

## Microstructures of Mould Slag Crawling at the SEN during Thin Slab Casting

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**Summary:** Experiments have been performed to investigate the morphology and chemistry of the interaction between mould slag, steel, and SEN refractory that are potentially happening during slag crawling. The experiments were designed to capture the initial stages of the reactions between original reactant partners. It was found that mould slag completely wets the refractory interface to steel, preventing steel-refractory contact. Mould Slag simultaneously reacts with the steel exchanging Si for Al; dissolves alumina from refractory, and undergoes silica reduction by carbon of the refractory. The net effect is that mould slag changes into a mixed silicate-aluminate composition which remains fully liquid and mobile, able to spread out across the submerged surface of the SEN, linking the more reactive parts to the mould slag by intense reaction driven flow.

### Key Words

Slag crawling, continuous casting, Submerged Entry Nozzles, chemical reaction, refractory wear, microstructures

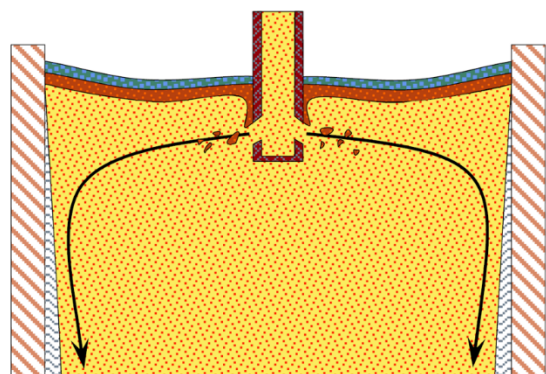
### Introduction

The cleanness of as-cast steel is dependent on potential contamination processes in the mould, especially the entrainment of mould slags and other materials present along the interfaces of the liquid steel in the mould. Many entrainment processes are known, as described in (Hibbeler) and measures to limit them are an important part of current efforts to improve steel cleanness. Among the entrainment processes described in Hibbeler [1], the occurrence of „slag crawling“ (infiltration of mould slag downwards between submerged entry nozzle and liquid steel) is a well known unwanted behaviour (See Fig 1 adapted from [1]), but it has found relatively little attention in recent work on steel-mould slag interactions. This is in contrast to the potentially strong impact its occurrence can have both on steel cleanness (by entrainment of slag from the SEN walls and port) as well as on SEN wear and lifetime.

### Morphology of Refractory Wear by Slag Crawling

At the thin slab caster at the Direct Sheet Plant (DSP), IJmuiden Works, Tata Steel Europe, occurrence of mould slag crawling was noted intermittently in recent years. In the course of post-mortem work on SEN wear and lifetime it was observed that slag and slag-steel-refractory interaction products cover the surface of the submerged SEN, and that specific areas of refractory wear occurred below steel meniscus that gave cause for concern. At the DSP, plain carbon and HSLA steels, among others, are cast through a 9 cm wide mould. The SEN is a classic four-port fishtail design

with a diamond-shaped submerged part that creates a double roll flow converging at the meniscus towards the SEN where the steel flow is directed downward [5]. The mould slags used, slightly variable per steel grade, are all variants of the fluorinated sodic calciumsilicate liquids (system CNS-F) that are commonly used in steel casting. As all steels cast are Ca treated, no Ar is used, which is beneficial for mould meniscus level stability. As the SEN is stopper controlled, this implies a potentially severe dynamic underpressure in parts of the SEN during casting [2]. Fig. 2 shows an example of the appearance of a SEN with indications of slag crawling after the casting. The post-mortem SEN's affected by this display both areas with excessive refractory wear, as well as areas with material buildup, over the inner and outer



Graphics: Lance Hibbeler et al, 2007-2010

*Fig. 1: Schematic sketch of mould slag crawling in a mould during the casting of steel, and its potential for slag entrainment into steel. From [1]*

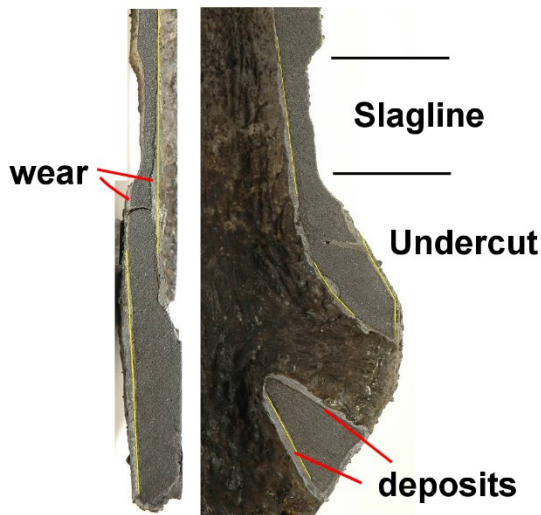


Fig. 2: Example of a post-mortem SEN of the type used at DSP, Tata Steel Europe, showing slag crawling and associated refractory wear. Note especially the „Undercut“ formation at the interface between zirconia-carbon antislack band and alumina-carbon body material.

surfaces of the SEN. Inspection of SEN's shows that the SEN wear/shape change occurs not always, but when it occurs, it has a consistent morphology. The most prominent, and for casting conditions most critical, location of refractory wear is the so-called Undercut – the lower end of the anti-slag corrosion zirconia-carbon (ZC) refractory band along the outsides of the SEN, where it contacts the alumina-carbon (AC) refractory of the general SEN body. An example of a severe Undercut is shown in Fig. 3 as optical overview image of a post mortem mount. Along the contact interface between the ZC and AC refractories, a narrow slit-like incision is formed in the refractory. At the broad faces of the fishtail SEN, this slit can break through to the inner side of the SEN while on the edges refractory integrity is still preserved. Notable features of the overall wear pattern indicate that the refractory wear is connected over the entire submerged surface of the SEN by a common refractory wear mechanism, of which the local processes at the various refractory-slag-steel interaction points are building blocks. On the inside of the SEN, the relatively thin band of AC refractory that backs up the backside of the ZC antislack corrosion insert, is being attacked from the inside of the SEN bore itself; this happens even in cases when the Undercut has not broken through, so is not itself a consequence of breakthrough and mould slag access to the inside. From the submersion depth (inside of the SEN on, deposits formed on the inside of the SEN, generally of a calcium aluminate makeup,

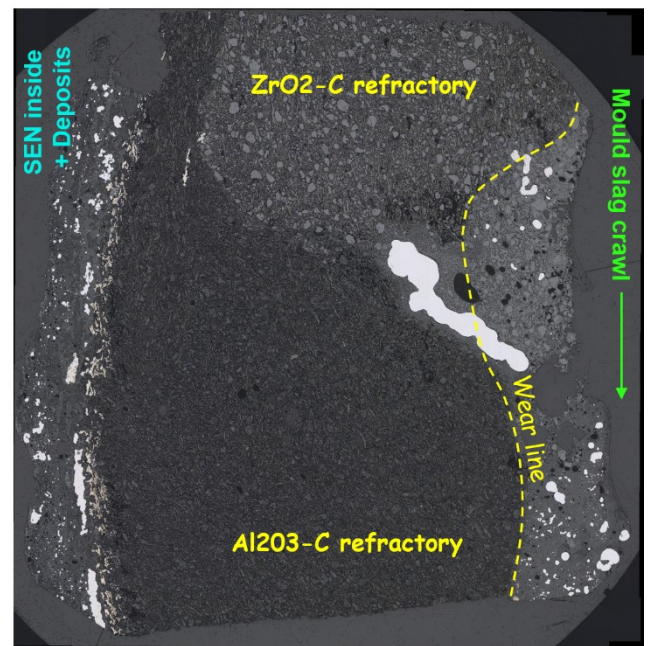


Fig. 3: Cross section through a post mortem SEN at the position of the „Undercut“ after a case of mould slag crawling. Reflected Light optical image, original wall refractory thickness 3 cm between SEN inside (left) and submerged outside (right). The materials are indicated. Note the deep, steel-filled cut into the SEN at the interface between the two refractories.

increase rapidly in thickness with depth, and they coat all the inner SEN surfaces, extend through the ports to the outer surface, and form the bulk of the growth (deposits) observable on the outer SEN broad face in Fig. 2. They are capped by the Undercut position, above which the SEN outer surface is smooth and slag covered leading up to the regular refractory slag line at the mould slag pool height. Further features indicating that location-specific reactions occur along the SEN surfaces in the affected cases is the appearance of a band of FeSi mixed-alloy droplets (liquid at casting temperatures) along the inner surfaces of the SEN below ZC anticorrosion band heights, and of a decarburised band of ZC refractory along the outer surface of the SEN, between slagline proper and undercut, which does not appear in cases apparently not affected by slag crawling.

## Experiments

From this overview of the morphological complexity of refractory wear in connection with slag crawling, it is clear that the mouldslag – refractory – steel interactions at this caster are not properly described by the classic Mukai et al [3] description of mould slag – SEN wear. Far more complex interaction occurs, but even at the same caster it occurs only under some, not all conditions. Thus, this interaction with its potential to degrade cast and product quality,

**Table 1: Experimental Starting Compositions Refractory and Slag**

Starting Material compositions in wt% oxides			
	R1	R2	M (*)
F2	-	-	8.2
Na <sub>2</sub> O	0.90	0.2	13.0
MgO	tr	tr	tr
Al <sub>2</sub> O <sub>3</sub>	65.9	42.5	3.2
SiO <sub>2</sub>	5.23	27.3	35.6
ZrO <sub>2</sub>	5.13	0.31	-
CaO	tr		35.1
TiO <sub>2</sub>	~ 0.7	tr	tr
MnO	-	-	-
FeO	tr		tr
C	22.71	29.3	(*)

tr = traces. (\*) = fused and decarburized.

**Table 2 Experimental Materials Steels**

Starting Materials – Steel Compositions, ppm or wt%							
	C%	Mn%	S	Si	Al(*)	Ti	Cr
S1	0.15	2.08	50	1060	7140	210	2000
S2	0.05	0.21	130	20	500	-	22

(\*) = soluble Al (excluding inclusion bound)

needs to be understood at a more fundamental level. To investigate the mechanisms of the interaction between steel, refractory and mould slag, an experimental program was designed to simulate processes under laboratory conditions. The structural observations described above suggest that the interaction may be partly controlled by a dynamic force balance between interfacial tensions (all of liquid-liquid, liquid solid, and liquid gas are involved), effects of chemical reaction on these interfacial tensions, and physical forces (shear and buoyancy) acting on the potential slag liquid film intercalated between SEN and liquid steel batch. This dynamic situation can not be easily reproduced in a static experimental test. Instead, in a static test with given amounts of steel slag and refractory, the fixed-reservoir sizes will soon change the reaction development to deviate from the situation in actual open-system casting conditions. Therefore, we designed slag-steel-refractory interaction experiments to minimize the interaction time and thereby approach the reaction conditions that are encountered at the onset of interaction in the real system.

The experiments had as goal to establish the initial morphology of the three-way refractory, slag, steel contact point, and the way it would change under the influence of incipient reactions between the three reacting partners. They were set up as conventional cup tests with a refractory cup made from the refractory of interest, into which steel and slag are placed. The cup tests were run at temperature similar to real world casting temperatures (1570 C), and

within a vertical-tube 1 atm gas mixing furnace (Gero HTRV). Refractory Cups were drilled out of as-delivered green SEN's meant for production. Of these three, data for two test series including refractories R1, R2 are reported here. The approximate composition of these, as measure by in-house analytical department by XRF, is given in Table 1. The experiments were run using a mould slag similar to the ones used in industrial practice, and using two different steels, which are distinguished by their alloyed Al content: a low Al steel and a high-Al steel (Compositions of the mould slag in Table 1, of the steels in Table 2).

The cylindrical refractory cups had bottom thickness and inner cup diameters of 3 cm with wall thicknesses of 2 cm and wall height of 5 cm. Typical cup weights were 130 grams. During experiments, the furnace was flushed with 2 l/min Ar and another 1 l/min Ar was blown directly into the cup through an alumina capillary, to ensure that CO production from carbothermic reactions within the refractory would not overwhelm the experimental charges. An alumina sheath around the outside of the refractory protected the oven tube against potential splashing. For these experiments, the different melting temperatures of the planned reactants is a challenge. The cups were ramped up by a mechanical lift system into the hot zone of the furnace, but the melting points of mould slag and steel are so different that simple charging of these would mean that the mould slag would have changed its composition already substantially by reaction with the cup before the steel even melted, and the target of the experiments – instantaneous reaction between the three fresh materials – could not be achieved. To avoid this, mould slag was decarburised, prefused and crushed, and then densely packed as powder into small containers of the steel chosen for the experiments, with lids to be screwed tight before heating. Test runs showed that this encapsulation of mould slag powder into steel containers did not leak significant amounts of molten mould slag before experimental temperature (1570C), avoiding premature reaction and devolatilisation of the mould slag. On reaching experimental temperature, steel containers melted down, liberating the mould slag; thus, refractory was simultaneously exposed to molten steel and molten (C-free) mould slag, as it is in the real process. Mass of the steel capsules were 45 gram, and mass of the mould slag filling was 7-8 gram.

Experiments were conducted for 1, 5, and 10 minutes of nominal duration. Experimental start time was taken as the time when a co-installed thermocouple sensor lost signal. It was found by examination that the signal loss was due to the exposure of its Pt wiring to a sudden flush of Na, F,

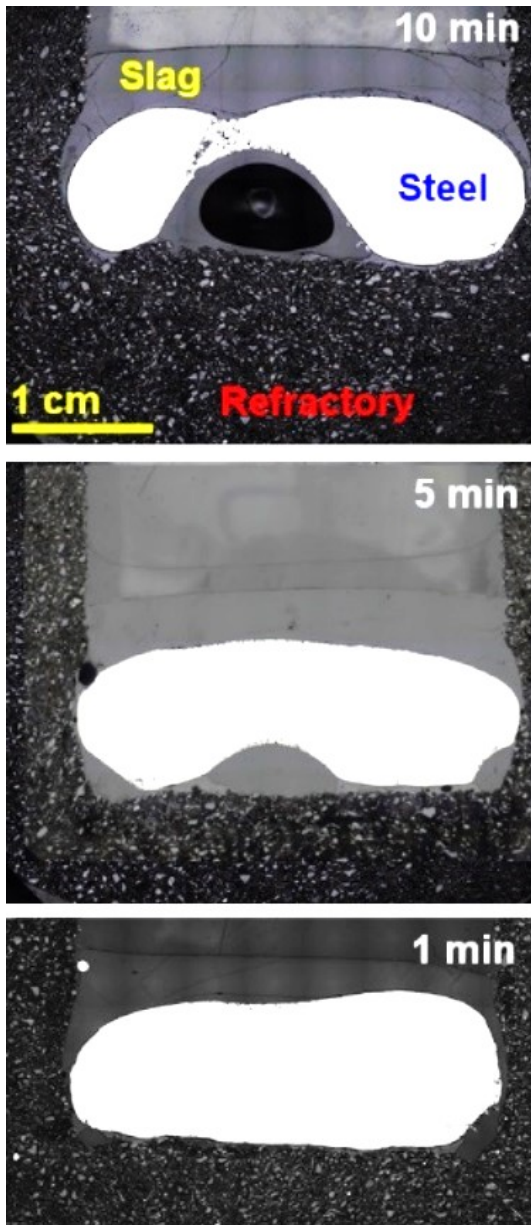


Fig. 4: Cross Sections through experimental cup tests. Refractory R1, Steel S1, and mould slag M, after 10, 5, and 1 min nominal interaction time respectively, from top to bottom (Reflected Light Optical Images).

and SiO containing gas, which reacted with the Pt, marking the liberation of mould slag and its immediate reaction and devolatilization. After nominal holding time from this point on, experiments were terminated, cooled by bringing the cups into the oven's cold zone, and fixed by impregnation with epoxy.

Experimental Cups were sectioned in half and prepared for optical and SEM microscopy and microanalysis by SEM EDS as well as by EPMA. Microanalysis of slag and oxide phases was done standardless on a Zeiss Ultra55 FEG SEM with EDAX analytical system; steel compositions were

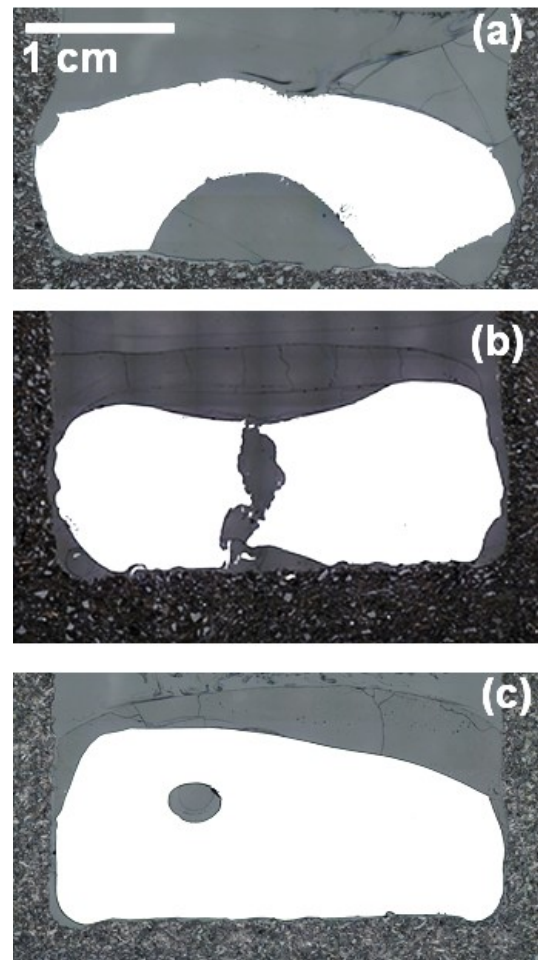


Fig. 5: Cross Sections through experimental cup tests. (a) R1, S2, M after 10 min holding time. (b) R1, S2, M after 1 min holding time. (c) R2, S1, M after 1 min holding time.

analysed for Si, Al, Mn, Cr on a Cameca SX100 EPMA system using pure metals as standards. Homogeneity of the steel and slag were tested by running linescans. Using the compositional data, reactions in the experiment were mass balanced, by measuring the amounts of reaction products (remaining refractory, slag, and steel) from optical images of the sectioned cups assuming rotational symmetry; and balancing the element streams using available data and general phase densities.

## Results

It was found that the steel charge had melted completely in all experiments even after only 1 min holding time. The post-experimental cup cross sections for 1, 5, and 10 minutes interaction time are shown in Fig. 4. for the combination R1-M-S1. As can be seen there, the mould slag has in all experiments completely escaped the original steel encapsulation and formed the desired configuration of a pool of slag sitting atop a body of molten steel that is shaped by the equilibrium between its surface tension and gravity. Further, we found in all experiments that

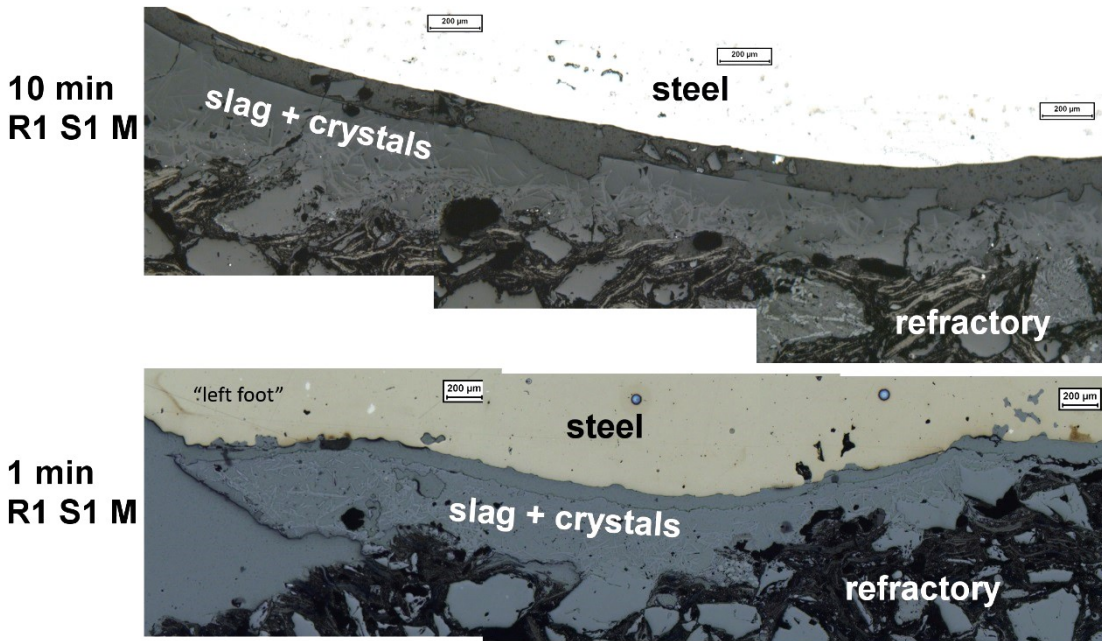


Fig. 6: Appearance of the interface between refractory cup bottom and underside of the molten steel charge in the experiments R1-S1-M after 1 and 10 min holding time (see Fig. 4-5. for full overview images). Optical reflected light images, the lower one with carbon coating. Small scalebars indicate 200 micrometer.

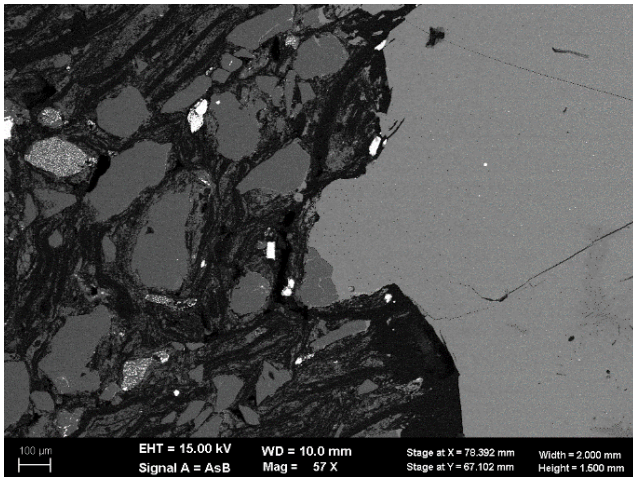
there is strong refractory wear, which is not unexpected as the slag is highly undersaturated in alumina. Morphologically, the wear is concentrated at the sidewalls of the experimental charge, in form of an „elephant foot“ or slagline-like excavation around the sides of the liquid steel body. In comparison of the experimental holding times, it can be seen that the extent of this refractory wear increases significantly from 1 over 5 to 10 minutes. Further, it is found that there is extensive development of a gas phase at the refractory surface inside the cup, which appears in form of small to large bubbles accumulating at the lower side of the liquid steel. As visible in Fig. 4, this accumulating gas tends to punch upwards through the center of the steel pill and not escape sideways along it. Whether the observed sizes of the gas bubbles reflect the gradual buildup of gas quantity over 10 minutes, or simply snapshots of a succession of gas accumulation, escape, and reaccumulation, is not known.

Fig. 5 shows cross sections through cup tests using the refractory R1 with the low-Al steel S2 (top and middle), as well as the refractory R2 with the high Al steel S1 after the shortest run time of 1 min (bottom), to compare with the time series in Fig. 4. In the two 1-min sections, the configuration of the central cavity in the visible molten metal can potentially still be affected by a relict of the mould slag encapsulation, however mould slag pool formation has taken place in all experiments. The main feature of interest in the mould slag / refractory / steel morphology of the experimental charges is that mould slag has formed a liquid film all around the steel, in all cases using the

silica-poor refractory R1. The steel does not only not touch the sides of the refractory cup in the „slag line“ refractory wear zones, but even at the bottom of the cup, a thin layer of slag is found to on which the steel rests, at no point is there direct contact between liquid steel and the refractory. This is illustrated in the following using detail imagery.

Fig. 6 shows the undersides of the steel charge after 1 and 10 min in the R1-S1-M experimental setup. There is very little difference. In both cases, a zone of idiomorphic crystals has formed in reaction between slag and refractory, overlain by a zone of liquid, crystal free slag, that also did not crystallize upon cooling. The steel has separated from this slag due to differential contraction upon cooling, but the slag and steel otherwise present congruent and smooth outlines interrupted by only a few bubbles, indicating that the steel-slag interface is observed in its syn-experimental state. At the sides of the experimental charges, where the slag pool infiltrates along the steel, the reaction between slag and refractory proceeds via preferential dissolution of contacted alumina grains, the carbon bearing matrix tends to stand out into the slag, but a coherent wear interface is nevertheless formed. Notably, the slag is bridging the significant porosity of the refractory and is in contact with the gas phase within the refractory.

A ternary triple point is observed in the experiments in the cases where a large gas bubble has formed and is frozen in below the steel pill before its eventual escape upwards. In the gas bubbles, gas is in direct contact to steel. Along these interfaces, in some



*Fig. 7 – Sidewall contact between Slag infiltrating next to steel, and refractory, in the R1-S1-M experiment. SEM BSE image, Slag at right. Bright particles are zirconia containing aggregates.*

experiments a thin (1-5 micrometer) sheath of alumina is visible tracing the steel surface. In the experiments using R1-S2-M setup, that is, interaction between refractory, mould slag and a low aluminium steel, the general morphological relations are not different, but the amount of reaction is less. In the short duration experiments, the slag-line like refractory consumption along the sides of the steel is barely visible, and the slag pool is not quenched to glass but rather quenched to variable amounts of cuspidine dendrite networks with intervening glass or other secondary crystals (nepheline). In these experiments too, the bottom of the cup shows the development of continuous layers of reaction crystals, between slag and refractory, which can morphologically be distinguished from quench crystals of the glass upon cooling. In both series of experiments, there is a tendency for the reaction crystals at the bottom of the cup (in the thin slag layer between refractory and steel) to be CA6; whereas crystals formed at the sides of the steel tend to be reprecipitated alumina.

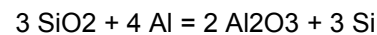
The steel itself shows systematically variable degrees of carburization during the experiment. In the 1-5 min experiments it is negligible to the extent that no traces of cementite can be found, implying that C uptake is restricted to ~ 300 ppm or less; in the longer experiments, patches of pearlitic cementite can be found. Along with the uptake of carbon in the 10 min experiments goes a change in the solidification behavior, with more pronounced formation of a dendrite – shrinkage porosity structure in the more carburized experiments.

### Compositional Changes

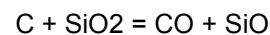
The compositional development of the slag in the experiments can be seen by comparison of Tables 3

and 4 with Table 1. As is to be expected, the mould slag being highly alumina undersaturated with only about 3 wt% Al<sub>2</sub>O<sub>3</sub> initially, aggressively attacks the alumina-based refractory. This is stronger in the series with steel S1 (Table 3) than in the low-Al steel series (table 4), in the former, the Al<sub>2</sub>O<sub>3</sub> content exceeds 20 wt% already after 1 min nominal contact time, while it only reaches 16% in the experiment with low Al steel. The mould slag in all experiments is found to be homogeneous – no compositional gradients were found either across the cup or over the depth of the slag pool atop the steel in all experiments. In those experiments that have partially crystallised on cooling (cuspidine), experimental slag compositions (as in Table 4) have been reconstructed by image analysis of the apparent phase abundances. Next to the expected alumina uptake, it must be considered that the reaction system in the experiment is not simple. The mould slag does not only attack the refractory, but as is well known, at the same time it reacts with the steel. Further, the mould slag is exposed to the atmosphere of the furnace, losing volatile elements and compounds to this atmosphere. This volatile loss accounts for the large decreases in F and Na contents of the slags after the experiments.

The reaction between mould slag and steel is characterized by the oxygen-conservative exchange:



For the slag, this exchange means that silica reduced by the steel is replaced by Alumina from oxidized Al from the steel. This creates a different compositional vector compared to the simple Al<sub>2</sub>O<sub>3</sub> dissolution into mould slag. The third reaction changing Al-Si relations in the slag is the reduction of silica by carbon of the refractory:



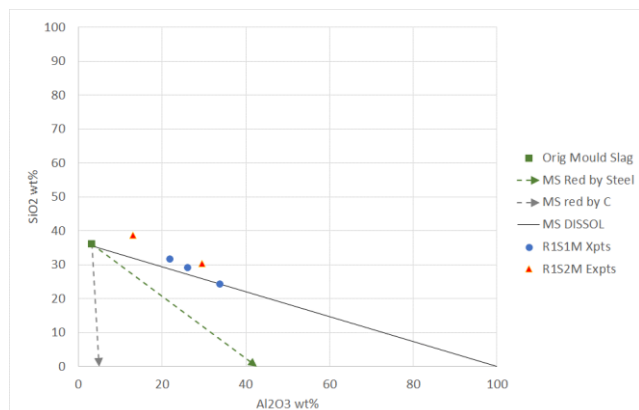
The compositional development of the slag in terms of Al<sub>2</sub>O<sub>3</sub> vs SiO<sub>2</sub> in the experiments with refractory R1 and the steels S1 (high Al) and S2 (low Al) is shown in Fig. 8. The slag composition develops largely along a path consistent with the alumina dissolution from refractory being the dominant reaction. This does not mean that the other two reactions do not run. To the contrary, their operation is shown by the development of the composition of the steels as analysed by EPMA. For these, two linescans are shown in Fig. 9. The steel compositions show significant change, as is most clearly seen in the case of the high-Al steel S1 (see Fig. 9): from ca. 0.7 wt% Al, 0.1 wt% Si, this steel changes to 0.9 wt% Si and < 0.1 wt% Al after 10 min interaction, and after only 1 minute, Si is already up to 0.6 wt%. Thus, the steel does react heavily with the mould slag

**Table 3: Average compositions of the post-experimental slags, R1S1M run**

Expt →	1 min	5 min	10 min
oxide			
F2	3.4	3.3	3.3
Na2O	3.6	2.5	4.5
MgO	-	-	-
Al2O3	21.9	26	33.7
SiO2	31.6	29.1	24.4
ZrO2	2.1	1.3	0.9
CaO	34.9	32.1	27.6
MnO	2.4	2.1	1.4
FeOT	-	-	-

**Table 4: Composition of post-experimental slags in R1S2M and R2S1M runs**

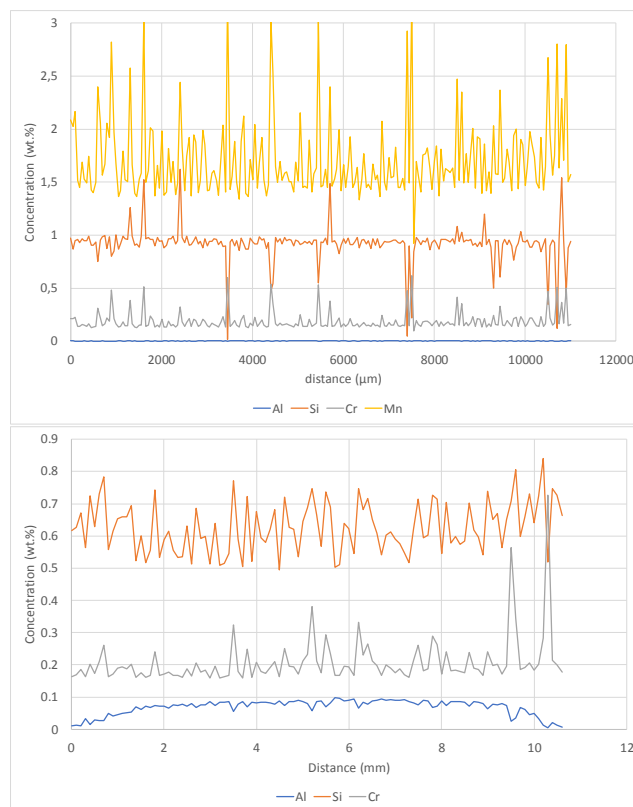
Expt ->	R1S2	R1S2	R2S1
oxide	1 min	10 min	1 min
F2	4.9	8.8	2.2
Na2O	1.1	1.1	9.7
MgO	-	1.1	1.5
Al2O3	13.0	29.5	16.1
SiO2	38.7	30.3	49.3
ZrO2	-	1.6	-
CaO	33.6	27.3	11.7
MnO	0.6	0.3	5.2
FeOT	-	-	-



**Fig 8: Compositional change of the mould slag in the R1S1M series of experiments (after 1, 5, 10 min interaction, blue) and R1S2M experiments (1 and 10 min, red), compared to the three ideal change vectors according to silica reduction by carbon ("red by C"), silica-aluminium exchange with steel ("red by steel") and pure alumina dissolution.**

according to the above exchange reaction, but it is masked on Fig 8 by the simultaneous dissolution of refractory.

The operation of gas-phase mediated reactions between refractory and slag, as well as between refractory and steel, is shown not only by the formation of significant amounts of CO, but also by the appearance of a melt phase within the refractory that approaches the experimental slag in the cup



**Fig. 9: Compositional line profiles across the steel in the experiments R1S1M after 10 min (top) and 1 min (bottom) interaction time. Compare to original reactant composition in Table 2.**

itself in composition. Microstructural examples for this are given in Fig. 10.

In total, as the mass transfers between slag, refractory and steel are complex, a mass balancing can give an indication about the relative mass fluxes involved. For this, use can be made of several conserved elements, which have only one source and/or which do not redistribute between the experimental products. For this, the two best candidates are ZrO2, and CaO. ZrO2 has only the refractory as source, and it is taken up in the slag, but not steel. Thus, under the assumption that the refractory (in its oxidic part) dissolves congruently in the slag, the ZrO2 content of the slag can be used as a measure of the amount of refractory. However, observations within the refractory [4] indicate that there is non-negligible volatility of Zr under carbothermic conditions, as indeed is required by the dispersoid formation of ZrC throughout the refractory. It is therefore possible that an unknown quantity of Zr is lost (along with volatile Na and F). Ca on the other hand has less volatility (no high vapor pressure compounds in the given system), its source is largely only the mould slag itself and it is not taken up in the steel. A balancing for the experiment R1S1M – 10 min is given in Table 5.

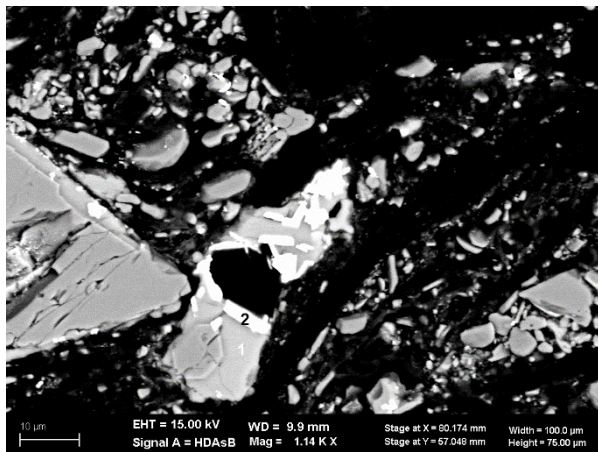


Fig. 10: reprecipitated Alumina, ZrC (white, 2) and a glass phase (1) in the experimental cup of R1S1M-10 min, next to graphite, SiC and and porosity.

## Discussion

The experiments reported here were set up to shed light on the likely processes at the location of slag crawling during casting of liquid steel. The traditional view of the SEN wear mechanism is that the slag-steel-refractory intersection moves dynamically over a region below the lower slag pool edge, allowing mould slag to dissolve the refractory when it wets the SEN, thereby concentrating residual carbon at the surface; and allowing steel to dissolve the carbon when it wets the surface in turn, leaving behind residual refractory oxides that invite wetting by mould slag. Our experiments indicate that this mechanism does not have universal validity. The „submersion depth“ of refractory in our experiments does not exceed 1-2 cm, but within this limit, we find that mould slag completely creeps around the steel, even lifting it from the bottom: surface tension overcomes buoyancy in this depth range. Observations on the real-world SEN describe in the introduction agree with this and indicate that surface wetting driven slag crawling does occur to process relevant depths, leading to contact of the mould slag to the alumina-carbon refractory of the SEN (which is supposed to be prevented by the zirconia-carbon anticorrosion band). In our experiments, the compositions indicate

**Table 5: Mass balance (in grams of elements) for experiment R1S1M, 10 min holding time**

	In	Out	In	Out	In	Out
	Steel		Refractory		Slag	
F	-	-	-	Tr	0.71	0.34
Na	-	-	-	Tr	0.8	0.34
Al	0.32	0	45.45	44.4	0.154	1.81
Si	0.05	0.4	Tr	Tr	1.34	1.16
Ca	-	-			2.02	2.01
Mn	0.91	0.75			0.01	0.11
Fe	43.8	42.2				
Zr	0	0	6.71	6.55	0	0.14

Tr = traces. In = Before experiment, Out = After.

that the slag film intercalated between refractory and steel covers the refractory completely, and direct contact between steel and refractory does not occur. The slag film reacts at both sides, and material transfer between steel and refractory takes place indirectly through the slag. The carbon consumption is not direct dissolution in metal, but is mediated through the formation of gaseous CO. The CO forming reactions are both refractory-internal carbothermic self-equilibration reactions, but also silica reduction of the mouldslag by carbon at the refractory interface. The produced CO encounters the steel and this is the contact that eventually transfers carbon to steel, in exchange for the fixation of Al and other alloy elements at the steel interface, to be incorporated by the mould slag. A major finding of our experiments is that the mould slag can assimilate very large quantities of refractory, without becoming saturated with a solid phase at the casting temperatures. This gives the slag which forms from original infiltrating mould slag large mobility across the submerged faces of the SEN. The result is further explored in a thermodynamic analysis of the reaction encountered, presented on this conference [4]. The fact that the slag intercalated between refractory and steel remains far above break temperature even after substantial dissolution of refractory, is what appears to allow the occurrence of the slag crawling in the cases described in the introduction.

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