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DEVELOPMENT OF LABORATORY TESTS TO EVALUATE STICKING TENDENCY[1]

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Abstract
One of the most important risks that appears in the process of direct reduction, is the tendency to the clustering or sticking that the pellets experience at high temperature. The Sticking test allows to predict the behaviour of these pellets against this phenomenon. TenarisSiderca asked Instituto Argentino de Siderurgia (IAS) to jointly develop a new methodology for the determination of the Sticking and Cluster as the standard test did not represent what happened in the operation. In these tests, pellets are reduced, carburized and cooled with both temperature and gas composition profiles simulating the reducing furnace operation. For over 20 years, a testing methodology with a maximum temperature of 840ºC has been used. However, reducing conditions have changed considerably due to different improvements implemented in the process, which enabled a very significant increase in plant productivity. For this reason we have developed a new test adapted to the present operating conditions regarding the residence time of the material, composition and temperature of gases and final quality of sponge iron. This new test allowed the identification of samples with most sticking tendency, this behavior could not be appreciated with traditional tests at 840°C. Besides, it was verified the positive influence of the coating applied in plant. Many samples were studied from different sources.
Key words: Sticking; Coating; Direct reduction.

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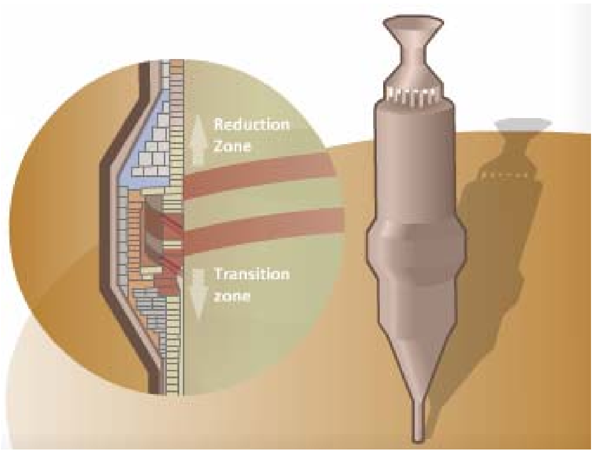
1 INTRODUCTION

The Midrex Direct Reduction process is based on reducing iron ore by reducing gases to produce sponge iron or direct reduced iron (DRI) in a reducing furnace or reactor. The main raw material used is iron ore pellets. This material is manufactured in the form of small balls, porous and resistant from milled and pre-concentrated iron ore.
The chemical, physical and metallurgical characteristics of the iron ore used in direct reduction are very important for the proper operation of the furnace and production of a reduced pellet (DRI) of good quality.
A very important variable in the reduction process is the temperature of the reducing gases when entering the furnace, since it depends on its performance. In order to produce high quality sponge iron in an efficient direct reduction plant, the temperature of the reducing gas or gas bustle must be maximized, without causing agglomerates or clusters. This temperature is currently between 960 and 980°C. The clusters are one of the major risks in this type of process. They are small pieces of pellets stuck together or bonded in a bunch, which can increase their size obstructing the flow channel of material and / or reducing gases in the furnace, causing a significant efficiency decrease and thus the productivity of the plant is affected. In the extreme case, these clusters could completely prevent the discharge of sponge iron, making the operation stop in order to free the obstruction, being in some cases a very complex and prolonged operation. Figure 1



Reduction Furnace sketch, Nozzles of Bustle Zone


A crown of Cluster plugging the Nozzles.


Flagstone clusters separated from the wall to remove and clean the nozzles.


Oversize derivation point with medium size clusters.

Figure 1. Clusters

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The sticking/ clustering test predicts the behavior of the pellets against this phenomenon and its results are of great importance in assessing the reliability of the raw material to be used.

In the late '80s, TenarisSiderca and IAS (Argentine Iron and Steel Institute) jointly developed a procedure for the determination of the Sticking and Cluster Index. In that test the pellets were reduced, carburized and cooled with both temperature and gas composition profiles simulating the reducing furnace operation. The maximum temperature of the reducing gases was 840°C and the sample was tested without coating.

Currently, the module operating conditions have changed considerably. Several improvements implemented in the process, such as the coating and oxygen injection, have allowed a significant increase in plant productivity. These improvements made possible to enter hotter gases to the reducing furnace, as a consequence the operating temperatures have increased, residence times have been reduced and thus the plant productivity. Taking this into consideration there is a need to adapt the Sticking Cluster test to simulate the current operating conditions and to obtain representative result of the material behaviour within reduction furnace.

Once again TenarisSiderca and IAS are working together in the challenge of achieving a new test. The maximum temperature of the reducing gases resulted from 980ºC and the sample is tested with and without coating. The residence time, temperatures and the gas composition used are similar to the current operating conditions.

As part of the starting up of the method, several tests were carried out where repeatability of the results was achieved. The metallization and carbon percentages achieved by the sponge iron were similar to those achieved in the direct reduction module. Thus the sticking test should provide more representative results.

It was essential to develop a uniform coating method to the pellet sample to be tested. Ensuring a uniform and identical coating on each application was the greatest challenge to face the new method.

Below, the two IAS methods are described for determining the cluster and sticking index: The traditional method, called IAS (840ºC), reaches a maximum temperature of 840°C and the recently developed method, called IAS (980ºC), where the maximum temperature reached is consistent with the current operating conditions. Besides, the IAS methods are compared to the standard ISO 11256: Determination of the Clustering Index.

2 MATERIALS AND METHODS

IAS methods attempt to simulate the longitudinal path downstream of the ferrous raw material within the module reactor. For this reason the IAS methods include three stages: Reduced, Transition and Cooling.

2.1 IAS Method (840ºC)

2.1.1 Stages

The method consists of different stages:

- Preheating: From room temperature to 300°C, with N_2.
- Reduction: This stage is divided in a series of reduction steps, where the reducing gas is enriched with H2 and CO ,reducing gases, and becomes poorer in H_2O and CO_2, gases produced by the reduction. The temperature is

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increased, reaching a maximum temperature, 840°C, which was the inlet temperature of the reducing gases in the industrial reactor some years ago.

- Carburation or also called Transition phase: Here starts the cooling of the burden, and carbon deposition on the surface of the ferrous raw material. The temperature decreases from 840°C to 450°C.
- Cooling: from 450°C to room temperature, with N2.

2.1.2 Gas composition and temperature profile at different stages

Table 1 shows gas composition at different stages and Figure 2 shows the temperature profile.

Table 1. Gas concentration at each stage, IAS Method (840ºC)

	Mix	$\%N_2$	$\%H_2$	%CO	$\%CO_2$	$\%CH_4$	$\%H_2O$
Preheating	**A**	100	-	-	-	-	-
	B	-	44	20	17	3,5	15,5
	C	-	44	20	17	3,5	15,5
Reduction	**D**	-	47	24	13	3,6	12,4
	E	-	50	27	9	3,8	10,2
	F	-	53	30	6	4	7
	G	-	55	33	2	4	6
Carburation	**H**	10	27	10	13	39	-
Cooling	**I**	100	-	-	-	-	-

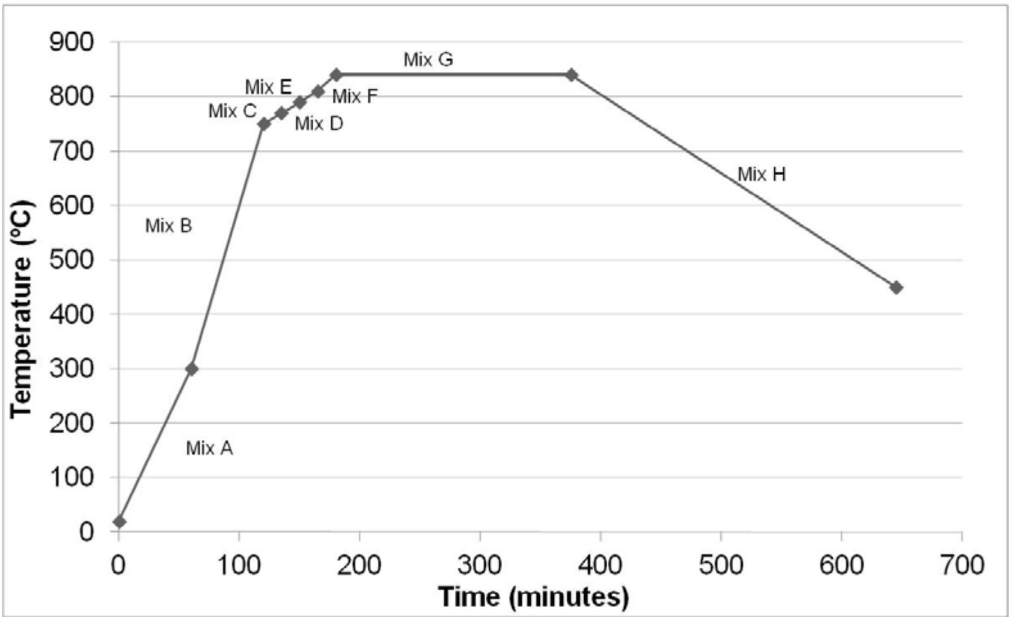

Figure 2. Temperature profile of IAS method (840 ºC)

2.2 IAS Method (980ºC)

2.2.1 Stages

Based on current plant data the new obtained from the new test parameters were designed. The gas composition remained fixed and temperatures, times and gas flow were varying to achieve the optimization of the technique. Then, the setting test was established.

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The method consists of different stages:

- Preheating: From room temperature to 300°C, with N2.
- Reduction: Formed by several steps of reduction, gas composition and temperature to simulate the downstream of the ferrous raw material in the reducing furnace. The temperature is increased, reaching a maximum temperature of 980°C.
- Carburation, also called Transition phase: The temperature drops from 980°C to 700°C, with a gas composition rich in methane.
- Cooling: Up to 600°C, with a gas composition rich in methane and small amount of H2.
- Cooling to room temperature, with N2.

2.2.2 Gas composition and temperature profile at different stages.

Table 2 shows the gas composition at different stages and Figure 3 describes the temperature profile.

Table 2. Gas concentration at each stage, IAS Method (980 ºC).

	Mix	$\%N_2$	$\%H_2$	%CO	$\%CO_2$	$\%CH_4$	$\%H_2O$
Preheating	**A**	100					
Reduction	**B**	-	41,3	23	13,4	3,3	19
	C	-	43,7	25,1	11,3	3,4	16,5
	D	-	51	31,7	5,1	3,8	8,4
	E	-	54	34,5	2,5	4,0	5,0
Carburation	**F**	1	-	-	1,5	97,5	-
Cooling	**G**	-	14	1	5	79,5	-

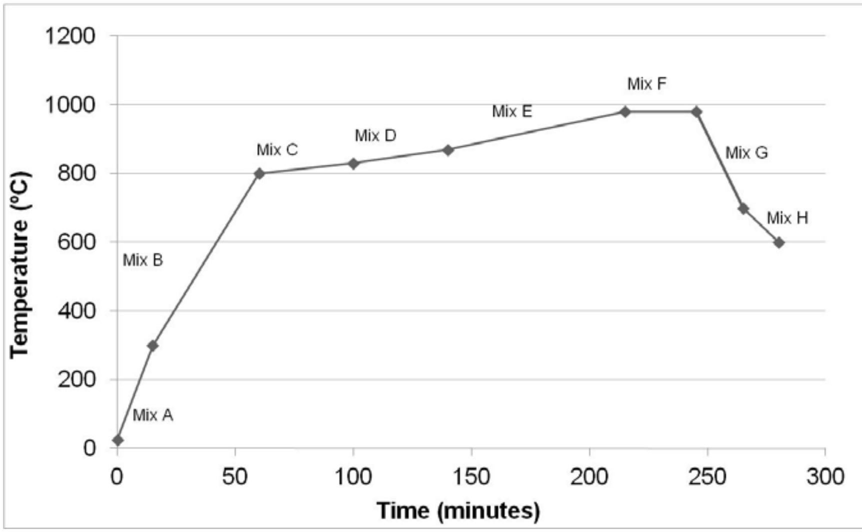

Figure 3. Temperature profile of IAS method (980 ºC)

2.3 Coating Technique

To find a suitable laboratory method of coating three comparative techniques were studied.

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2.3.1 Spray coating

A spray gun is used. During the spraying, the pellets are placed on a screen which is moved circularly in order to promote a uniform coating. Cold and preheated pellets are used to promote coating dispersion.

2.3.2 Coating applied in bag

The sample is placed inside a polythene bag along with the suspension to be applied. It is stirred until there is no remaining suspension inside the bag.

2.3.3 Applied coating simulating the conveyor

The pellets are dropped onto a plane slightly inclined and the suspension is added to form the coating.
Direct observation of the obtained samples is made to evaluate the surface aspect in each method of application. Figure 4 shows that the most uniform coating is achieved by spray coating both in cold and preheated pellet. Coating applied in bag and the applied coating simulating the conveyor provide a more heterogeneous coating, in some cases with a great amount of lime in certain parts of the pellets or no coating at all.

Figura 4. Pellets aspect with different coating methods.

A microscopy study was carried out to compare different coatings. Figure 5 shows each coating thickness.

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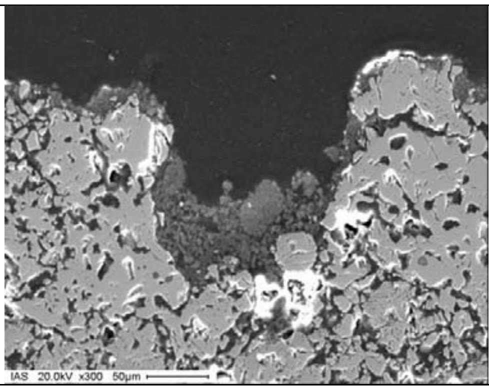
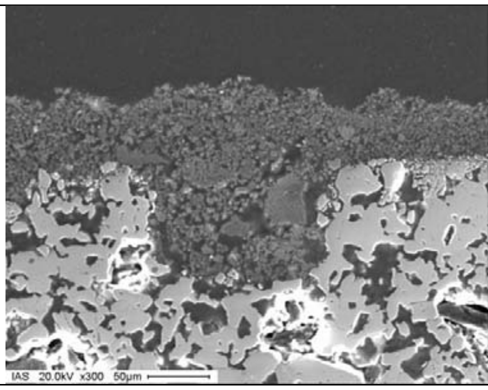
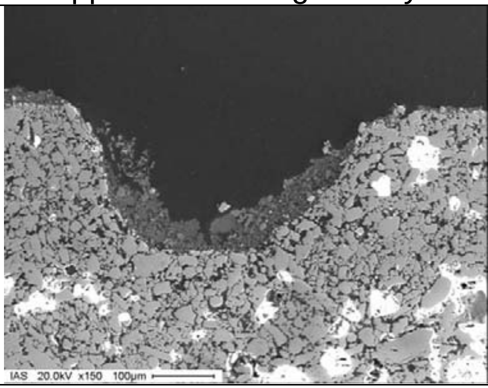


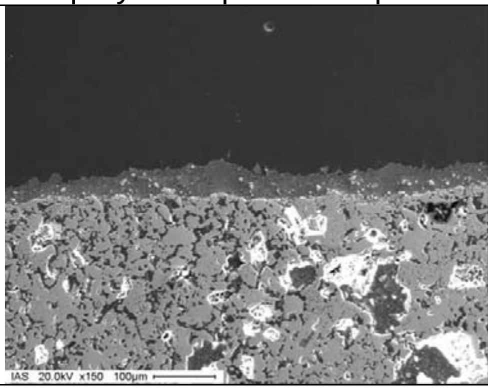
Applied simulating conveyor



Sprayed on preheated pellet



Applied in bag



Sprayed on cold pellet

Figure 5. Coating thickness in the different methods used.

The most uniform coating regarding the layer thickness, hiding power and adhesion on the surface of the pellet is achieved with the spray method. The application technique chosen will be the spray on cold pellets.

2.4 Process of the Tested Sample and Results Expression of the IAS Method

The process of the tested sample consists of the following stages:
• The reduced sample is weighed: **Wt**.
• The sticked fraction is then separated, sieves for 2 minutes with vibrating equipment and the fraction which remains sticked is weighed: **Ws**.
• This material is subjected to the microtumbler for some time and then the material that remains sticked is separated and weighed: **Wc**.
With these measurements the main results of the test are obtained:

2.4.1 Sticking Index

(IS): Ws/Wt x 100

2.4.2 Clustering Index

(IC): Wc/Wt x 100

Furthermore, the metallization index (Met = (Fe ° / Fe Total) x 100) and the percentage of carbon is determined on the reduced sample.

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2.5 Comparison with the ISO method

The technique described in ISO 11256, to assess the clustering index, uses a constant temperature and gas composition. The IAS methods consider thermal profiles and a variable gas composition simulating different stages in the reducing furnace. Table 3 compares the main characteristics of each method.

Table 3. Main methods characteristics: ISO 11256 – IAS Method

	ISO 11256	IAS Method
Reactor	Cilindrical (inner diameter: 125 mm), vertical	Cilindrical (inner diameter: 53 mm), vertical
Sample to be tested	2000 g pellets (50%: -12.5 + 10 y 50%: -16 + 12,5)	500 g • pellets (50%: -12,5 + 9,5 mm y 50%: -16 + 12,5 mm) • lump (-19 +16 mm)
Temperature	850ºC (constant)	Variable. Max Temp.: 840 / 980ºC
Reducing gases	30%CO, 15%CO2, 45% H2, 10%N2 (constant)	Variable Composition.
Gases flow	40 l/min	15 l/min
Duration of the	Until reaching a 95% of the material reduction.	Constant.

3 RESULTS AND DISCUSSION

To compare the advantages of the new and the old method, results of seven samples from two different suppliers are presented: A and B. Sticking tests were performed by IAS Methods: 840°C and 980ºC (with and without coating). The following figures, 6 and 7, summarize the results:

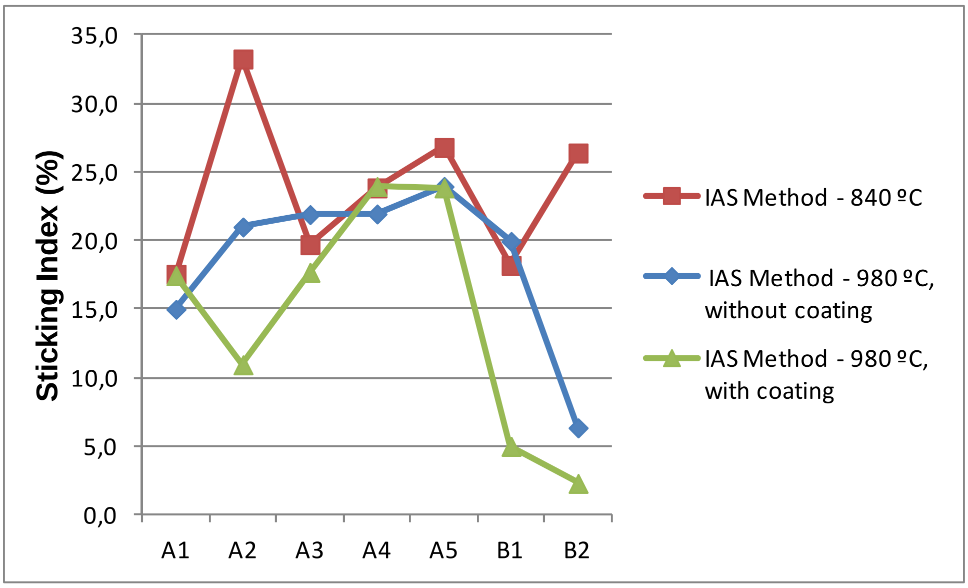

Figure 6. Sticking index results.

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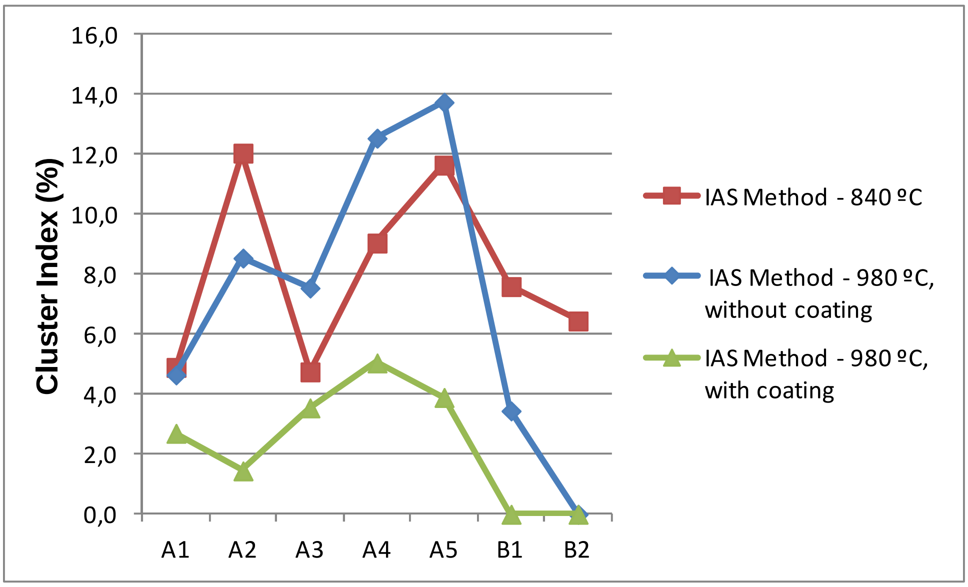

Figure 7. Cluster index results.

The IAS method 840ºC results allow to identify the samples A2 and A5 with a strong sticking tendency. However, the IAS method 980°C indicates that the samples A4 and A5 have the greatest tendency to sticking. A4 and A5 samples also showed a high tendency to sticking in plant.
Samples B, under the IAS method 980°C, have a less sticking tendency compared with samples A. This behavior is not reflected under the IAS method 840°C. The coating application over the tested samples at 980°C produces a significant decrease in the sticking tendency. The effect of this coating on samples B is greater compared with samples A, since they make no cluster formation.

4 CONCLUSIONS

- The new IAS test method (980 ºC) permits to evaluate the sticking tendency of the ferrous raw material, with the current operating conditions of the reducing furnace.
- A special feature of this test regarding the existing ones, is that it simulates the various stages at which the burden is subjected to: reduction, transition and cooling.
- A uniform and repetitive coating technique was achieved in order to ensure similar applications for all tests and not to be a factor of variation in the results.
- Sticking test at high temperature help distinguish samples with high sticking tendency, not identified with traditional tests at lower temperatures.
- The samples which showed a high sticking tendency in the laboratory test, so did at the plant.

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