Experimental investigation into the influence of Ti on the clogging of ULC-steels in continuous casting

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INTRODUCTION

Ti-stabilized ULC-steels are well known to be prone to the appearance of alumina clogging in the flow control system during continuous casting. In literature, this is either attributed to

- the formation of an Al-Ti-O-containing interfacial layer between the alumina inclusions and the liquid steel with increased surface wettability and thus, a reduced separation efficiency, leading finally to a higher number of small alumina particles,
- the reduction of less stable oxides from the SEN-material or
- a morphology change of the alumina inclusions due to the Ti-addition.

The chemical analysis of particles from clogged SENs after the casting of Ti-alloyed steels at steel plant No. 3 of voestalpine in Linz indicates that most particles mainly consist of alumina with only a small amount of Ti. Automated inclusion analysis by OES-PDA and SEM/EDS shows a decrease in size of oxides with increasing Ti/Al-ratio in the steel whereas the number of small alumina inclusions increases.
Wetting experiments with a sessile drop apparatus at the Department of Metallurgy led to contradictory results: The formation of a reactive interfacial layer between the steel droplet and the substrate results in a remarkable increase of the wettability but Ti does not influence the wettability in absence of an interfacial layer. Thus, the reaction between Ti and alumina inclusions seems to be a decisive factor for the clogging tendency of Ti-alloyed ULC-steel grades.
A number of different deoxidation and Ti-addition practices have then been simulated in laboratory experiments. A Tammann furnace was used for these experiments. The time between Al-deoxidation and Ti-addition proved to be the key influencing quantity on the final composition of the oxide particles.
Ti seems to diminish the size of alumina-oxides only if a wettable surface layer forms. Generally, smaller alumina inclusions enforce clogging. The time between deoxidation and Ti-addition might thus be a decisive process variable to prevent clogging.

LITERATURE REVIEW

Numerous studies have been conducted to elucidate the mechanisms of nozzle deposition in continuous casting, and various adhesion theories have been proposed in the past. In general, the clogged material inside SEN is attributed to either

(1) the agglomeration and sintering of deoxidation products at the steel/refractory interface,
(2) air aspiration into the nozzle and subsequent re-oxidation of the steel at the steel/refractory interface,
(3) a chemical reaction between the nozzle refractory and the steel or
(4) solid steel buildup during the start of casting[1].

For the purpose of simplification also four main influencing factors can be distinguished, see Figure 1:

(1) Metallurgical factors, mainly the metallurgical practice during ladle treatment and tundish metallurgy.
(2) Factors related to the material properties and thermophysical properties of the SEN-material (e.g. porosity, wettability, chemical stability, ….).
(3) Hydrodynamic factors, respectively the fluid flow characteristic in tundish, SEN and mold.
(4) The operational practice of the caster.

In reality, however, a given nozzle deposit is often the result of more than only one of the above mentioned mechanisms and the identification of its root as well as the initiation of countermeasures cause a delicate task.

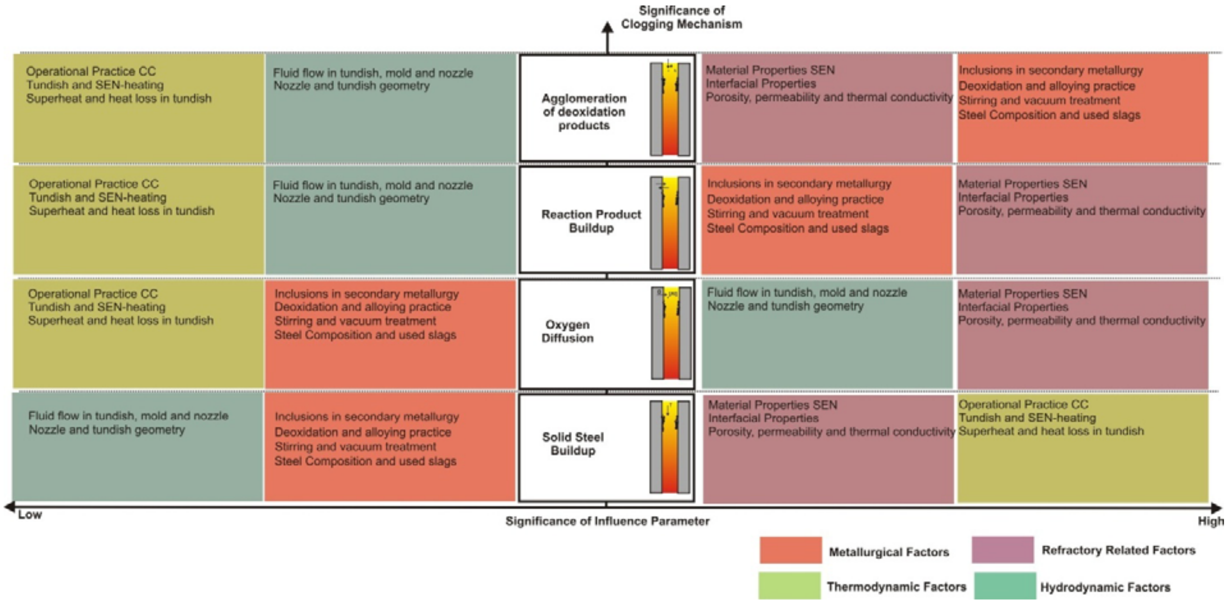


Figure 1. Main clogging mechanisms and related influencing factors.

In every ranking of clogging sensitive steel grades by their clogging index, Ti-stabilized ULC steels would be found at the very top. Figure 2 shows results from a recently published work, summarizing the operational experience of voestalpine with more than 300 heats on a slab caster[2]. The clogging index - including the relevant continuous caster operating parameters like stopper rod position and mold level fluctuations - rises significantly with increasing Ti-content of the ULC steel[17]. The clogging index for ULC steel with Ti between 0.07 and 0.10 wt.-% is twice as high as for Ti-free ULC steel grades.

In the casting of Ti-stabilized ULC steel grades the adhesion of solid Al_2O_3-particles in the flow control system is most often found the main reason for clogging. As a matter of fact, the influence of Ti on the nozzle clogging phenomenon has been thoroughly investigated and several theories have been put forward[3-14]:

According to Kawashima et al., the exacerbated clogging experienced during the casting of Fe-Al-Ti alloys is a combined effect of: (a) the Ti-induced increase of wettability between the nozzle refractory and molten steel resulting in an acceleration of the SiO_2 reduction in the refractory by [Al] and a change of nozzle surface quality; (b) the decrease of the oxygen activity in the steel melt causing the decomposition of Al_2O_3 particles and thereby increasing the number of finer inclusions[9].

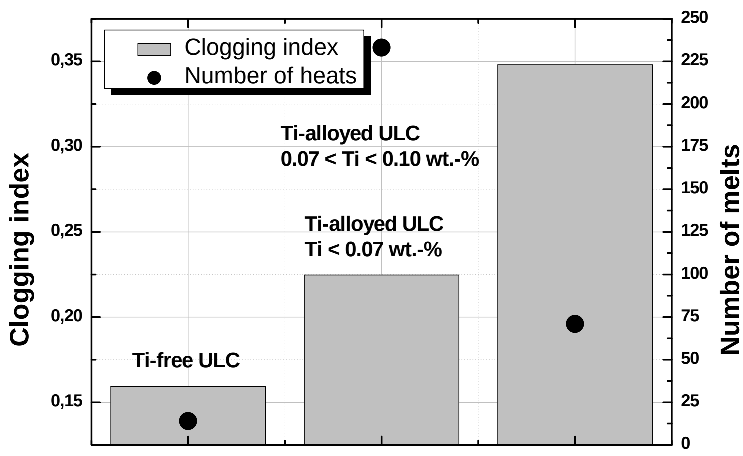


Figure 2. Influence of Ti-content on the clogging index for ULC steel[2].

Kimura postulated that the reaction between the SEN refractory and alloyed Ti favors the adhesion of Al_2O_3 inclusions to the nozzle wall[10]. Recently, Cui et al. showed the existence in clogging deposits of $FeOTiO_2$ phases which hold those deposits together firmly enough to lead to the worse castability of Ti-stabilized ULC steels[7].

The formation of Al-Ti-O inclusions – or at least the formation of an Al-Ti-O-layer covering the Al_2O_3-inclusions - has been given as one of the major reasons for the severity of SEN clogging during the casting of Ti-bearing Al-killed steels: Ruby-Meyer et al. showed that Ti exists in the form of a binary TiO_x-Al_2O_3 phase in the inclusions observed in Ti-bearing Al-killed low carbon steels[11]. Those TiO_x-Al_2O_3 inclusions are reported to be wetted more easily by the liquid steel compared to the pure Al_2O_3 inclusions[12]. As a result, the agglomeration and separation of TiO_x-Al_2O_3 inclusions at the steel/slag interface is inhibited. Basu et al. laid particular emphasis on the existence of Al-Ti-O inclusions covering Al_2O_3 core oxides. Those complex oxides are also wetted by liquid steel[6].

In deoxidation experiments, Sun et al. found a negligible influence of subsequent Ti-addition on the morphology and chemistry of Al_2O_3-deoxidation products. According to their results, Al-Ti-O-particles should more likely be generated by the contact between inclusions and molten slags[13].

In a work by Matsuura et al., the evolution of non-metallic inclusions as a result of Al and Ti additions to molten Fe was investigated[4]. It was asserted that if Al_2O_3 is thermodynamically stable, adding Ti effects a temporary modification of inclusions -both morphologically and chemically - as a result of local super-saturation. The morphology of Al_2O_3 inclusions would then change from spherical to polygonal shapes, which could be the cause of more intense SEN clogging. Wang et al. investigated the effect of a gradual increase of Ti addition on the inclusion evolution after Al deoxidation[14]. They found out that the Ti/Al ratio is a controlling factor for the change of inclusion shape and that a critical value exists between 1/4 and 1/2, which promotes the morphology to be dominated by irregularly shaped inclusions after Ti addition.

Summing up, most authors assume an – at least temporary – interaction between the Al_2O_3-deoxidation products and Titanium, substantially enabled by local non-equilibrium (e.g. super-saturation, contact of particles with slag). Al-Ti-O-phases are more wettable by liquid steel compared to pure alumina. Thus the agglomeration and separation of the inclusions after the Ti-addition could be inhibited. This should finally lead to a larger number of fine particles in the tundish. In this regard, Wilson et al. demonstrated that small inclusions are more sensitive to turbulence than larger ones[15]. How far a Ti-induced change in the morphology of the Al_2O_3-inclusions might also result in a higher clogging tendency is still unclear.

The present work focuses on results of plant investigations and complementary laboratory experiments which have been conducted in order to get a better understanding of the results.

PLANT INVESTIGATIONS

All plant investigations have been performed in the LD III steel plant at voestalpine steel work in Linz, Austria. The metallurgical practice for the production of Ti-stabilized ULC steel grades is described in detail elsewhere[16].

At first, the deposit inside four clogged SENs has been investigated by SEM/EDS. The investigated SENs refer to five casting sequences for different ULC steel grades at caster No. 3, a single-strand slab caster. For the given configuration, clogging most frequently occurs in the stopper area and at the exit port of the SEN. The deposit mainly consists of small sized particles with a diameter of less than 5 µm. The morphology of the particles is mostly regular shaped and dendritic. For the chemical analysis of SEN clogging deposits, SEM/EDS is applied to areas of 22 x 33 µm², each. Ti- and P-content of the ULC steels and the actual composition of the clogging deposits are given in Table 1. The buildup material consists in the mean of between 87 and 98% Al_2O_3 and between 0.58 and 3.58% TiO_2 (balance: MgO, Fe and FeO). Even though the Ti-content in the deposited particles is rather small, it is seen as considerable and increases significantly with increasing Ti/Al ratio in the steel.

Table I. Ti- and P-content of cast steel grades and analysis of the related clogging deposits.

	Steel composition			**Clogging deposition analysis**				
Sample No.	**wt.-% Ti**	**wt.-% P**	**Ti/Al**	**wt.-% Al_2O_3**	**wt.-% TiO_2**	**wt.-% MgO**	**wt.-% Fe**	**Ti/Al**
1	0.067	0.008	1.9	91	2.3	1	5.34	0.028
2	0.024	0.049	< 0.7	98	0.58	0.9	0.4	0.007
3	0.071	0.005	2	93	2.53	0.87	3.16	0.031
4	0.054	0.007	1.4	87	3.58	1.05	8	0.047

In order to compare the composition of the deposited particles with the composition of the inclusions in the steel bulk, samples from four different ULC steel grades with variable Ti and Al contents were taken from the tundish and analysed by means of automated SEM/EDS. The investigated steel grades contain each below 0.0033 wt.-% C, below 0.05 wt.-% Si, between 0.1 and 0.6 wt.-% Mn and 0.035 to 0.043 wt.-% Al, according to a Ti/Al-ratio of between 0.1 and 1.6. For the analytical study, 100 mm² from every sample were prepared for automated SEM/EDS and between 600 and 1000 particles were characterized. The inclusion types found were mainly pure Al_2O_3 and in smaller proportions Al-Ti-O complex oxides. No pure TiO_x particles were detected. The automated SEM/EDS analysis results are presented in Figure 3. An increasing Ti/Al-ratio in the steel sample results in a very low, but significantly rising, mean Ti/Al-ratio in the inclusions. The comparison of the Ti/Al-ratio in clogging deposits and in inclusions in the steel sample shows approximately similar values. These results indicate – without statistical significance – that the deposited particles preexist in the tundish, neither providing the evidence of the mechanism for inclusion formation/modification by the Ti-addition nor the mechanism of the build-up of the deposit in the SEN.

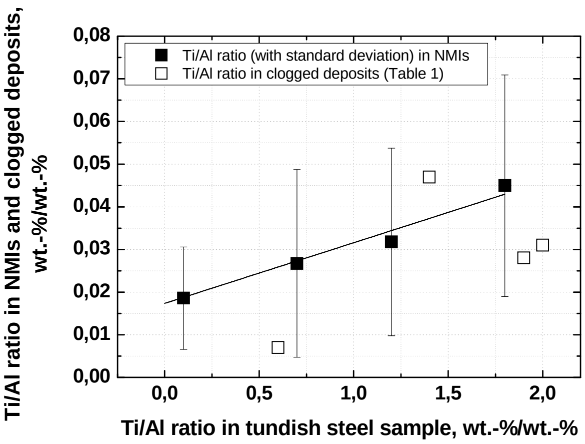


Figure 3. Ti/Al-ratio in NMI vs. Ti/Al-ratio in steel.

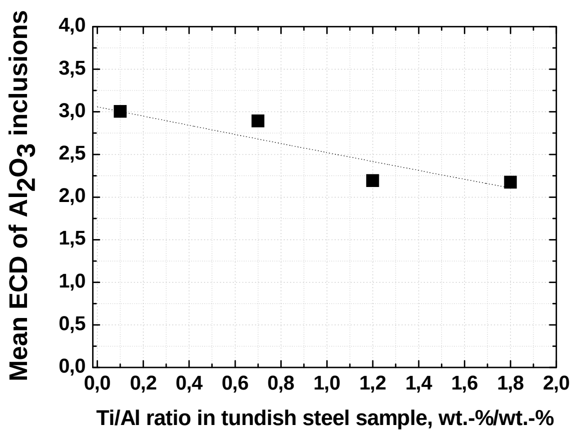


Figure 4. Influence of Ti/Al-ratio in steel on ECD of Al_2O_3.

A further result of the SEM/EDS-investigations is the decrease of the Equal Circle Diameter (ECD) of the Al_2O_3-inclusions with rising Ti/Al-ratio, shown in Figure 4. The shift of the ECD towards lower values is mainly caused by a significantly higher number of small inclusions. This confirms observations as described in literature, such as Ruby-Meyer et al.[11].

In order to strengthen the statistical basis of these results, more than 5000 heats of ULC steel grades with different Ti contents were investigated by OES-PDA. The OES-PDA method allows the determination of a size index for certain types of inclusions[17]. In comparison with SEM/EDS, OES-PDA is less time-absorbing. In Figure 5 the results are categorized in five nominal steel compositions. An increasing Ti-content results in a significant refinement of Al_2O_3 inclusions, in accordance with the results of the SEM/EDS measurement.

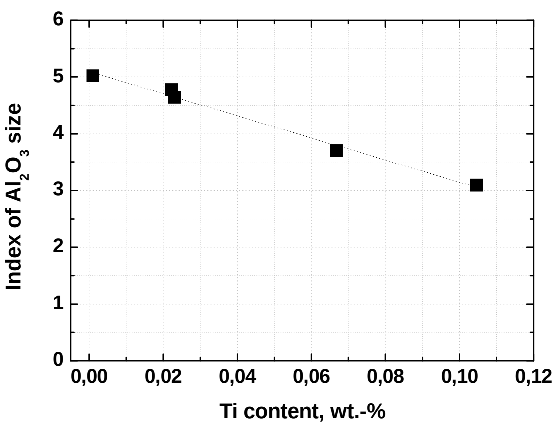


Figure 5. Index of Al_2O_3-size from OES-PDA measurement.

The results of the plant investigations can be summarised as follows:

(1) The metallurgical practice for the production of ULC steel grades at voestalpine results in an increasing Ti/Al-ratio in alumina inclusions in samples from the tundish with increasing Ti-content in the steel.
(2) The content of Ti in the inclusions is rather small. Thus, the inclusions consist not of complex Al-Ti-oxides but rather an Al-Ti-layer covering the Al_2O_3-oxides.
(3) The Equal Circle Diameter (ECD) of the alumina inclusions decreases significantly with increasing Ti-content.
(4) The deposited particles in SENs correspond to the preexisting inclusions in chemistry and shape. The mechanism of clogging by particle deposition seems to be most likely.

These results suggest an – at least temporary – interaction between the alumina inclusions and the added Titanium. The results cannot provide an answer to the question for the reaction mechanism and are restricted to the considered operational practice[16]. The number of small Al_2O_3-inclusions increases with increasing Ti-content in the inclusions. This raises the question for the influence of dissolved Ti on the wettability of alumina inclusions and their possible inclination to agglomerate and separate. This has been investigated by sessile drop experiments, as will be described in the following section.
Finally, the question for the kinetics of the Al_2O_3-Ti-interaction remains open. For this purpose, Al- and Al-Ti-deoxidation experiments have been performed and the chemistry and shape of the initiated inclusions was analyzed by automated SEM/EDS.

EXPERIMENTAL PROCEDURE

Sessile drop experiments

A high-temperature sessile drop system Kruess DSA HT10 was used to measure the wetting angles of molten Fe-Ti on solid Al_2O_3 substrates depending on steel chemistry at temperatures ranging from 1550 to 1620°C[22]. For the measurement in the System Fe-Ti/Al_2O_3, also ceramics from different suppliers were used. These ceramics show a similar chemistry, but nevertheless a different chemical resistance against the Fe-Ti droplet.
The experiments were conducted in a horizontal tube furnace with a linear heating process (rate: 15°C/min). A holding time of 10 min at 1620°C was followed by cooling down at a rate of 10°C/min. Once the sample was melted, the measurements of the contact angle as a function of temperature and time started. They consisted in a continuous monitoring of the shape of the sessile drop by a digital video camera connected to a computer, enabling automatic digital image analysis. The characteristic dimensions of the droplet were extracted with an accuracy of ± 2° for θ and ± 2 % for R and H. The oxygen partial pressure was kept substantially below 10 ppb by use of an oxysorb system and a Ti-getter in the furnace.
The results of the wetting experiments in the Fe-Ti/Al_2O_3 system together with data from literature are presented in Figure 6. Two different types of general dependencies can be identified: Zhong et al.[19] and own experiments on stable ceramics show only a relatively weak effect of increasing Ti-content on the wetting angle. An increase in the Ti-content to 0.2 wt.-% lowers the wetting angle by only 5°. In contrast, results of works by Kishimoto[20] and Ueda et al.[21], but also own experiments on less stable ceramic substrates show a remarkably increasing wettability with increasing Ti-content. Just a moderately lifted Ti-content of 0.174 wt.-% reverses the system Fe-Ti/Al_2O_3 from non-wetting to wetting ($\theta < 90°$).
The reason for these contradictory results can be understood from Figure 7[2], an SEM image of a cross-section of the interfacial zone droplet/substrate after a wetting experiment on a less stable ceramic. An interfacial layer composed of complex oxides has formed between the droplet and the substrate. After experiments on Fe-Ti alloys with Ti contents < 0.1 wt.-% and low oxygen content, the reaction layer between the droplet and the substrate is rather thin and consists mainly of $FeAl_2O_4$ with low content of Ti. In the case of experiments under higher Ti-contents, this layer gets continuously thicker and richer in Ti. As soon as a layer covers the interface between the droplet and the substrate, the measured wetting angle represents the wetting angle between steel droplet and interfacial layer. This phenomenon is referred as to reactive wetting. Even for a low-Ti-containing $FeAl_2O_4$ layer, the wetting angle decreases remarkably. For higher Ti-content, the system reverses to a wetting system with $\theta < 90°$.
The presented results give not an answer to the question for the existence of an Al-Ti-O-phase after Al-deoxidation and subsequent Ti-addition in the production of ULC steel grades. But if such phases form, and even if these phases cover only the surface of the alumina inclusions, the wettability of these inclusions will be remarkably increased. The tendency towards agglomeration and separation of the inclusions will decrease. If the existence of Al-Ti-O-phases is only temporary, the alumina inclusions might finally again consist of alumina with only a small amount of Ti in them, but the number of small alumina inclusions might be higher.
In order to simulate the transient behavior of aluminates after Ti-addition, experiments in a Tammann type furnace as will be described later on.

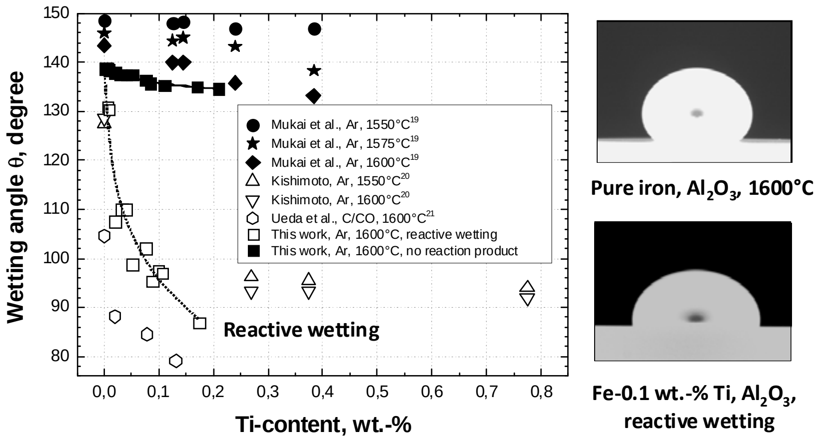


Figure 6. Measured wetting angle in the system Fe-xTi/Al_2O_3 with data from literature[19-21].

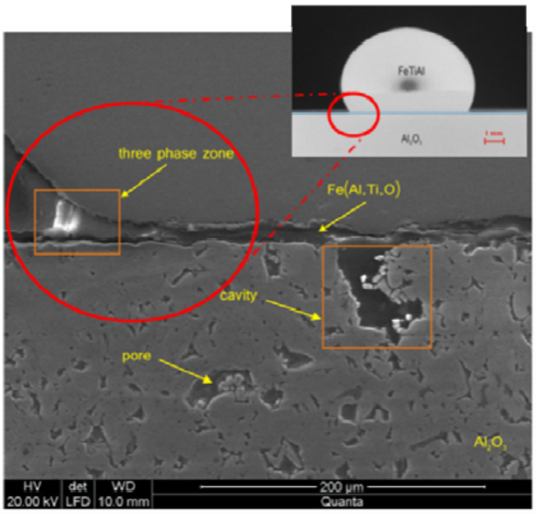


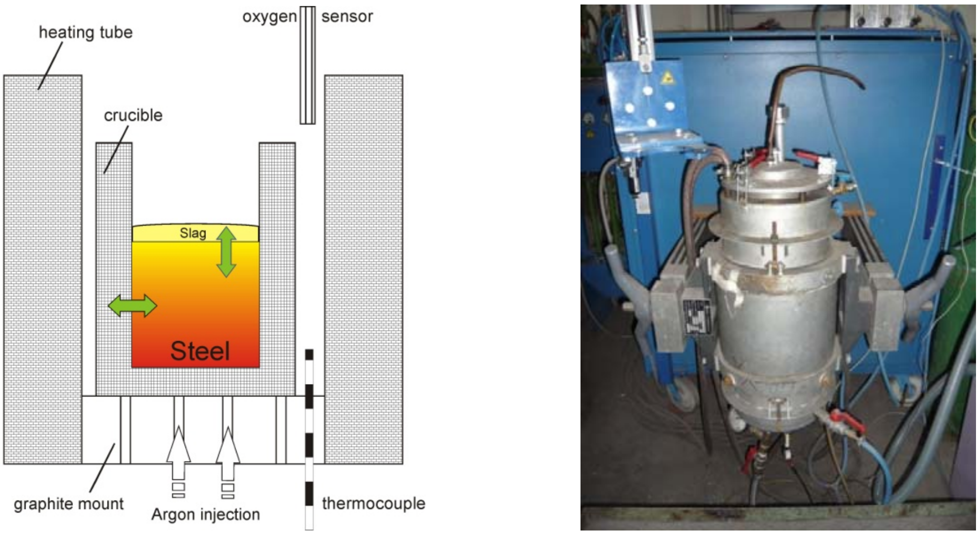
Figure 7. SEM of interfacial layer between Fe-0.0065 wt.-% Ti droplet and Al_2O_3 substrate[2].

Transient behavior of oxides after Al/Ti-deoxidation experiments

The experiments to study the transient behavior of oxides in laboratory scale have been conducted in a Tammann type furnace. This is a high-temperature electric resistance furnace, which can be heated up to 2000 °C. Due to the carbon heating tubes inside the furnace and their reaction with the residual oxygen, the experiments are performed under slightly reducing atmosphere. The schematic experimental setup for the remelting experiments as well as the furnace itself is shown in Figure 8, details for the experimental setup can be found in[23]. All experiments in the Tammann furnace are carried out under Ar-atmosphere (Argon 5.0), using a sample weight of 500 g commercial ULC steel with the composition listed in Table 2. The steel is remelted in an MgO-crucible together with FeO. The initial oxygen content in the remelted steel amounts to 300-400 ppm, similar to the oxygen activity after RH-treatment of ULC steels[16]. After reoxidation the steel was deoxidized with Aluminium (1 Al-Ti TM) or simultaneously with Al and Ti (2 Al-Ti TM) or with Al and subsequently with Ti (3 and 4 Al-Ti TM). After a holding time of two minutes, the samples solidified under accelerated cooling. Finally, the inclusions in the samples were analyzed by automated SEM/EDS.

Table 2. Composition of raw material for experiments and samples after Al/Ti-addition.

	wt.-% C	wt.-% Si	wt.-% Mn	wt.-% Al	wt.-% Ti	Deoxidation practice
ULC steel	0.0020	0.0010	0.0740	0.020	0.0040	Raw material
1 Al-Ti TM	0.0148	0.012	0.0691	0.065	0.002	Al-deoxidation, 2 min holding time
2 Al-Ti TM	0.0138	0.011	0.0721	0.058	0.105	Al-deoxidation and Ti-addition at the same time, 2 min holding time
3 Al-Ti TM	0.0127	0.012	0.0732	0.054	0.11	Al-deoxidation Ti-addition after 2 min, 2 min holding time
4 Al-Ti TM	0.0143	0.013	0.0698	0.062	0.109	Al-deoxidation Ti-addition after 5min, 2 min holding time



a) b)

Figure 8. a) Schematic illustration of the test arrangement and b) photograph of the Tammann furnace used for laboratory experiments[23].

The results of the automated SEM/EDS analyses on the four samples are summarisein Figure 9. The chemical composition of all detected inclusions is represented in the quasi-ternary system Al-Ti-O. The inclusions are classified in four categories: *Alumina* inclusions (containing neither Ti nor N), *Ti-oxides* (containing neither Al nor N), *AlTi-oxides* (containing no Nitrogen) and finally *Alumina inclusions with TiN*, see Figure 10. It is worth noting that the alumina inclusion population includes also pre-existing alumina particles from the ULC steel and also reoxidation products. However, the use of a Ti-free ULC steel guarantees that only alumina inclusions preexist and prevents the introduction of other – and more difficult controllable – inclusions or trace elements.

Experiment 1 Al-Ti TM, deoxidation with Al, proves that no other oxides beside Al_2O_3 exist. The simultaneous addition of Al and Ti (2 Al-Ti TM) results again mainly in the formation of alumina inclusions but also in a significant number of Al-Ti-O inclusions. The number of Ti-oxides is very limited and so is the number of alumina inclusions with surrounding TiN. These categories will further on not be discussed in detail. The category *Al-Ti-oxides* consists in surn mainly of two populations: Ti-rich inclusions with a low Al-content (TiO_x and $TiO_xAl_2O_3$) and Al-rich inclusions with low Ti-content (Al_2O_3 with $TiO_xAl_2O_3$). The Ti/Al-ratio of the lowTi-containing inclusions (Ti/Al < 1) is depicted in Figure 11.

Experiment 3 Al-Ti TM, addition of Titanium 2 minutes after Al-deoxidation, results in the formation of a very similar inclusions population with one significant difference: The total number of the AlTi-oxides is significantly lower and there exist only few Ti-rich Al-Ti-oxides. This trend is continued with further increasing the waiting period between Al- and Ti-addition: In sample 4 Al-Ti TM, nearly all Al-Ti-oxides are close to the composition of pure Al_2O_3.

From the results of these experiments one can draw the following conclusions:

(1) The addition of Al and Ti to the reoxidised ULC steel results under the present conditions mainly in the formation of Alumina inclusions. However, also Al-Ti-oxides form. In most inclusions of this category, Ti is evenly distributed over the entire inclusion and not enriched in a surface layer. This justifies the conclusion that the Al-Ti-oxide type inclusions nucleate after the Ti-addition. The preexisting alumina inclusions seem to be largely unaffected.

(2) These results seem to be consistent with thermodynamic considerations: According to the data published by Matsuura et al.[4], a high initial supersaturation of Al, Ti and Oxygen while simultaneously adding Al and Ti should result in the preferential formation of Ti_3O_5 and Al_2TiO_5. After reducing the oxygen activity, Al_2O_3 and Al_2TiO_5 will become the most stable reaction product and finally, at very low oxygen activity, only Al_2O_3 should form. When Ti is added after the Al-deoxidation, viz. with lowest oxygen activity in the steel, the assumption of a local cooling of the melt around the FeTi-particles together with a high local supersaturation would result in the formation of Al_2O_3 and – to a lesser extend - Al_2TiO_5. The nucleated alumina particles would then be very small, difficult to separate and might contribute significantly to the higher clogging tendency.

The waiting time between Al- and Ti-addition seems to be a key parameter to control the number and composition of alumina inclusions containing Ti. However, conclusions on the industrial process have to be drawn very carefully. The absence of both a forced convection and covering slag in the experiment results in a massive inhibition of the separation of inclusions. Additionally, the absence of a covering slag changes the thermodynamic system.

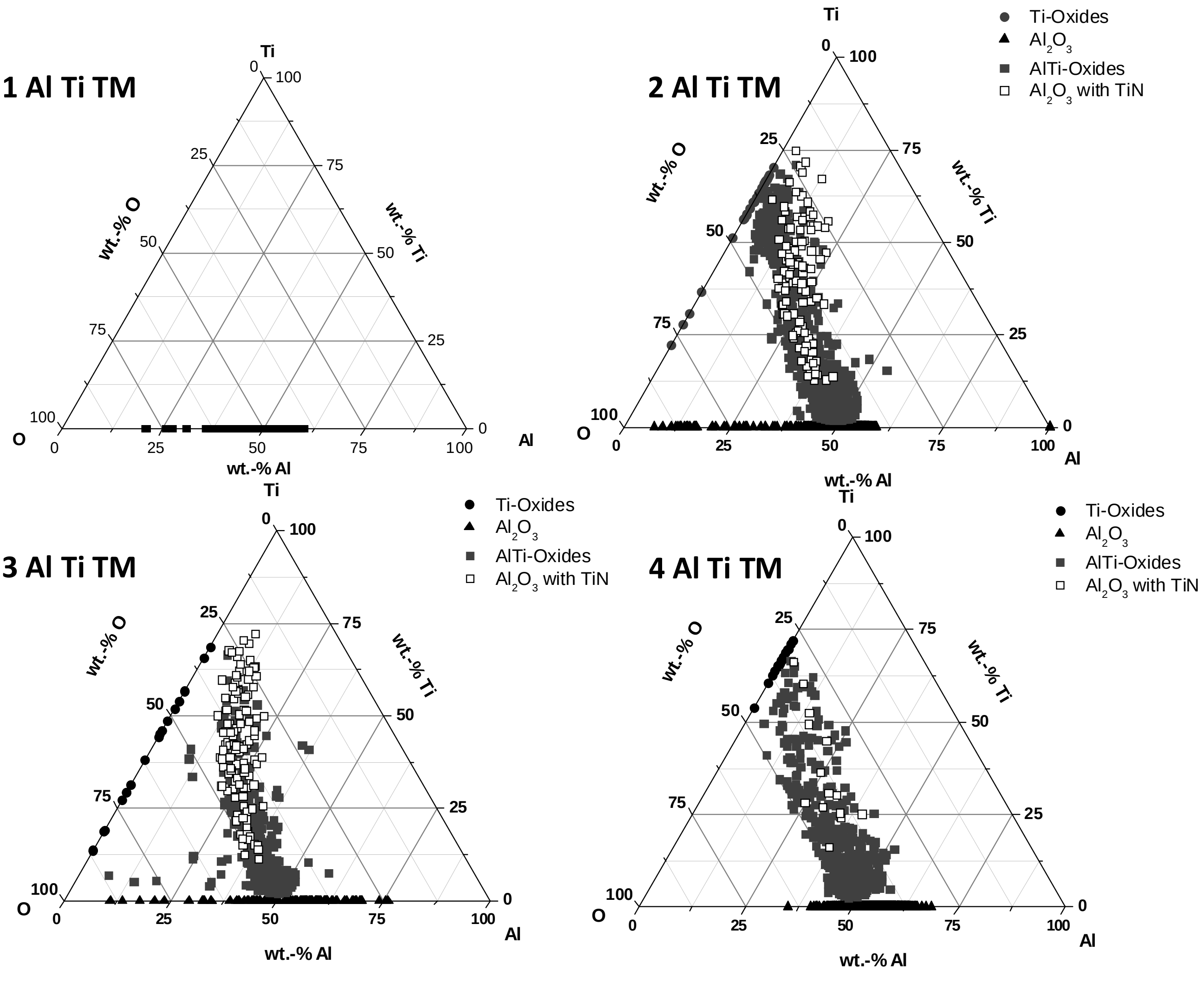


Figure 9. Composition of the inclusion population in the quasi-ternary system Al-Ti-O.

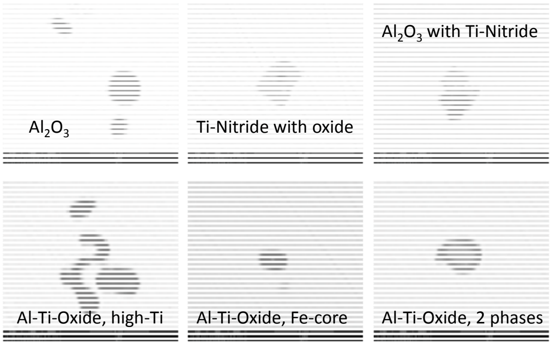


Figure 10. Classification of inclusions.

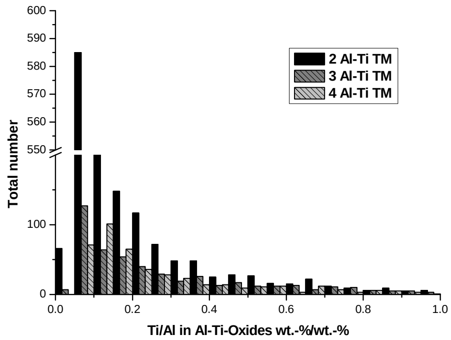


Figure 11. Ti/Al-ratios lower than 1 in Al-Ti-Oxides.

In a further test campaign, different Ti/Al-ratios have been adjusted. The FeTi was again added 2 minutes after Al-deoxidation and cooled down after additional 8 minutes. The results confirm the plant observations: An increasing Ti/Al-ratio results in a significant decrease of the mean diameter (ECD) of the alumina inclusions. Again, Al-Ti-oxides and aluminates form after the Ti-addition and the preexisting aluminates remain unmodified.

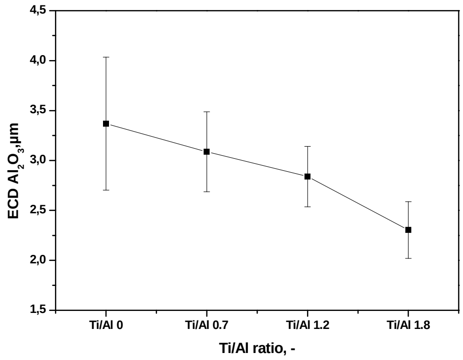


Figure 11. ECD of Al_2O_3-particles versus Ti/Al-ratio of the steel.

SUMMARY AND OUTLOOK

The present work focuses on plant investigations and laboratory experiments on the castability of Ti-stabilized ULC steel grades. The results of the plant investigations can be summarised as follows:

(1) Ti-alloyed ULC steels show a significantly higher clogging tendency, compared to Ti-free ULC steel grades.
(2) SEM/EDS investigations show that an increasing Ti/Al-ratio in the steel results in both an increase of the Ti/Al-ratio in microscopic aluminates and a decrease in the ECD of alumina inclusions. The latter has been confirmed by OES-PDA investigations on a total number of 5000 heats.

From literature it can be concluded that a higher number of small alumina particles in the steel increases the clogging tendency significantly. In order to understand the interaction between Ti addition and the existence of a large number of small inclusions, laboratory experiments have been conducted with the following results:

(1) Ti increases the wettability of alumina particles by liquid iron only in case of reactive wetting, viz. the formation of a complex (Al, Ti, Fe)-oxide interfacial layer between steel and inclusion. A significant influence of Ti-addition on the agglomeration and separation of aluminates could thus only be expected in case of the modification (e.g. the formation of an Al-Ti-O surface layer) of a large quantity of alumina inclusions by Ti.

(2) In laboratory experiments, the addition of Ti some minutes after Al-deoxidation did not result in a modification of already existing aluminates but in the nucleation of additional alumina inclusions and Al-Ti-oxides. This contributes significantly to the decrease of the mean particle diameter and an increase of the mean Ti/Al-ratio in the inclusions.

Conclusions on the process have to be drawn very carefully as the experimental conditions are far away from the conditions during an RH-treatment. Thus, further experimental work with a wide variety of different initial conditions and under consideration of a covering slag will be necessary.

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