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Review and planning of experiments with steel and slag in laboratory furnace[☆]



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ABSTRACT

During the steel secondary refining, the elimination of impurities, adjustment of the chemical composition and adjustment of the temperature of the liquid steel occurs. The removal of non-metallic inclusions usually occurs in this step, being performed by the slag. In this work, a planning of laboratory experiments was defined so that in a second stage, the obtained knowledge can be compared with previous studies and transferred to the industrial practice. First, a review of the literature about experiments carried out in laboratory furnaces with samples with steel and slag in studies focused on the removal and/or modification of inclusions was done. Then, two experimental tests were conducted to validate the configuration adopted in the first part of this work. In the optimized experimental arrangement, the experiments will be performed in a resistive furnace with protective atmosphere granted by argon passage. The samples will be melted in MgO crucibles at 1600 °C and maintained by 90 min at this temperature to reach the thermodynamic equilibrium. The samples will be cooled with water and then prepared for future analyses. The review showed a promising experimental arrangement that will be used as a standard for steel and slag studies carried out in the Ironmaking and Steelmaking Laboratory, at UFRGS. In relation to the experimental tests, the results found agreed with the literature and thus, the experimental arrangement for future studies was validated. The future experiments will be done in order to achieve the optimized arrangement.

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1. Introduction

The secondary refining process is one of the most important stages to obtain a high quality product. During the refining process occurs the removal of impurities, chemical composition adjustment and control of temperature of the liquid steel. In this process it is used a slag, which has a very important impact in the control of those factors. The main functions of the secondary refining slags are: absorb impurities [1,2], avoid reoxidation [3] and ensure protection against heat loss [3]. Two types of slag systems are commonly employed in this step: the CaO-SiO₂-Al₂O₃-MgO(-CaF₂) and the CaO-SiO₂-Al₂O₃-MgO [2,4].

the influence of the chemical composition of the slags in the steel cleanliness [1,2,7,9–11].

In order to obtain a better understand and future improvements in the industrial process, a good alternative is to execute experiments with steel and slag in a laboratory furnaces. In these experiments, it is possible to study the different properties of the slag, such as its binary basicity, alumina wt.% (Al₂O₃), silica wt.% (SiO₂) and MgO wt.% content. With a better control of these properties, it could be easier to understand their effects on the control of inclusions. A good strategy is to reach the initial knowledge in the laboratory, and then, subsequent tests in the industry to validate the results obtained [8,12–15]. The objective of this work was to design

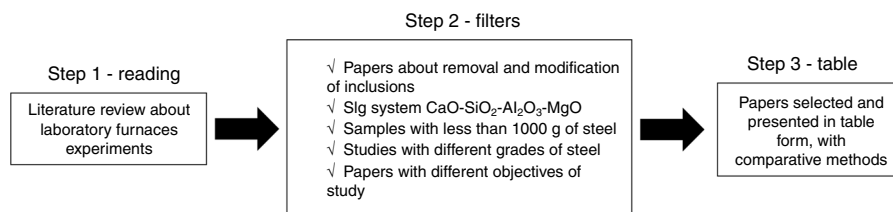


Fig. 1 – Flowchart used to design a table with comparative experimental data.

Table 1 – Chemical composition (wt.%) of the materials.

Material	CaO	SiO ₂	Al ₂ O ₃	MgO	FeO	MnO	CaF ₂
Calcium aluminate slag	45.65	2.84	22.88	20.75	0.57	0.06	7.25
Rice husk ash	0.44	95.59	–	2.58	0.87	0.53	–

One of the main parameters for measuring the steel cleanliness is the control of non-metallic inclusions. Among these parameters, the most important are composition, size, morphology and distribution [5]. The demand for clean steels is getting higher every year and thus, the control of inclusions becomes increasingly important [6]. Depending on the application of the material, the control of non-metallic inclusions must be done differently, so different applications require different practices for the control of inclusions [5].

The inclusions are generally removed through absorption by the slag. In the steel production, the removal happens in the secondary refining and at the continuous casting (tundish). The removal process can be performed in three steps: (i) flotation, (ii) separation, (iii) dissolution [7]. The first step involves the transport of the inclusion to the steel/slag interface. At this interface, surface tensions are present. They must be reached to carry out the separation of the inclusion from the liquid steel. Therefore, the inclusion can be stabilized at the steel/slag interface. Finally, the inclusion is considered removed when it is fully incorporated into the slag [1,7].

Refining slags have great influence on the steel cleanliness, resulting in a decrease in the total oxygen content due to its deoxidation capability. This property could lead to a decrease in the number of inclusions [8]. Other factors that can be modified with the use of refining slags are the shape, composition, and melting point of the inclusions. Numerous studies have already been done with the objective to evaluate

an experimental arrangement, based on a literature review, which would be adopted as a standard for laboratory experiments in the Ironmaking and Steelmaking Laboratory, at Federal University of Rio Grande do Sul (UFRGS). After choosing an optimized configuration, two experiments were done, and the results were compared with the literature to validate the experimental arrangement. A positive result would lead to future studies of secondary refining phenomena and their effects in the removal and modification of inclusions.

2. Materials and methods

2.1. Experimental arrangement

In order to define an experimental arrangement for laboratory tests with slag and steel in secondary refining conditions, three initial steps were defined according to Fig. 1.

The first step was focused on the reading of articles selected from the literature review. In the second step, some filters were applied to reduce the number of articles in this review. Inside the second step, works done with low alloy special steels and stainless steels were selected. With this filter, it was tried to verify different experimental practices depending on the type of steel studied. In the first step, it was identified that few experiments used fluorite in the composition of the slag. So it was applied a filter to leave this factor out of the review. For this review, only the CaO-SiO₂-Al₂O₃-MgO slag system will be

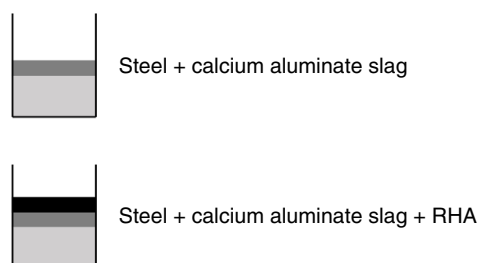


Fig. 2 – Experimental configuration of crucible and materials.

evaluated. Also, studies developed with more than 1000 g of steel were excluded due to the limiting factor of the size of the furnace that will be used in the future experiments. In addition, articles were chosen that had the removal and/or modification of inclusions as its main objective.

2.2. Experimental tests

Two materials used in the tundish were tested to validate the experimental arrangement selected. A calcium aluminate slag and rice husk ash (RHA) were analyzed to verify the formation of inclusions because of reoxidation events. The chemical composition of these materials are presented in Table 1

A modified SAE 1055 was used in this work; the sample was obtained after the rolling process. Two experiments were conducted; first, the interaction between steel and slag was analyzed. Then, the experiment was repeated with the addition of RHA. The experimental configuration is shown in Fig. 2.

After the experiments, an ASPEX Explorer equipped with a SEM/EDS system was used for the inclusion analysis. The inclusion density and the chemical composition of inclusions were evaluated. The total oxygen content (T.O.) in the steel was measured using a LECO TS-436 equipment. A Philips equipment, model PW2600 was used to measure the chemical composition of the materials by X-ray fluorescence. These results were compared with studies in the literature to verify if the experimental arrangement was correct to proceed with future laboratory studies focused in steel and slag experiments.

3. Results and discussion

3.1. Parameters of the experiments

After the development of the previously steps mentioned, twelve papers were selected [4,8,10–13,16–21]. They are presented in ascending order of publication year. In Table 2, the following parameters are presented: furnace type, protective gas, type and dimensions of crucible, steel mass, slag mass, moment of slag addition, slag/steel proportion, temperature, number of experiments, time and cooling method. The parameters that can be varied depending on the objective of study are: slag mass, slag/steel proportion, number of experiments and time for withdraw of samples during the experiment. All other parameters should remain constant. The authors did

not report some parameters, and then some fields in the table were not filled.

Afterwards, the experimental parameters mentioned in Table 2 will be analyzed and discussed in order to compose the experimental arrangement to be adopted as a standard for future experiments.

3.1.1. Type of furnace and protective gas

The experiments were performed in two types of furnace: inductive and resistive. The utilization of induction furnaces is characterized by the use of higher steel and slag masses when compared to the resistive furnace. The experiments in this type of furnace have the benefit of the existence of induction lines that can cause a natural stirring in the liquid bath [22]. The literature review showed that when using induction furnaces, the steel mass varied between 400 and 1000 g [4,19–21].

For resistive furnaces, the heating is done by the passage of electric current through an electric conductor, generating heat to the equipment due to the resistance of these conductors. With the passage of electric current, it may be possible to generate small magnetic fields, capable of performing a slight stirring in the liquid bath. Another phenomenon that could carry out some stirring in the liquid bath is the convection. Due to the temperature difference in the lower and upper furnace region and the presence of a protective gas during the experiments, this phenomenon would be possible to occur [22]. The use of this type of furnace was characterized by the presence of steel mass between 100 and 200 g [10–13,16–18].

The experiments will be conducted at the Ironmaking and Steelmaking Laboratory, located at the UFRGS. In this laboratory, there is a high temperature resistive furnace to be used for these experiments. Some parameters of this furnace are presented in Table 3.

The high purity inert gas used will be argon, following the standard in all the experiments reviewed. The function of the inert gas will be to protect the interior of the furnace and reduce the partial pressure of O_2 . With a low value, it should be possible to avoid reoxidation events [23]. The partial pressure inside the furnace was estimated assuming the use of argon gas with 99.999% purity and 1 ppm O_2 . This gas will be inside a volume of 4.4 L. By the equation $PV = nRT$ [24], the partial pressure of O_2 was calculated in 1.08×10^{-6} atm. During the literature review, it was found the value of 10^{-3} atm for partial pressure of O_2 measured with a gas analyzer. Thus, the estimated value lies in an acceptable range in order to avoid reoxidation events [23]. A suggestion for future works is the installation of a gas analyzer to measure the partial pressure of O_2 during the experiments.

3.1.2. Type of crucible and dimensions, steel mass, slag mass, slag addition and slag and steel proportion

The type of crucible varied in the experiments reviewed. The use of MgO , Al_2O_3 and ZrO_2 crucibles was verified. Crucibles of MgO are the most indicated, they are similar to the type of refractory used in most of secondary refining ladles. Crucibles of Al_2O_3 can be used as well; therefore, it needs to pay attention to the MgO saturation. In experiments with this type of crucible, it is intended to use a chemical composition in order to obtain a slag saturated in MgO . Otherwise, the formation of $MgO-Al_2O_3$ inclusions may not occur as expected [21].

Table 2 – Review of experimental parameters adopted in laboratory.

Author	Year	Type of Furnace	Gas	Crucible and Dimension (mm)	Steel Mass (g)	Slag Mass (g)	Moment of Slag Addition	Slag/Steel Proportion	Temperature (°C)	Number of Experiments	Time (min)	Cooling
Jiang [16]	2008	Resistive	Argon	MgO	150	30	Beginning	0.2	1600	8	25	Water
Jiang [17]	2010	Resistive	Argon	MgO	100	50	Beginning	0.5	1600	–	–	Water
Jiang [10]	2010	Resistive	Argon	MgO	100	50	Beginning	0.5	1600	29	1600	Water
Wang [13]	2011	Resistive	Argon	MgO ($\varnothing = 30$)	100	50	Beginning	0.5	1600	34	0.05	Water
Jiang [11]	2012	Resistive	Argon	MgO	100	50	Beginning	0.5	1600	33	During	Water
Jiang [18]	2014	Resistive	Argon	ZrO ₂	150	30	Beginning	0.2	1600	8	50	Water
He [12]	2014	Resistive	Argon	MgO	200	20	Beginning	0.1	1600	13	1000	Water
Yu [8]	2015	–	Argon	MgO	200	40	Beginning	0.2	1600	–	MgO and Al ₂ O ₃	Water
Kumar [19]	2016	Inductive	Argon	MgO($\varnothing = 61$) ZrO ₂ ($\varnothing = 31$)	600 and 100	200 and 15	Beginning and During	0.33 and 0.15	1600	3	–	Air
Wang [4]	2016	Inductive	Argon	MgO($\varnothing = 55$)	400	70	During	0.17	1600	5	Inductive	Water
Stephano [20]	2016	Inductive	–	MgO($\varnothing = 58$)	620	182	During	0.29	1600	2	2016	Air
Li [21]	2016	Inductive	–	MgO and Al ₂ O ₃	1000	50	During	0.05	1600	–	–	Air

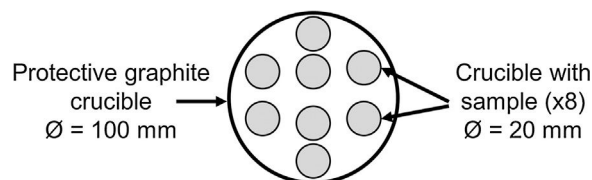
Table 3 – Technical data of the operation in the resistive furnace adopted in future studies.

Technical information	
Graphite tube dimensions	Diameter: 150 mm
Total height: 470 mm	
Working height (with insulation system coupled): 350 mm	
Graphite tube volume	4.41
Maximum operation temperature	2100 °C
Heating rate	5 °C/min (for T > 25 °C)
Atmosphere	Inert (Argon or Nitrogen)
Temperature measurement	Mantel-thermocouple WRe 6/25
Cooling system	Water (inlet: ~13 °C/outlet: ~37 °C)
Voltage/frequency	3 × 380 V/50–60 Hz

Another problem for this type of crucible is the saturation of Al_2O_3 . If the slag is not saturated in this component, it will react with the crucible wall to reach the saturation in Al_2O_3 . The use of ZrO_2 crucibles showed the same concern about MgO saturation [18,19]. In addition, it was verified the presence of many inclusions with ZrO_2 in their chemical composition [18]. The presence of those inclusions could difficult the interpretation of the results regarding industrial applications, where it is not usual the presence of inclusions with ZrO_2 in its chemical composition.

It was defined that the tests will be carried out in Al_2O_3 or MgO crucibles. The preferred type of the crucible for the experiments will be MgO . Only four papers from the review showed the dimensions of the crucibles [4,13,19,20]. This information is not shown frequently during those studies. Thus, the definition of the dimensions of the crucibles was made based on the size of the graphite tube present in the resistive furnace that will be used. The crucibles will have an internal diameter of 20 mm and a height of 50 mm. This size allows the placement of eight crucibles inside the furnace in each experiment. With this size, the maximum productivity can be achieved. It was intended to put the largest number of crucibles each time the furnace was turned on. A protective graphite crucible with internal diameter of 100 mm will be used to protect the samples and the interior of the furnace. This practice is used in some studies reviewed [19,20]. The steel mass will be 50 g. The slag mass could vary between 5 and 12.5 g, respecting the height of the crucible, and it will be added at the beginning of the experiment. Experiments with the addition of slag at the beginning could present better results regarding steel cleanliness. With the melting point of the slag smaller than steel, liquid slag will be formed before the melting of the steel sample. This condition could favor the phenomena of removal or modification of inclusions [19].

Due to the variation of slag mass, studies can be carried out to verify the influence of the slag/steel proportion during the treatment of inclusions. When this factor is not interest of study, it will be maintained constant at a value of 10 g of slag. With this mass of slag, the proportion of the experiment will be 0.2. This proportion was used in some reviewed studies [4,8,16,18,19].

**Fig. 3 – Arrangement of crucibles with samples inside the protective graphite crucible.**

3.1.3. Temperature, number of experiments, time and cooling

The temperature chosen for the experiments was 1600 °C, this value is used in all the experiments reviewed with focus on the study of the secondary refining process [4,8,10-13,16-21].

The number of experiments ranged from 2 to 33 in the references present in this review and in some cases, this value is not reported. The future experiments will follow statistical models based on design of experiments methodology. Experiments will be carried out starting from an experimental matrix generated in a 2^k factorial project. The varied factors will be chosen with the objective to verify their influence in studies with secondary refining slags. Their significance in relation to steel cleanliness will be evaluated as well. Based on a literature review, it is possible to choose the factors and levels that could be studied.

The residence time at the chosen temperature, 1600 °C, will be 90 min. With this time, it is desired to reach the thermodynamic equilibrium between steel and slag in a resistive furnace [16]. Studies with different times of residence can also be performed. In this case, it becomes possible to analyze the evolution of the format and composition of the inclusions during the time. For this arrangement, it is proposed the withdraw of samples in different times to perform the analyzes. All samples will be cooled in water, this practice is adopted in almost all the references reviewed [4,8,10-13,16-18].

3.1.4. Optimized experimental arrangement

From the observations and definitions presented in the previous subitems, the future experiments will have as standard the following arrangement:

- Furnace: Resistive;
- Protective Gas: Argon;
- Type of Crucible: MgO ;
- Number of Crucibles per Experiment: Eight;
- Mass of Steel: 50 g;
- Mass of Slag and Moment of Addition: 10 g in the beginning;
- Slag/Steel Proportion: 0.2;
- Temperature: 1600 °C;
- Number of Samples: chosen by 2^k factorial project;
- Time: 90 min;
- Cooling Method: Water.

Due to the dimensions of the crucible, it would be possible to use up to eight crucibles per experiment. These crucibles will be placed inside a protective graphite crucible. At 1600 °C there will be liquid steel and slag inside the crucible, in case of an accident such as slag leak, the protective crucible will

Table 4 – Amount of materials used and interactions studied in each experiment.

Experiment	Interaction	Steel (g)	Material (g)
A	Steel-tundish slag	150	20
B	Steel-tundish slag-RHA	150	20 and 5

prevent damages to the furnace interior. Fig. 3 shows the layout of the crucibles with the samples inside the protective crucible.

The utilization of eight crucibles maximizes the productivity of the experiments, being possible to realize then more efficiently. The withdraw of the samples in the temperature of 1600 °C should be done valuing the security of the people involved. After cooled, the samples will be prepared and analyzed according to the main objectives of the future works.

3.2. Experimental results

As it was explained before, two experiments were conducted to verify the experimental arrangement. For these experiments, three different parameters from the optimized arrangement were used. The material of the crucible was alumina, since the use of MgO crucible was postponed because of the difficulty to find a provider for this type of crucible in Brazil. The temperature chosen was 1580 °C because this is the approximately temperature of the steel during the tap operation from the ladle furnace to the tundish. The samples were cooled inside the furnace to avoid air contamination that could lead to unwanted reoxidation events. Table 4 presents the amount of steel, material and the interactions analyzed.

The silicon (Si) content in the steel sample was measured to verify its variation. The literature shows that the increase of SiO₂ in the tundish slag would increase the Si content [23,25–27]. The presence of RHA, a silica based material, increased the Si content in experiment B from 0.220 (original sample) to 0.310 wt.%. Experiment A, with low SiO₂ content, presented a final value of 0.186 wt.% of Si. The first parameter analyzed for the inclusions was their chemical composition. Their values were plotted in a CaO-Al₂O₃-SiO₂ ternary diagram and the result is presented in Fig. 4. Experiment B had an increase in the average SiO₂ content in the inclusions, thus an oxide system with SiO₂ was chosen for the analysis of chemical composition.

The composition of inclusions in the initial steel sample, Fig. 4(a) presented mostly alumina silicate inclusions, also a good steel cleanliness is observed. Experiments A and B presented different types of inclusions than that observed in the initial sample, indicating that the inclusions observed in these experiments were formed by reoxidation. Related to the types of inclusions observed in Fig. 4(b) and (c), experiment A presented mostly alumina inclusions, Fig. 4(b). It is important to note that the calcium aluminate slag used in the experiments reacted with the crucible to reach the saturation in alumina. This effect probably generated numerous pure alumina inclusions. In the other hand, the experiment B had the formations of many oxide inclusions in the CaO-Al₂O₃-SiO₂ oxide system, being detrimental to the quality of the steel. The presence of RHA increased the SiO₂ content of the slag. This would lead to the formation of numerous inclusions with Al₂O₃ in their composition [23,25,27,28]. This result is confirmed in Fig. 4(c). Both experiments increased the number of inclusions, being harmful to the steel cleanliness. The chemical composition of the slag after the experiments are presented in Table 5.

Results in Table 5 show that the slags reacted with the crucible increasing the content of Al₂O₃ in their composition. Yan et al. [23] used a calcium aluminate slag in alumina crucible and verified this increase in the Al₂O₃ content as well. The experiment with RHA had a lower value for this element. In the literature, it is observed that the increase in the SiO₂ content in the slag reduces the Al₂O₃ content [23,27]. The presence of RHA appears to reduce the reaction between the slag and the crucible, thus presenting a lower content of Al₂O₃ in the final chemical composition for experiment B. Excluding the pure alumina inclusions, the experiment without the presence of RHA presented a better steel cleanliness. The use of only a calcium aluminate slag is preferable during the tundish metallurgy in order to achieve a better final product. The presence of RHA would lead to higher reoxidation events, and a decrease in the capability of inclusion removal [29,30]. The inclusion density was the other property analyzed to verify the formation of

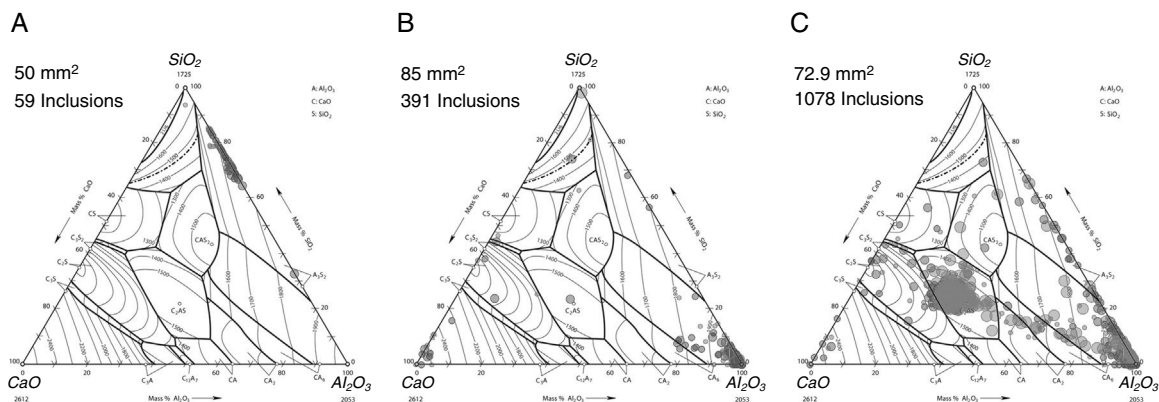
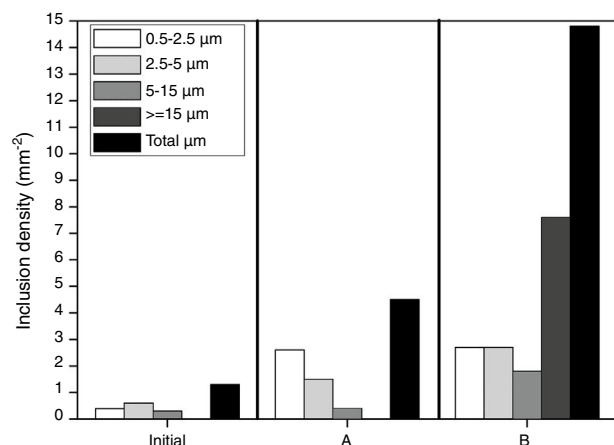


Fig. 4 – CaO-Al₂O₃-SiO₂ ternary diagram with the chemical composition of inclusions. (a) Initial steel sample; (b) Experiment A; (c) Experiment B.

Table 5 – Chemical composition (wt.%) of the slags after the experiments.

Experiment	CaO	SiO ₂	Al ₂ O ₃	MgO	FeO	MnO	CaF ₂
A	26.29	3.70	54.43	11.71	1.20	0.23	2.44
B	29.90	12.84	41.82	10.89	1.02	0.37	3.16

**Fig. 5 – Inclusion density for the initial steel sample, experiment A and experiment B.**

new inclusions. Four ranges of inclusion diameter were analyzed: 0.5–2.5, 2.5–5, 5–15 and ≥ 15 μm . Fig. 5 shows the results for this analysis.

The initial steel sample presented a total inclusion density of 1.30 mm^{-2} . Furthermore, none inclusion with diameter above $15 \mu\text{m}$ is observed. Inclusion in this range are detrimental to the mechanical properties and should be avoided during the steel production [6,31]. Both experiments presented a total inclusion density greater than the observed in the initial steel sample, with the experiment B presenting the higher value. The presence of RHA in the experiment B increased the inclusion density from 4.50 to 14.80 mm^{-2} . Kim et al. [27] and Yan et al. [23] verified an increase in the number and size of inclusions with the increase in the SiO₂ of the slag. The presence of inclusions $\geq 15 \mu\text{m}$ is another harmful effect and should be avoided. In the experiment B, many inclusions were observed in this range. Experiment A, with only the calcium aluminate slag, presented mostly inclusions between 0.5 – $5 \mu\text{m}$ and none inclusion with diameter $\geq 15 \mu\text{m}$. These results are similar with the initial sample, which presented an adequate steel cleanliness according to the literature [6,31]. From the results presented until now, it is confirmed that the tundish materials could be responsible for reoxidation events, being capable to deteriorate the cleanliness obtained before the tundish steelmaking. In order to diminish reoxidation events in the tundish steelmaking, it is preferable to use only a calcium aluminate slag, without the presence of RHA.

An important aspect that was pointed out in the parameters review was the oxygen partial pressure. Despite that the experimental arrangement did not present an equipment to measure this property, it was proposed a theoretical value for the furnace used in the experiments. The total oxygen content (T.O.) was measured for both experiments to verify a possible contamination because of the furnace atmosphere.

The initial T.O. of the steel samples was 13 ppm. Experiment A and B presented 11 and 7 ppm, respectively. This value decreased for both configurations and proves that the atmosphere granted by the passage of argon gas was effective in providing a low oxygen partial pressure.

4. Final considerations

With the realization of an individual analysis about each parameter that could affect the accomplishment of experiments in laboratory, it was possible to reach a suitable experimental arrangement for future works. This arrangement will be adopted as standard in the Ironmaking and Steelmaking Laboratory. With the development of these experiments, it is expected that the laboratory acquire practice in this area of research, which is very promising in the coming years. With the increase in the recycle of recently produced steels, there is a possibility that the concentration of some elements could be higher than nowadays in the secondary refining process. Those elements cannot be eliminated during the selective oxidation in the primary refining. Then with a higher concentration in the secondary refining, it may be responsible for the appearance of inclusions that still need a better understanding in the processes of removal and modification. Therefore, the realization of experiments in laboratory aiming to reach fully acknowledgment about these inclusions presents a field of research very prospective in the future.

Another field of research, which was the aim of the experimental tests, is the reoxidation of steel in the tundish. With the demand of special steels increasing every year, the presence of an active slag in the tundish is desirable in future years. Beyond the common functions desired nowadays, a tundish slag should be capable to provide one more step for the removal of inclusions before the casting process. The results presented in this work were in a good agreement with the literature. Thus, the experimental arrangement selected proved to be effective for the future studies in laboratory conditions with the presence of steel and slag.

Conflicts of interest

The authors declare no conflicts of interest.

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