial tension between steel and slag (13).

Among the results found, it was concluded that the steel-slag interfacial tension is increased with:

- Increase of Al<sub>2</sub>O<sub>3</sub>, MnO and MgO contents in the mould powder;

- Reduction of Na<sub>2</sub>O and CaF<sub>2</sub> contents in the mould powder.



## Figure 6 "a" to "e" illustrates the results found in the paper:









Figure 6 – Effect of % elements in interfacial tension <sup>(13)</sup>

Process issue and resolution strategy

Ternium steel plant is currently using the material "A". When it is applied in low casting speed, the product shows good performance, however when it is applied in high speed = 1,6 m/min, steelplant have problems with slag entrapment. So, the powder was adjusted for high speed, to avoid slag entrapment and was named, material "B".

The adjustments done were to increase: viscosity and interfacial tension.

The aim of the present work is to compare the performance of mould fluxes "A" and "B" targeting to avoid slag entrapment in ultra low carbon steel slab casting, performing laboratory tests and industrial trials, considering the technological parameters: evaluation of melting and fluidity behavior, chemical composition, viscosity and temperatures. Considering the steel casting parameters: evaluation of heat flux, mould friction, slag layer thickness and powder consumption.



# 2. PROCEDURE

- 2.1. Laboratory tests
- 2.1.1. Determination of chemical composition

The chemical composition of the product and slags was determined by the X-Ray Fluorescence technique (15). Figure 7 shows the X-ray fluorescence equipment.



**Figure 7** – a) X-Ray fluorescence analysis by the X-Ray spectrometer Axios 4KW (Panalytical), b) fused bead formation (Source: Imerys Steelcasting do Brasil)

# 2.1.2. Boat test

Boat Test is a test that evaluates the melting rate of mould powders, see Figure 10. Based on the visual inspection and the experience of the analyst, comparing the sample being tested with a pre-defined standard sample, using the inversely proportional relation between the necessary time for the sample to melt. For this test, a porcelain boat is used at a temperature of approx. 150°C above the melting temperature in different times: 1, 3 and 5 minutes, until the complete melting of the product. The analysis is made comparing visually the melting of the slag (liquid flux) of the sample with the one obtained by the standard.

## 2.1.3. Ramp test

It is a standard test for the mould powder industry, see Figure 12, which evaluates the fluidity of the fluxes based on visual inspection and on the experience of the analyst, comparing the test sample with a pre-defined standard sample. The Ramp Test allows making a comparative qualitative analysis of the fluidity of various flux powders. For this test, a porcelain boat is used at a temperature of approx. 150°C above the melting temperature in different times: 1, 3 and 5 minutes, until the complete melting of the product. The analysis is made comparing visually the extension of the trail formed by the slag (liquid flux) in the boat with the sample with the one obtained by the standard (14, 15).



## 2.1.4. Melting Temperature

For the evaluation of melting characteristics it was used a heating microscope, increasing the temperature at 10 °C/min, obtaining the characteristic temperatures. The working method refers to the German standard DIN 51730 (16).

#### 2.1.5. Viscosity

The viscosity for the mould powders was calculated through the IRSID model (19) at 1300 °C. Viscosity measurements were performed using a rotation viscometer using for high temperatures according to international standards. After sample preparation (decarburized at 1350 ° C and grinded to obtain granules with 2-5 mm of diameter) and the preparation of the measuring device, the procedure is executed automatically. A computer calculates and prints the graphic "viscosity [Pa s •] versus temperature [°C]". From the liquid mould slag, a cooling rate of 10 °C/min was applied, measuring the viscosity at a certain rotation speed.

## 2.2. Industrial tests

At a Brazilian steel plant, Ternium Brasil, industrial tests were performed during continuous casting of slabs.

During the casting of ultra-low carbon steel grades, mould fluxes "A" and "B" were tested at the same time, but in different strands, to compare the main process parameters in the mould during casting.

The tests were performed in 46 heats (15600 tons of steel), for different mould dimensions and casting speeds. In order to study and determine the average heat flux, and friction behavior of the fluxes A and B, 20 heats were performed with the same mould dimensions, aiming the casting speed between 1,5 and 1,6 m/min.

During the tests, slag pool thickness and mould flux consumption were measured. The powder consumption is frequently determined as the number of bags of known mass of casting powder used in the entire cast, for which the total mass of steel cast is known (7). Thus powder consumption can be calculated and correlated with the slag pool measurement.

Heat flux and mould friction were measured throw the Mould Expert System ® (Primetals). The Mould Expert is an on-line mould monitoring and partly control system that enables a qualitative and quantitative insight into the inner life of the mould. Observing the critical area of mould, thermocouples embedded in the copper plates give valuable thermal information of the early solidification. These data are used for breakout prevention as well as for interpretation of heat transfer inside the mould. Regarding the mould friction, an online monitoring system shows the friction information and may, therefore, be used to optimize the casting process and its parameters. A dramatic change in friction stresses might indicate critical situations.

In order to characterize the mould fluxes behavior, statics tests were conducted using the software Statistica®. The hypothesis t-test was used to compare and determine the difference of heat flux and friction between mould flux "A" and "B".



## 2.2.1. Slag pool thickness and melting performance

The slag pool thickness is related to the balance between the mould powder consumption and liquid slag generation. Failure to maintain it can lead to lack of lubrication and ultimately to surface defects or sticker breakouts. The thickness of the slag pool influences on the amount of liquid slag infiltrating into the mould/strand gap and the number of inclusions transferred from the steel to the slag (20).

For doing slag pool thickness measurements steel wire and copper wire are simultaneously immersed in the mould according to Figure 8. The temperature for the liquid slag in contact with the steel is ca. 1350°C, enough to melt the copper wire. However, this temperature is not enough to melt the steel wire. So, the difference between the lengths of the wires after the immersion represents the slag pool thickness (L1-L2). The total length of the steel wire represents the total layer (powder in nature, sintered and liquid), assuming that the wire will be melted by getting into contact with the liquid steel.

To fix the wires a standard measurer was developed. It consists of a retractable aluminum rod that allows the fixation of the wires in its extremity, guaranteeing herewith the safety of the operator during the measurements. The immersion time is ca. 5 seconds (it depends on the diameter of the used wires).



Figure 8. Slag pool thickness measurements

#### 2.2.2. Inclusions absorption

During the steel production process, depending on the treatment, variations in the quantities of oxides, silicates, aluminates and sulfates absorbed by the flux powder can occur, and in case these inclusions get caught in the skin solidification, they can cause sub-superficial, superficial defects and cracks. The main non-metallic inclusion absorbed by the powder is the alumina (Al<sub>2</sub>O<sub>3</sub>); despite not being the only inclusion in balance with the steel, it is responsible for the worst effects due to its refractoriness characteristics. Flux powders must absorb the maximum of alumina from the liquid steel in the mould, since the inclusion of alumina in the steel reduces the quality of the molten steel.

The slag samples have been collected during the steel casting and their compositions were determinate by X-ray fluorescence (item 2.1.1)



## 3. RESULTS

## 3.1. Laboratory tests

Laboratory tests were performed comparing powder "A", reference material and powder "B", adjusted material, which are produced by Imerys Steelcasting and used at steelworks for slab casting. The necessary tests are reported as following.

## 3.1.1. Chemical composition

The compositions of mould powders "A" and "B" were determined through X-ray fluorescence and shown in Table 1.

	Mould Powders			
Elements	Α	В		
	Reference	Optimized recipe		
Binary Basicity (CaO/SiO <sub>2</sub> )	1,05	1,05		
$MgO + Al_2O_3 + Fe_2O_3$ (wt%)	12,0	16,0		
Na <sub>2</sub> O + F (wt%)	12,0	10,0		
Carbon free (wt%)	1,90	1,90		
Temperature Softening (°C)	1174	1173		
Temperature Melting (°C)	1178	1186		
Temperature Flowing (°C)	1180	1189		
Irsid Viscosity 1300°C (Pa.s)	0,15	0,25		
Interfacial Tension (mN/m)	260,66	292,77		

 Table 1: Compositions and some technological parameters of the mould powders A and B

Powder "B" presents higher viscosity and higher interfacial tension than powder "A".

#### 3.1.2. Melting characteristics

#### 3.1.2.1. Temperatures

According to the results in Table 1, the both powders have similar characteristic temperatures.

#### 3.1.2.2. Boat Test

Figure 9 shows the result of the Boat Test for the products "A" and "B", made at a temperature of 1240°C, during 1, 3 and 5 minutes.





Figure 9 - Photos of boat test of products "A" and "B" during 1, 3 and 5 minutes

From the results shown in Figure 9, the melting rate of "B" is similar in relation to "A", since:

- In the time of 1 minute, the melting process begins in both samples;
- In the time of 3 minutes, samples A and B are almost totally melted;
- In the time of 5 minutes, samples A and B melted completely

So, no changes were expected in the melting speed and slag pool thickness during use of powder B at steelplant, which were confirmed during industrial trials.

## 3.1.3. Fluidity behavior

#### 3.1.3.1. Viscosity

Figure 10 shows the result of the Viscosity Test for the products "A" and "B".



Figure 10 – Measured viscosity of the products "A" and "B"  $\,$ 



Table 2 shows the values determined by the viscosity analysis.

	Material				
Characteristics	A - reference	B - optimized recipe			
Irsid viscosity of Mould Powders at 1300°C (Pa*s)	0,145	0,212			
Break Temperature (°C)	1140	1140			

Table 2: Measured viscosity of the mould powders "A" and "B"

The viscosity of mould powder "B" is higher than than "A", which was expected as "B" has lower Na<sub>2</sub>O and lower F than "A".

The Break Temperatures for both products are the same. The break temperature is the point where there is a sudden increase of the viscosity due to crystals precipitation (during cooling).

## 3.1.3.2. Ramp Test

Figure 11 shows the result of the Ramp Test for samples A and B, made at the temperature of 1240 °C, in 1, 3 and 5 minutes.



**Time (minutes)** Figure 11 – Photos of ramp test of products "A" and "B" during 1, 3 and 5 minutes

In Figure 11, it can be observed that in the time of 1 minute, the samples "A" and "B" have a similar displacement of melted material. In the time of 3 and 5 minutes, a difference between the distances covered by the melted materials is observed, showing that "B" covers a lower distance than "A", that is "B" shows a lower fluidity than "A".

According to Figure 10, at temperature of 1300°C, that is the average temperature in the liquid slag layer, the viscosity of "B" is higher than "A". As described in Table 1, sample B shows lower quantity of Na<sub>2</sub>O (2,3%) in relation to sample A (3,3%). The effect of Na<sub>2</sub>O in viscosity behavior was also observed by Schulz et al (23). This



result is favorable in the continuous casting process of ultra low carbon steels to avoid slag entrapment.

#### 3.2. Industrial tests

#### 3.2.1. Heat Flux

The graphs below show the heat flux behavior according to the casting speed in the mould.



Figure 12 – Graph of Heat Flux x Casting Speed Scatterplot comparing Powder "A" and "B" (Statistica ®)

High casting speeds steel grades have lower solidification time in the mould, in other words, the thickness of the solidified shell is smaller as the steel residence time inside the mould decreases. The ferrostatic pressure strains the shells against the mould, increasing the local heat flux.

As shown in Figure 13, casting powder "A" and "B" demonstrate a linear behavior according to the casting speed. In order to characterize the difference between casting powders heat fluxes, the hypotheses t-test were conducted, as shown below. For the statistical analyses were selected the slab data for casting speed between 1,5 to 1,6 m/min and same mould dimensions.



Figure 13 – Graph of Heat Flux comparison throw t-test and data results (Statistica ®)



Through the hypothesis t-test, it is possible to conclude that there is a significant difference between the heat flux results in the mould presented by flux "A" and "B". Based on the obtained results, the hypothesis of equal means is rejected (p-value <0.05). Therefore, it can be stated that the heat flux work presented by flux "B" is more significant than "A".

The obtained result can be explained by the increase in viscosity, which leads to the generation of a thin slag film between steel and mould, due to the lower fluidity of the high viscosity mould flux, leading to a lower thermal resistance imposed by this film and consequently more heat extraction by the mould.

## 3.2.2. Mould Friction

The Figure 14 shows the mould friction behavior according to the casting speed.



Figure 14 – Graph of Mould Friction x Casting Speed Scatterplot comparing Powder "A" and "B" (Statistica ®)

According to MILLS, 2017 (8), the liquid friction force is inversely dependent upon the powder consumption and directly related to the slag viscosity and the difference between the velocities of the mould and the strand.

As the slag pool thickness keep results between 15 and 20 mm, for all the casting speeds, a strong relationship between friction and velocity was not verified during the tests performed.

In order to characterize the difference between casting powders frictional forces, the hypotheses t-test were conducted, as shown below. For the statistical analyses were selected slab data for casting speed between 1,5 to 1,6 m/min and same mould dimensions.



Figure 15 – Graph of Mould Friction comparison throw t-test and data results (Statistica ®)

The t-test results shows that the hypothesis of equal means is rejected (p-value <0.05). This result is expected, as soon as the mould flux increase, the consumption decreases (8). The lower lubrication, generated by the lower flux consumption, leads to increased mould friction. As concluded by the t-test, but without any negative impact on casting conditions.

#### 3.2.3. Mould powder consumption

The average mold powder consumption was 0,35 kg/t, which could be considered enough for good slab casting lubrication.

## 3.2.4. Slag pool thickness and melting performance

The slag pool thickness measurements results are the following: for the A and B 15-20 mm. It means that for this parameter the mould powder B is adequate.

It was visually observed that the melting performance of the B mould powder is similar to A. The melting behavior depends on chemical and physical properties, like free carbon,  $CO_2$  content, bulk density, and choice of the raw materials. The melting rate has a significant effect on powder performance since it determines the ability of the molten slag to maintain a stable liquid pool depth.

#### 3.2.5. Inclusions absorption

During casting using the mould powder "B", 8 mould slag samples were collected and submitted to X-ray fluorescence analysis using an apparatus Panalytical Axios 4KW to determinate  $Al_2O_3$  contents. One sample was collected per heat. Table 3 shows the result.

Al <sub>2</sub> O <sub>3</sub> %									
Granules	Slag samples of "B" / Heat								
of "B"	1	2	3	4	5	6	7	8	
6,90	10,10	11,50	10,42	12,02	11,97	11,58	10,83	10,79	

Table 3. X-ray fluorescence results for Al<sub>2</sub>O<sub>3</sub> content of mould slag samples for B powder [wt%]



As reported before,  $Al_2O_3$  is the main absorbed non-metallic inclusion, and this absorption by the reference material is around 6,0 %, and as can be observed in above table, the "B" material absorbed an average of 5,0%, close values.

3.2.6. Material rejection rate

For high casting speed, we had a significant laminated volume, where there was a 78,6% reduction in material rejection rate.

## 4. CONCLUSION:

In mould slag entrapment is dependent of slag viscosity and liquid slag/steel interfacial tension. As interfacial tension and viscosity increase, liquid slag is more difficult to be emulsified and, by the way, be a source of defects. The product "B" presented higher viscosity and higher interfacial tension, keeping stable the other important characteristics of mould slag as the melting behavior.

Regarding the following evaluations, the results were similar between mould powders A and B: slag pool thickness, melting behavior. It means that these slag modifications did not impact the casting process. There was a higher reduction in material rejection rate for high casting speed. Therefore, the first steel quality evaluation showed improvement due to these changes and a long-term quality evaluation has started.

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