DEVELOPMENT OF MOULD FLUX FOR HIGH SPEED THIN SLAB CASTING

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ABSTRACT

Mould powders impact the stability of the continuous casting process of steel. The main functions of mould powder are to provide sufficient lubrication and to control the mould heat transfer in the horizontal direction. Actual casting speeds for the thin slab caster at Corus IJmuiden are between 5.4 and 6 m/min; the production is around the design level of 1.3 Mt/year as coils. It has been decided to increase the production to 1.8 Mt/year as coils. In order to meet this demand, the steel in mould time has to be increased to approximately 85% and the maximum casting speed to 8 m/min.

A collaborative project between Sumitomo Metal Industries (SMI) and Corus IJmuiden was initiated to develop mould powders which facilitate casting speeds up to 8 m/min. Main subjects of this project are: powder design, characterisation of mould powder and mould slag, trials at the Sumitomo pilot caster and plant trials at the Corus thin slab caster.

Mould powder design was initially based on Sumitomo’s experience with high-speed casting (QSP-Process). Characterisation was done using advanced methods like high-temperature X-ray diffraction, several microscopic techniques as well as viscosity measurements which include the determination of the slag solidification temperature. A special condition is to use mould powder as a granulated material. As a consequence, the work focussed on the choice of raw materials and on the corresponding phase relations at elevated temperatures.

Typical of the developed mould powders are so-called mild cooling properties which will result in a controlled mould heat transfer during casting.
INTRODUCTION

Mould powders impact the stability of the continuous casting process for steel at all casting speeds. The main functions of mould powder are to provide sufficient lubrication and to control the mould heat transfer between the solidifying steel shell and the copper mould [1]. At higher casting speeds associated with thin slab casting, the role of the mould powder is even more important.

The thin slab caster at Corus IJmuiden (Direct Sheet Plant, DSP) started production in 2000. The caster has one strand and is equipped with a funnel shaped mould (mould dimensions 90 * 1000 - 1560 mm), a specially designed Submerged Entry Nozzle (SEN) and an adjustable multiple pole ElectroMagnetic Brake (EMBr). Liquid core reduction decreases the slab thickness from 90 mm to 70 mm. The designed production level is 1.3 Mt/year (coils), the maximum casting speed is 6.0 m/min. The current production is around the design level with actual casting speeds between 5.4 and 6 m/min. It has been decided to increase the production of this caster to a level of 1.8 Mt/year (coils) with one caster strand. In order to meet this demand, the steel in mould time has to be increased to approximately 85% and the maximum casting speed will be increased to 8 m/min.

A collaborative project between Sumitomo Metal Industries (SMI) and Corus IJmuiden was initiated to develop mould powders which facilitate casting speeds up to 8 m/min. Main subjects of this project are: mould powder design (chemical and mineralogical composition, physical properties), characterisation of mould powder and mould slag, trials at the Sumitomo pilot caster and full scale plant trials at the Corus thin slab caster. Evaluation of the trials will be based on the in-mould behaviour of the mould powder with respect to slag formation, mould heat transfer and strand lubrication followed by inspection of the slab surface. A special point of attention is the condition to use mould powder as a granulated material at the thin slab caster of Corus. As a consequence, the characterisation work focuses on the choice of raw materials and on the corresponding specific phase relations at elevated temperatures i.e., during heating of mould powder and cooling of mould slag.

MOULD POWDER DESIGN

Mould powder design for this project was initially based on Sumitomo’s experience with high-speed casting in the Quality Strip Production Process (QSP Process). Sumitomo developed this casting process (mould dimensions 90 * 1000 mm) as well as suitable mould powders focussing on low carbon steel grades with a maximum casting speed of 8 m/min and peritectic steelgrades with a casting speed up to 5 m/min [2, 3]. Based on the experiences for low carbon steel grades, Sumitomo selected a mould powder for first trials at the pilot caster and at the thin slab caster. It is common for the casters at Corus IJmuiden to use mould powder as a granulated material; this in contrast with the practice at Sumitomo. So, the developed mould powders had to be adapted. Based on the same chemical composition, the raw material choice of the original powder was partly changed in order to manufacture a granulated mould powder.

A summary of the chemical composition of the mould powders is given in Table 1. Powder A is the initial mould powder, selected by Sumitomo and Powder B is the granulated version of this material. The granulation was done by the Japanese mould powder supplier of Sumitomo. For reasons of logistics however, it was decided to granulate (and manufacture) future mould powders by a European supplier, based on the composition as formulated by Sumitomo and Corus. Mould powder X is the standard (granulated) powder, used at the thin slab caster of Corus [4]. This powder can be considered as a reference
material. Mould powders B and X have been tested at the pilot caster of Sumitomo. A next step was a full scale plant trial of powder B at the thin slab caster of Corus.

Table 1: Chemical composition (wt%) of mould powders, as measured at Corus IJmuiden

<table>
<thead>
<tr>
<th>Component</th>
<th>Mould powder type</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A</td>
</tr>
<tr>
<td>CaO/SiO₂</td>
<td>1.21</td>
</tr>
<tr>
<td>MgO + Al₂O₃</td>
<td>11.7</td>
</tr>
<tr>
<td>Na₂O + F</td>
<td>17.0</td>
</tr>
</tbody>
</table>

* Mould powder as a granulated material

Based on the casting experiences with mould powder B and X, the design of mould powders for high-speed casting was adapted. The criteria are given in Table 2, Figure 1 and Table 3. The experiences are described in more detail below.

Table 2: Mould powder design for high-speed casting

<table>
<thead>
<tr>
<th>Basicity (CaO/SiO₂)</th>
<th>1.2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solidification point or Tbreak (°C)</td>
<td>1150</td>
</tr>
<tr>
<td>Crystallisation of mould slag</td>
<td>cuspidine</td>
</tr>
</tbody>
</table>

Figure 1: Mould powder for high-speed casting – crystallisation of cuspidine

The mould powder is designed for a basicity (CaO/SiO₂) of 1.2, a solidification point or Tbreak of 1150°C (to be obtained during viscosity measurements) and a preferred crystallisation (mould slag) of cuspidine (3CaO·2SiO₂·CaF₂ or Ca₄Si₂O₇F₂) [5].

These criteria will result in mild cooling properties, aiming to control the heat transfer between the solidifying steel shell and the mould copper plates (horizontal heat transfer). Mild cooling properties are required in order to prevent the formation of slab surface cracks (especially longitudinal surface cracks) during casting and to protect the mould copper plates with respect to the increased heat load, resulting from high casting speeds [6]. The corresponding chemical composition as designed is given in Table 3.
Mould powders C and D (Table 1) are based on these criteria. Mould powder C is a powder, as formulated by SMI. Mould powder D is a granulate with an almost identical chemical composition. The granulation (and production) of this material was done by a European mould powder supplier.

Mould powders C and D have been tested at the pilot caster of Sumitomo, followed by a plant trial at the thin slab caster of Corus with mould powder D. Based on these test, next steps will be defined.

**MOULD POWDER CHARACTERISATION AND PHASE RELATIONS**

**Characterisation**

The chemical composition in Table 1 does not specify the mineralogical composition of the chosen raw materials. Room-temperature X-ray diffraction (XRD) was used to determine the mineralogical composition i.e. the raw material choice of the mould powders. In general, the mould powders are composed of silicates, fluorites and carbonates. Additionally, amorphous components (glass) can be present as well as smaller amounts of raw materials (accessory). Each mould powder can show significant differences in raw material composition.

The two powders A and B are characterised by the use of Portland Cement as a main raw material (silicates) and by a significant amount of periclase (MgO). Furthermore, mould powder A contains two other sources of silica: quartz and cristobalite (SiO₂). Powder A also uses natrite (Na₂CO₃), whereas mould powder B contains a considerable amount of amorphous material (glass).

Main differences between mould powders C and D are the use of Portland Cement and quartz (SiO₂) in mould powder C. Mould powder D uses wollastonite (CaSiO₃) as a major raw material with a minor amount of quartz. Furthermore, mould powder C contains periclase (MgO) as a magnesium-source and mould powder D forsterite (Mg₂SiO₄).

The standard mould powder X corresponds roughly to mould powder D. Some exceptions are the use of albite (NaAlSi₃O₈) as major raw material in powder X and the use of forsterite (Mg₂SiO₄) and corundum (Al₂O₃) in powder D.

All the mould powders use fluorite (CaF₂) as source of fluorine.

Based on the mould powder characterisation and on supplier information, mould powders A, B and C use two carbon sources (free carbon) in order to control the melting rate during casting. Mould powders D and X only contain one source of free carbon [7].

The viscosity of the mould powders and the corresponding solidification point or Tbreak were measured. Results obtained at Sumitomo are summarised in Table 4 and are based on an oscillating plate method with a cooling rate of 2°C/min. Note that powder D does not show the required solidification point of 1150°C, due to the deviations in sodium and fluorine.

<table>
<thead>
<tr>
<th>CaO/SiO₂</th>
<th>MgO + Al₂O₃</th>
<th>Na₂O + F</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.2</td>
<td>8.5</td>
<td>20</td>
</tr>
</tbody>
</table>
Table 4: Viscosity and solidification point, as measured at Sumitomo

<table>
<thead>
<tr>
<th></th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>X</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity at 1300°C (Pa.s)</td>
<td>0.09</td>
<td>0.08</td>
<td>0.09</td>
<td>0.08</td>
<td>0.08</td>
</tr>
<tr>
<td>Solidification point (T&lt;sub&gt;break&lt;/sub&gt;) (°C)</td>
<td>1101</td>
<td>1115</td>
<td>1161</td>
<td>1187</td>
<td>1134</td>
</tr>
</tbody>
</table>

Phase Relations

Mixtures of raw-materials of mould powders are not in chemical equilibrium but the systems tend towards it during heating, which results in a series of reactions between the phases and eventually formation of secondary phases. These reactions may include solid-state reactions or partial melting and solid-liquid interaction. The mineralogical composition will change continuously during heating until the mould powder melts completely. Subsequently, during cooling and solidification the composition will also change. The sequences of reactions are specific for a mould powder and are controlled by the mineralogical and chemical bulk composition.

The mineralogy of the major phases for the powders has been observed in situ using high-temperature X-ray diffraction (HT-XRD) with additional powder X-ray diffraction and microscopic techniques. XRD-patterns were collected in the 2-Theta range from 20 to 60 degrees, while the temperature was raised from 500°C to 1350°C and subsequently lowered to 500°C. Heating and cooling rates were 200°C/min. XRD-patterns were taken in temperature increments of 50°C. A second sequence of XRD-pattern was collected by heating with an offset of 25°C compared to the first run to obtain a higher resolution of phase stabilities. The crystallisation path was investigated additionally by heating directly at 300°C/min to 1250°C, followed by cooling to 500 °C and by using a slower scan rate. Phase relations and temperature stabilities were derived by combining all three data sets.

The XRD-patterns can be summarised as a 3D-plot, see Figure 2. The data on powder melting (heating) gives information on the processes leading to slag formation and the formation of rims. Results on slag solidification (cooling) are essential for a better understanding of slag crystallisation and the control of mould heat transfer during casting. A summary of the results of powder B, C and D is given in Figure 3.
During heating, each mould powder shows a specific melting sequence i.e., disappearance of raw materials and appearance of intermediate (secondary) phases, before melting takes place. Melting is complete around a temperature of 1250°C. During cooling, one or more crystalline phases can be formed.

Cooling of powder B showed two major crystalline phases: cuspidine \((\text{Ca}_4\text{Si}_2\text{O}_7\text{F}_2)\) and melilite \((\text{Ca,Na})_2(\text{Al,Mg})(\text{Si,Al})_2\text{O}_7\). Both phases show a strong peak-overlapping, which makes the exact temperature at which melilite becomes visible difficult to determine. Cuspidine crystallises at ~1075-1050°C followed by melilite around 900-850°C. No minor components could be detected.

Mould powder C and D both clearly show crystallisation of cuspidine and Na-Al-Silicate. The start of crystallisation for powder C is around 1250°C (cuspidine) and 975°C (Na-Al-Silicate). For mould powder D, these values are 1200°C and 1150°C respectively.

![Figure 3: Results of HT-XRD analyses and indications of phase stabilities of major phases within powder B, C and D (heating and cooling). Note that these stabilities may be dependent on actual heating and cooling rates and are strictly valid only for the given experimental conditions.](image)

The amounts of crystals in the post-heated samples after HT-measurements were determined by Rietveld-analysis of final room-temperature XRD-patterns. Results are given in Table 5. Note that glass phases are not reflected in the relative amounts.

<table>
<thead>
<tr>
<th>Table 5: Relative amounts (wt%) of crystalline phases in mould slag</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Mould powder type</strong></td>
</tr>
<tr>
<td><strong>Major phases</strong></td>
</tr>
<tr>
<td>Cuspidine ((\text{Ca}_4\text{Si}_2\text{O}_7\text{F}_2))</td>
</tr>
<tr>
<td>Melilite ((\text{Ca,Na})_2(\text{Al,Mg})(\text{Si,Al})_2\text{O}_7)</td>
</tr>
<tr>
<td>Na-Al-Silicates</td>
</tr>
<tr>
<td><strong>Minor phases</strong></td>
</tr>
<tr>
<td>Fluorite ((\text{CaF}_2))</td>
</tr>
<tr>
<td>Monticellite ((\text{CaMgSi}_2\text{O}_6))</td>
</tr>
</tbody>
</table>
TRIALS AT THE PILOT CASTER

The pilot caster of Sumitomo is of a vertical type with a casting length of 3.7 m. The ladle capacity is 2.4 ton. The mould dimensions are 800 * 100 mm, the mould level control is based on an eddy-current system. The caster uses a hydraulic oscillator. The maximum casting speed is 3 m/min.

Caster Data

Trials have been done with mould powders B, C, D and X using a low-carbon steel grade (C ~ 0.05%) at casting speeds up to 2 m/min, the casting time is around 4 minutes. Evaluation of the caster data concentrated on the mould heat transfer including the mould thermocouple temperatures, strand friction, mould level fluctuations, the melting behaviour of the mould powder etc.

All casting trials were successful. Based on the local heat transfer, 35 mm below the meniscus, powders C and D showed a decreased heat transfer and some increase in mould level fluctuations.

A summary of the caster data is given in Table 6. Immediately after a trial, it was tried to collect slag film samples at the meniscus area and under the mould.

Table 6: Local heat flux and mould level fluctuations at the pilot caster

<table>
<thead>
<tr>
<th>Mould powder type</th>
<th>Local heat flux 35 mm below the meniscus (MW/m²)</th>
<th>Mould level fluctuations (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>2.3</td>
<td>1.9</td>
</tr>
<tr>
<td>C</td>
<td>1.7</td>
<td>2.9</td>
</tr>
<tr>
<td>D</td>
<td>1.8</td>
<td>3.0</td>
</tr>
<tr>
<td>X</td>
<td>1.9</td>
<td>2.7</td>
</tr>
</tbody>
</table>

Slag Films

Slag films realise the main mould powder functions strand lubrication and mould heat transfer and can be considered as a key for a further understanding of the mould powder functions and to guide mould powder design [8]. Characterisation of the films was done using SEM/EDS-techniques and concentrated on the structure and the composition. Additionally, XRD was used.

All slag films are broken in the transverse direction showing a flat and sometimes a wavy surface. Most likely the flat surface corresponds to the mould side. An illustration is given in Figure 4.

Figure 4: Slag films obtained after the casting trial (under the mould) – Mould powder C, pilot caster
**Mould Powder B**

Slag films were sampled under the mould. The films show a glass phase, fine crystals (monticellite, melilite and cuspidine) and some unreacted/partly reacted raw materials (periclase, fluorite and Ca₃SiO₅ – haturite, a Portland Cement) – these materials adhere to the surface of the films. Note that monticellite is an intermediate (secondary) phase formed during powder melting, indicating that melting of the mould powder was not completely finished. The film thickness varies between approximately 1 and 2 mm, the thicker part can possibly be seen as the end of a slag rim. In the glass phase, some (very) small droplets of Fe can be seen, probably originating from the cast steel.

**Mould Powder C**

After the trial with powder C, slag films were sampled from the meniscus area (adhering to a slag rim) as well as under the mould. In this study, characterisation of slag films obtained under the mould is described. An illustration of the films is given in Figures 4 and 5. The average thickness of the slag films is 1.5 mm. The films show a glass phase and a crystalline phase. In general, the crystalline phases can be detected in the outside parts of the films (both mould and strand side), the inner parts of the films mainly consist of a glass phase.

Detailed analyses of the slag films showed that the crystalline phases mainly consist of large cuspidine crystals (dendrites), together with fine Na-Al-silicate crystals in a glass matrix. All slag films show bubbles and small Fe-droplets. Furthermore, parts of mould powder raw materials as well as intermediate phases are present, adhering to the surface of the film.

![Figure 5: Detail of slag film, obtained after the casting trial (under the mould) showing glass and crystalline phases – mould powder C, pilot caster](image)

**Mould Powder X**

The films corresponding to mould powder X show one area containing only glass and another area containing cuspidine and glass. The thickness is between 0.4 and 1 mm and again here Fe (droplets) can be observed. Some remnants of powder can be seen adhering to the surface; no intermediate phases have been detected.

In general, results of the slag film analyses confirm results obtained with HT-XRD (cooling experiments).
TRIALS AT THE THIN SLAB CASTER

Evaluation of the mould powder trials at the thin slab caster is based on slag formation (liquid pool depth and rim formation), powder consumption, strand lubrication and mould heat transfer followed by inspection of the slab surface. The desired minimum liquid pool depth during casting is 5 mm. The liquid pool depth is measured by immersing a stainless steel sheet in combination with a copper strip into the mould. The operators observe the formation of rims. The maximum thickness of slag rims is approximately 10 mm; excessive rim formation should be avoided for reasons of process stability [9]. The powder consumption should not be lower than the current average value of 0.05 kg/m² (kg powder/slab surface).

Homogeneous and controlled heat transfer and controlled strand lubrication are desired within the given operational windows of the caster. Mould heat transfer is calculated using mould cooling water temperatures and flow; additional data is obtained via the mould thermocouples. Strand friction is obtained from the hydraulic oscillating system of the mould.

Trials were done with mould powders B and D. During the trials, it was not possible to collect slag films. For this reason, characterisation focussed on slag rims, sampled during trials with powder B.

Caster Data

Mould Powder B

Several plant trials have been done with mould powder B at casting speeds up to 5.0 m/min while casting low carbon steel grades (C ~ 0.045%). The casting speed showed several variations during the trials.

The powder showed good mild cooling properties but significant rim formation during casting. Investigations of the rims indicated sintering behaviour of mould powder B as well as mould level fluctuations, both causing the formation of rims. The sintering behaviour can probably be related to the chemical composition and raw material choice, more specifically the use of MgO and the use of coke particles as one of the free carbon sources. The presence of monticellite - an intermediate crystalline phase - was also observed.

The liquid pool depth was between 3 and 4 mm; the standard powder only showed a thickness of 2 mm at the same casting speed.

The average mould heat transfer during the trial decreased by 5–10%; the friction forces increased by 8% and the torque (withdraw force) also increased. The friction force is also affected by the taper settings. At the same time, the slab surface, measured at the end of the caster increased by approximately 15°C (pyrometer measurements). The trials were stopped because of the rim formation during casting. During and after the trials, several rims have been collected. A more detailed description, based on microscopic analyses is given below; an illustration of a rim is given in Figure 6.
Mould Powder D

First plant trials with mould powder D have been done on a low carbon steel grade (C ~ 0.045%), a mould width of 1250 mm and a casting speed of 5.0 m/min (because of the high solidification point of 1187°C).

The powder shows stable slag formation with values between 4-6 mm (reference 4 mm). No rims were detected. Comparing to the standard mould powder (X), the upper rows of thermocouples of the wide faces show a more stable and decreased mould heat transfer (20-30% lower) whereas the lower part of the mould shows an increased mould heat transfer. The average mould heat transfer of the wide faces (based on mould cooling water data) remained the same for both powders. These findings indicate that the shell thickness is the same for both powders, but that the distribution of mould heat transfer changes in a favourable way. The narrow faces show a decrease of the average mould heat transfer by 20-30%. The friction and mould level fluctuations increased during the trial by approximately 15% and 50% respectively. These values are also affected by the taper settings during the trial. However the operational windows were not exceeded. The powder consumption of powder D was roughly the same showing values of around 0.06 kg/m².

The slab surface temperature, measured at the end of the caster increased again; an increase of 30-40°C was reported (pyrometer measurements). Consequently, the gas consumption in the first segments of the tunnel furnace decreased by approximately 17%. This phenomenon is not fully understood yet. Inspection of the slab surface showed no surface cracks.

The powder clearly showed mild cooling properties, with a reduced mould heat transfer especially at the upper part of the wide faces and a comparable average mould heat transfer. These properties seem to be very favourable for high speed thin slab casting. Trials will be continued with a mould powder according to the specifications as given in Table 2. Special attention will be given to mould heat transfer and the increased values of strand friction and mould level fluctuations. Besides, powder melting and slag consumption will be addressed.

Slag Rims

Mould Powder B

A rim was extracted approximately 45 minutes after changing from the standard powder (X) to powder B. The rim is illustrated in Figure 6.
The presence of cuspidine, melilite and monticellite indicates that the mould slag in the rim predominantly originates from powder B. The slag contains a significant amount of coarse coke particles which is a raw material, used as a source of free carbon. This observation makes the effectiveness of coke during melting of this powder questionable. The powder particles in the rim originate both from powder B and from the previous powder (powder X). This indicates a long residence time of powder X after the mould powder change.

![Figure 7: Detail of slag film, adhering to a rim showing glass (1), the crystals cuspidine (2) and monticellite (3) and some porosities (4) – Mould powder B](image)

A part of a slag film adhering to a slag rim was obtained and analysed. The film matches the composition of powder B. The film consists of glass and of the crystals monticellite and cuspidine; the crystals are mostly present at the mould side. Furthermore, porosities were observed, see Figure 7.

Based on the casting behaviour of powder B, especially rim formation and mould heat transfer, the composition was changed. This resulted in a basicity of 1.2, a solidification point of 1150°C and a preferred crystallisation of cuspidine. See Table 2.

**CONCLUSIONS**

- Mould powder design for high speed thin slab casting concentrates on mild cooling properties, to be realised by specific values of the basicity (1.2) and the solidification point (1150°C), promoting the crystallisation of cuspidine.
- Mild cooling properties will show a reduced mould heat transfer in the upper part of the mould, an increased mould heat transfer in the lower part of the mould while maintaining a comparable average mould heat transfer during casting.
- Characterisation of mould powder, slag rims and slag films, in combination with casting trials proved to be essential for a further understanding of mould powder behaviour and for a proper mould powder design.
ACKNOWLEDGEMENTS

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