The role of FeTi addition to micro-inclusions in the production of ULC steel grades via the RH process route

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Summary

Titanium is the most commonly used stabilizer for Ultra Low Carbon (ULC) and Interstitial Free (IF) steel grades. In the production of ULC steel grades via the RH process route, Al is first added for deoxidation after the end of decarburization and FeTi follows after a certain period of separation of alumina particles and the related reduction of the total oxygen content. The FeTi addition is well known to cause clogging problems in the following casting process.

The present work deals with results from plant investigations, indicating an increasing clogging tendency with a higher Ti/Al ratio in the steel. Automated SEM/EDS investigations on lollipop samples show the existence of a newly nucleated Ti-containing alumina particle population after the FeTi addition. These particles are comparably smaller than the alumina particles and even if thermodynamically unstable, they still exist as a large population of small particles in samples taken from the tundish.

The addition of Al and FeTi into a molten steel sample with controlled initial oxygen activity for varying Ti/Al ratios was simulated in laboratory scale afterwards. Just like in the plant, a new population of small Ti-containing alumina particles nucleates, with the size and number depending on the Ti/Al ratio in the melt. The local supersaturation of Ti and O during the dissolution of the FeTi particles seems to be the main reason for the nucleation of these inclusions.

Sessile drop experiments indicate that the wetting angle between these Ti-containing alumina particles and ULC steel is at the same level as for pure alumina particles. However, due to only moderate convection of the melt in the ladle after the FeTi addition, the agglomeration tendency for these particles is low and as previous work has shown, a large number of non-wetting small particles is absolutely critical for clogging.

Laboratory experiments and plant observations are in consistence with each other and indicate - for the underlying process route and process parameters - some countermeasures for the better control of clogging.

Key Words

Continuous casting, clogging, titanium, ULC steel.

Introduction

Ti-stabilized ULC and IF steel grades frequently cause the build-up of oxide layers or oxide networks in the flow control system of the casting machine. This process is known as clogging and has a negative impact on productivity and product quality. According to the extensive literature, different mechanisms like chemical reactions between steel and refractories, the solidification of steel inside the submerged entry nozzle (SEN) or the adhesion of nonmetallic inclusions (NMI) on the inner surface of the SEN may trigger clogging. However, clogging incidences can commonly not be assigned to a single mechanism. In addition, differences in plant configurations, Al deoxidation and FeTi addition practices as well as the design of the fluid flow control system of the casting machine also have a significant influence on clogging [e.g. 1]. This complexity of possible influencing parameters may contribute to the widely spread and partly inconsistent research findings on the clogging of Ti-stabilized ULC steel; a comprehensive overview of the state of knowledge was recently published by Dorrer et al. [2].

The present paper addresses only one of these aspects, namely the formation of a new inclusion population due to the FeTi addition after RH treatment as a possible cause for clogging. Even for this isolated phenomenon, different explanations exist; in the following, an extracted summary: Kaushik et al. [1, 3] detected Ti-bearing alumina inclusions after FeTi addition with a significantly smaller diameter compared to the pure aluminates. The morphology, size and number depend not only on the Ti content, but also on the plant configuration and vacuum treatment practice. The authors of the present paper have also reported the existence of Ti-containing alumina particles with a smaller diameter compared to pure aluminates and a decreasing diameter with an increasing Ti/Al ratio in the steel [4].

Several explanations for the formation of this Ti-bearing inclusion population exist, most of which are from laboratory experiments: IF steels typically contain 0.02 – 0.05 wt.-% Al and 0.02 – 0.10 wt.-% Ti. For this reference composition alumina is the thermo-dynamically stable oxide [5]. Nevertheless, the local supersaturation of Ti and O during the dissolution of FeTi in the steel might either result in the nucleation of Ti-containing alumina inclusions as well as a temporary or permanent modification of pre-existing alumina particles. It should be noted that the total oxygen content of FeTi, depending on the grade, may range from 0.1 to more than 1.0 wt.-% and the Al content may amount to appr. 2 – 5 wt.-% [8].

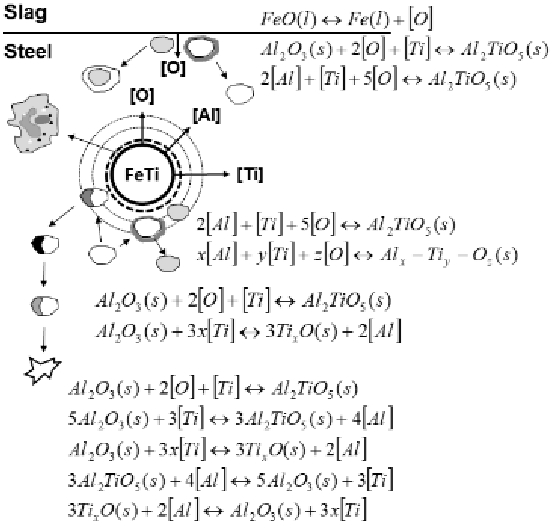

Figure 1: Schematic overview of possible reaction sites after FeTi addition according to the literature given in the paper.

Among other researchers, Matsuura et al. [6] investigated the evolution of non-metallic inclusions as a result of Al and Ti additions to molten iron: They found that the Ti addition results in a temporary morphological and chemical modification of the alumina inclusions and finally in a change of the aluminate morphology from spherical to polygonal shaped. In a later publication, the same authors investigated the effect of gradually increasing Ti additions and found a critical value for Ti/Al that suggests that morphology is dominated by irregularly shaped inclusions after Ti addition [7].

Publications on the transient behavior of inclusions after Ti addition have one thing in common: After assuming the formation of a Ti-containing phase by, e.g. the nucleation of TiO_X after complex Ti/Al deoxidation [e.g. 8], the temporary modification of Al_2O_3 towards $Al_2O_3{\cdot}TiO_2$ or the heterogeneous nucleation of a $Al_2O_3{\cdot}TiO_2$ layer around the alumina core [e.g. 9], these phases transform later on towards the thermodynamically stable phase, Al_2O_3. A further potential source for oxygen is the ladle slag: After RH treatment and Al deoxidation, these slags may still contain more than 15 wt.% FeO [9, 10] and thus deliver the oxygen for the heterogeneous nucleation of $Al_2O_3{\cdot}TiO_2$.

A last possible source for inclusions is the FeTi itself: Pande et al. [8] dissolved ferroalloys and among them also FeTi70 in acid and detected a large number of complex phase Ti-Al-O inclusions afterwards.

Fig. 1 presents an overview of possible reaction sites and oxygen sources after FeTi addition.

The present work deals both with analytical investigations of inclusions in samples from steel production and the results of the simulation of isolated phenomena in laboratory scale.

Plant Observations

The Austrian steel producer voestalpine Stahl GmbH (vaS) in Linz produces ULC/IF steel grades typically in heat sizes of 180 tons via the BOF-LF-RH-CC route. A general description of the production process can be found in [10].

After tapping and a possible temperature adjustment in the ladle furnace, degassing in the RH degasser starts at a level of appr. 600 ppm oxygen. After achieving the target value for carbon, Al is added depending on the actual oxygen activity and the Al target value. During the next minutes, most alumina inclusions are separated and the total oxygen content decreases to less than 30 ppm. After another eight minutes, FeTi is added. The slag still contains more than 15 wt.-% of FeO at this time and only soft bubbling is applied to minimize reoxidation. Finally, the ladle is transferred to the casting machine.

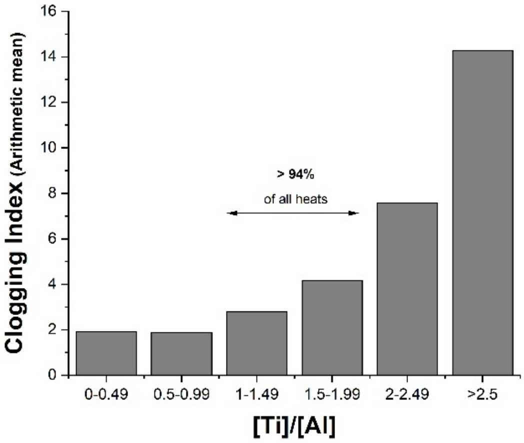

Figure 2: Clogging index (according to the vaS internal definition) vs. Ti/Al ratio in the steel according to [2].

The negative influence of an increasing Ti/Al ratio on the clogging tendency has already been reported in two earlier publications [4, 11]. **Fig. 2** shows the vaS internal clogging index for more than 3000 heats, produced in the years 2017 and 2018 and graded according to the Ti/Al ratio of the produced steel. For the vast majority of these heats, the Ti/Al ratio ranges from 1 to 2. The negative influence of an increasing Ti-Al ratio is obvious. Some details behind the statistics in order to emphasize the relevance for the casting process: The Ti/Al class 1-1.49 comprises more than 2000 heats. Assuming a clogging index of 10 as the critical upper limit for problem-free casting, appr. 6 % of all heats exceed this limit. This is still a remarkably high number. In contrast, only 120 heats belong to the Ti/Al class 2-2.49, but among them appr. 25 % result in clogging issues during casting. Accordingly, the number of clogging incidences increases substantially with an increasing Ti/Al ratio, but a higher Ti/Al ratio does not necessarily result in clogging. The Ti/Al ratio is therefore an indicator for increasing clogging sensitivity of a steel grade but not suitable as the only clogging criterion.

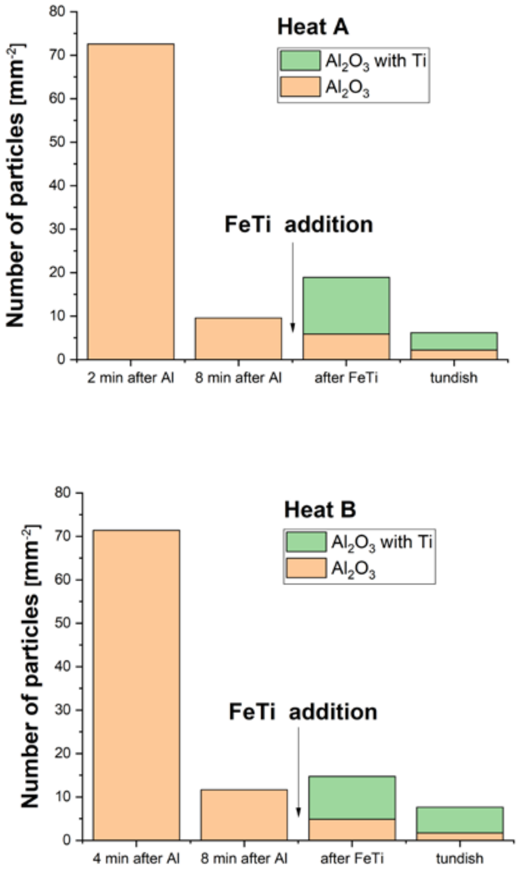

Figure 3a (top) and 3b (bottom): Number of particles in samples taken from production process for heats A (3a) and B (3b).

For a specific analysis of the evolution of the number, size and morphology of inclusions over the time between Al addition and casting, samples taken from the production were analyzed by means of automated SEM/EDS. Representative of a large number of investigated heats, the results for two of them will be discussed exemplarily: Samples were taken 2 minutes and 8 minutes after Al addition, immediately after FeTi addition and from the tundish after casting of half of the heat. The final Ti/Al ratio amounts to 1.25 for heat A and to 1.65 for heat B. Details on the sampling, metallographic/analytic investigations and classification of the inclusions can be found in [2].

Figures 3a and b present the detected number of inclusions containing alumina in the four samples for heats A and B. Two minutes after deoxidation, still a large number of alumina inclusions are present in both cases. The number decreases significantly during the next 6 minutes. After FeTi addition (8 minutes after deoxidation), a new type of inclusion can be detected, termed here as “Al_2O_3 with Ti” or “AT” (Al, Ti, O). Between that point and the sample collection from the tundish, the greater part of the inclusions is again separated but the class AT survives and finally forms the majority of inclusions. The given numbers stand in surprisingly good correlation with data from [1].

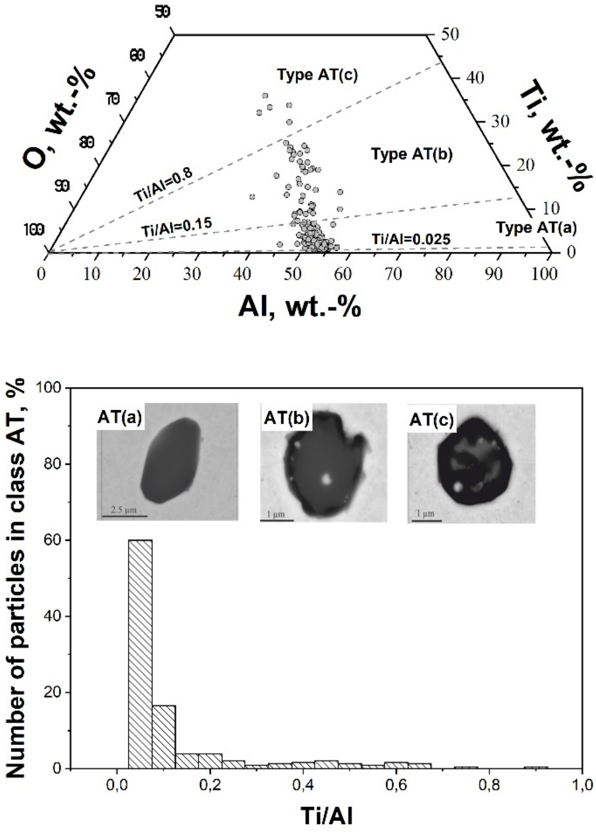

Figure 4a (top, [2]) and 4b (bottom): Composition of particles of class “AT” (heat A) in the ternary system Al-Ti-O and number of particles in the same group classified via Ti/Al.

Figures 4a and 4b show the inclusions of the class AT from heat A, ranged in the ternary system Al-Ti-O (in wt.-%), together with a bar chart of the number of inclusions vs. the Ti/Al ratio and representative micrographs of AT inclusions. In the following figures, the class AT is divided into the subgroups AT(a) with $0.025 < Ti/Al < 0.15$, the group AT(b) with Ti/Al between 0.15 and 0.8 and the group AT(c) with Ti/Al

> 0.8. The large majority of the inclusions (appr. 80%) belongs to the class AT(a). The necessity for the assumption of a lower limit for the Ti/Al ratio results from uncertainties in the measured Ti content for small particles as well as from heterogeneous nucleation of TiN on alumina particles. For the latter, nitrogen might not be detected due to its low mass fraction in the particle and the classification as a particle of the group AT would then be misleading. The particles of AT(b) and AT(c) are mostly heterogeneous and contain phases with high Ti content (TiO_2 or $Al_2O_3{\cdot}TiO_2$). The morphology of these inclusions is similar to inclusions as described in literature [6-9]. They might form as a result of the modification of inclusions between the time of FeTi addition and sampling in the tundish.

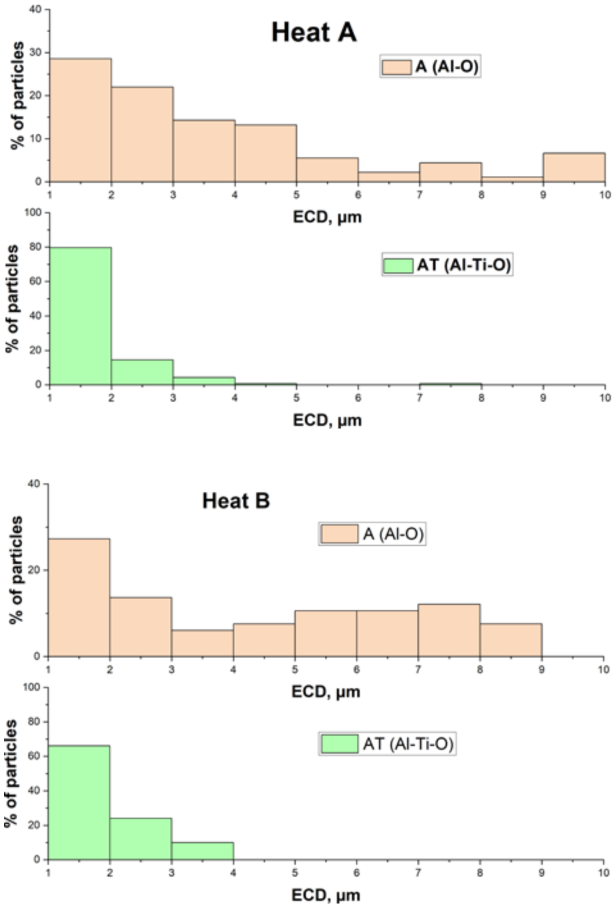

Figure 5a (top) and 5b (bottom): Size distribution for particles in classes Al_2O_3 (Al-O) and AT (Al-Ti-O) for heats A and B.

The nature of inclusions of the class AT(a) is mostly homogeneous and conclusions on their formation may be drawn on the basis of their ECDs: **Figures 5a and 5b** compare the ECD statistics for the classes Al_2O_3 (Al-O) and AT in tundish samples from heats A and B. In general, the total number of inclusions is small and statistics are therefore limited. A bimodal distribution is indicated for class Al-O with an aged population of particles with higher ECD (presumably resulting from deoxidation) and a population of small particles, pointing to the later nucleation, maybe due to the FeTi addition. For the class AT(a), all particles show a diameter of less than 3 µm, clearly indicating that they nucleated after FeTi addition and are not the result of the modification of pre-existing oxides.

The possible role of particles of type “AT” for clogging may be exemplified by means of the results from former investigations on clogged submerged entry nozzles after the casting of Ti-ULC and P-ULC steel grades [4]: The SEM/EDS investigations indicate that the majority of particles in the clog are spherical particles with a mean diameter between 2 and 4 µm. In addition, much larger particles with elongated shape or dendritic morphology can also be found. Their size may amount to 10 µm and even more. The chemical composition of the cast steels and the analyzed clog is given in **Table 1**: For a Ti/Al ratio between 1.4 and 2.0 in the steel, the clogging deposit consists mainly of Al_2O_3 with 1.4 – 2.1 wt.-% Ti. The size, shape and chemical composition of the particles indicate that the inclusions of class “AT” are at least involved in the build-up of the clogging layer.

Steel composition		Mean particle composition	
Al, wt.-%	Ti, wt.-%	Al_2O_3, wt.-%	Ti/Al wt.-%/wt.-%
0.039	0.054	87	0.047
0.035	0.067	91	0.028
0.036	0.071	93	0.031

Table 1: Selected numbers for composition of steel and clog according to [4].

Experimental Results

The main objectives of the lab experiments were as follows:

- Adding Al and FeTi75 (5 minutes afterwards) to a steel melt with controlled initial oxygen content under Ar protection in Al_2O_3 crucibles without top slag to observe whether the population of AT inclusions forms due to the FeTi addition or not.
- Variation of initial oxygen content and the Ti/Al ratio to observe the influence on number and volume fraction of newly nucleated particles.

The lab experiments were conducted in a resistance heated furnace (Ruhrstrat HRTK 32 Sond.) at 1600 °C with 300g of liquid iron. The carbon heating tube reacts with traces of oxygen in the Ar5.0 and thus provides a slightly reducing atmosphere. In case of the present experiments, the crucibles are made of Al_2O_3. The initial oxygen content was adjusted to target values of 60, 120 and 150 ppm by adding an Fe-O master alloy with 0.18 – 0.20 wt.-% O. The Ti/Al ratio was gradually changed between no FeTi addition (Ti/Al = 0) and Ti/Al = 2.3. The limited accessibility of melt for alloying additions in the small crucibles made a precise adjustment of the composition difficult. Nevertheless, the target values were met in a sufficient manner to deliver the expected results. The main parameters for the experiments performed are given in **Table 2**. The FeTi75 used contains, according to our own measurements, between 0.1 and 0.3 wt.-% of oxygen and is highly heterogeneous. The hardly controllable oxygen input into the system is unquestionably a factor of uncertainty for the experimental results (as it is for the plant).

Test series	Main parameters
Series I (5 experiments)	Initial oxygen: 140 – 170 ppm Al: 0,043 – 0,062 wt.-% Ti/Al: 0/0.8/0.9/1.89/1.93
Series II (3 experiments)	Initial oxygen: 127 – 176 ppm Al: 0,016 – 0,033 wt.-% Ti/Al: 0.83/1.37/2.33
Series III (3 experiments)	Initial oxygen: 62 – 85 ppm Al: 0,030 – 0,035 wt.-% Ti/Al: 0.56/1.28/2.30

Table 2: Main parameters for the performed experiments according to [2].

Sampling was performed after melting and homogenization, 3 minutes after Al addition and 1 and 8 minutes, respectively, after FeTi addition. Details regarding the experiments, sampling techniques, reproducibility of the results and SEM/EDS analysis can be found elsewhere [2].

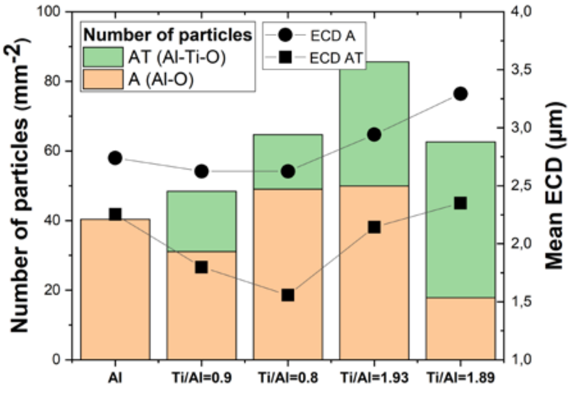

Figure 6: Number of particles in classes "A" and "AT" with mean ECD for experiments of series I.

In the following, only the results of automated SEM/EDS on the very last sample taken (8 minutes after FeTi addition) will be considered for a discussion. **Figure 6** summarizes the results of series I: The class AT is detected in all samples with FeTi addition, and the number of AT particles grows with increasing Ti/Al ratio and the size of the AT particles (here the mean ECD) is significantly smaller than in the class A.

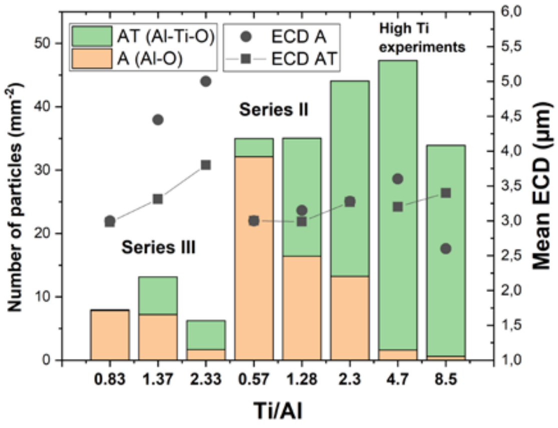

Figure 7: Number of particles in classes "A" and "AT" with mean ECD for experiments of series II and III and two experiments with a high Ti/Al ratio.

Similar to Figure 6, **Figure 7** presents the number and mean ECD for the particles in the series II and III experiments, together with two experiments with extremely high Ti addition. Series III represents experiments with an initial oxygen activity of 62 – 85 ppm. The resulting number of "A" and "AT" particles is very low. "AT" only forms above Ti/Al = 0.83. For series II, the initial oxygen content is 127 – 176 ppm (target value 140 ppm). The number of particles is significantly higher and particles of class "AT" form already at Ti/Al = 0.57. For the two high Ti experiments, the resulting particles belong almost exclusively to the class "AT."

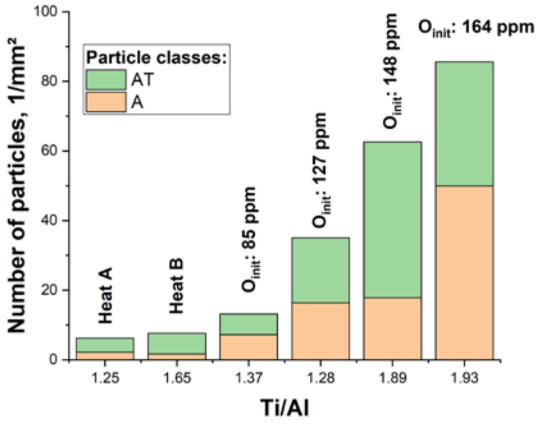

Figure 8: Number of particles in classes "A" and "AT" versus Ti/Al and gradually increasing initial oxygen content.

Figure 8 compares the results from experiments in the range of Ti/Al = 1.37 – 1.93 and different initial oxygen content with the results for heats A and B from the plant observations. The significant influence of an increasing oxygen content is obvious.

The following conclusions can be drawn from the experimental results:

- Despite the exclusion of slag and air as oxygen sources, the FeTi addition results in the nucleation of a new population of Al_2O_3 + Ti inclusions (class "AT").
- Initial oxygen content and Ti/Al ratio are the main influencing factor for the final number and volume fraction of AT inclusions.
- Even though the AT inclusions are thermodynamically unstable, they show no tendency to modify their shape or chemistry during the 8-minute holding time after the FeTi addition.

It should be noted that both the separation and agglomeration tendency are rather small in the experiments, as fluid flow in the melt only results from natural convection and in addition, thermal gradients are marginal. The separation of particles is limited to the adhesion at the crucible/steel interface. The significance of the results is limited to the above-listed conclusions and the results should not be interpreted with respect to the behavior of particles in a ladle.

The results of the plant observations and experiments will be discussed below with respect to their possible relevance for clogging.

Discussion

Following the approach of Sasai et al. [13], a model for the detachment of particles from refractory/steel interfaces under some simplifying assumptions for the hydro-dynamics close to the wall/steel interface was recently postulated and published [12]. Several forces act on a particle adhered to a steel/refractory interface; **Figure 9**: Adhesion between particle and wall, the buoyancy force and forces resulting from the fluid flow, namely the drag force and lift force.

The force balance may either result in the permanent agglomeration and sintering of the particle or the detachment from the wall. The two key parameters are the size of the particles and the wetting angles between steel/particle and steel/refractory. The higher the tendency towards a non-wetting system (like, for example, for Al_2O_3 in contact with ULC steel), the higher the required fluid flow velocity for the particle detachment. In contrast, liquid particles (like calcium aluminates) form wetting systems and the adhesion force decreases to very low values or even changes direction. Under these conditions, agglomeration at the refractory becomes unlikely. The smaller the particles, the higher the necessary flow velocity becomes for particle detachment. The final aspect to be considered is the number of particles: The higher the number of particles, the higher the number of collisions between particle and wall. Summing up, a large number of small and non-wettable inclusions typically results in a higher clogging probability.

The class of "AT" particles fulfills these conditions: The particles nucleate very late in the process, so the size of the particles is comparably small. The tendency towards agglomeration and separation in the ladle is low. The soft bubbling strategy after the end of RH treatment may contribute to this behavior, like in the absence of a forced convection in the experiments.

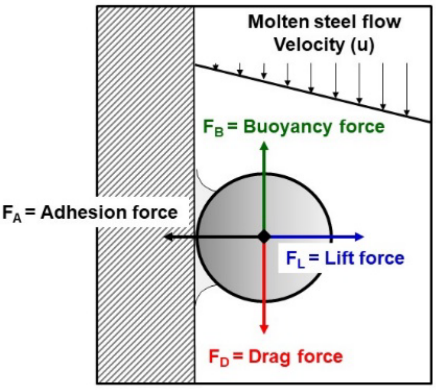

Figure 9: Force balance on adhered particle at the steel/refractory interface [16].

The influence of a gradually increasing TiO_2 content in Al_2O_3 on the wetting angle remains the only unknown. To answer this open question, sessile drop experiments in a Krüss DSA 10-HT apparatus were performed at 1600°C [15]. The substrates used in the experiments consist of a molybdenum disc and a plasma spray-coated surface layer from commercial powder mixtures of Al_2O_3 and TiO_2. It should be noted that the nature of the resulting spray-coated layer simulates neither the mineral structure nor the surface roughness of a homogeneous inclusion precisely. Nonetheless, the measured wetting angles for pure Al_2O_3 coatings and pure TiO_2 layers correspond to the results of former measurements on ceramic substrates very well [14]. This indicates at least a qualitative correctness of the results on the Al_2O_3/TiO_2 mixtures. Two steel grades were used for the measurement, the first one being a typical ULC grade without Ti and the second one a Ti-stabilized ULC steel with 0.083 wt.-% Ti. The results in **Table 3** indicate that a gradually increasing TiO_2 content does not significantly influence the wetting of the two steel grades on the surface as long as the TiO_2 content is still below 10%. As expected from [14], the wetting angle for the Ti-stabilized ULC is slightly lower but still clearly in the non-wetting regime. As a result, the last conditions for a higher clogging tendency are also fulfilled. The system "Ti-stabilized ULC" in contact with particles of the class "AT" is non-wetting and the particles are attracted by the refractory wall. The number of particles is high and so is the presumable number of collisions between particle and wall. The size of the particles is small and the detachment of the particles should become more difficult.

Steel grade	ULC	Ti-ULC
Substrate		
Pure Al_2O_3	151°	141°
97% Al_2O_3 + 3% TiO_2	153°	-
90% Al_2O_3 + 10% TiO_2	154°	141°

Table 3: Wetting angle of ULC and TI-ULC on substrates with gradually increasing TiO content [15].

Conclusion

Plant observations and laboratory experiments suggest the nucleation of a new inclusion population of Al_2O_3 particles with traces of Ti after FeTi addition. According to the understanding of the interfacial physics between steel and refractory in the fluid flow control system of casting machines, these particles will be attracted by the interface and as they are small and their number is high, they should increase the risk for clogging. It should not be concluded that a higher Ti/Al ratio is the only decisive parameter for clogging; nor is the adhesion of oxides by the interface the only clogging mechanism involved. Nevertheless, the control of O input by the added FeTi could be a beneficial measure for clogging prevention.

Acknowledgments

The authors gratefully acknowledge the funding support of K1-MET GmbH, metallurgical competence centre. The research program of the competence centre K1-MET is supported by COMET (Competence Centre for Excellent Technologies), the Austrian program for competence centres. COMET is funded by the Federal Ministry for Transport, Innovation and Technology, the Federal Ministry for Digital and Economic Affairs, the provinces of Upper Austria, Tyrol, and Styria, and the Styrian Business Promotion Agency. Beside public funding, the project is financed by the industrial partners voestalpine Stahl, voestalpine Stahl Donawitz and RHI Magnesita, Austria.

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