

Dedicated to Professor Dr.-Ing. Dieter Neuschütz on the occasion of his 65th birthday

Metallurgical process engineering for castability-improvement in steel with elevated sulfur contents

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Trouble-free casting of Al deoxidized steel heats with high sulfur contents necessitates controlled addition of Ca in secondary metallurgy, in order to avoid the nozzle clogging effect. With too little Ca input on the one hand, clogging occurs as a result of the deposition of high melting point calcium aluminates, and at excessively high Ca inputs, on the other hand, precipitation of calcium sulfide occurs. Process engineering for precisely targeted Ca input is developed on the basis of thermodynamic calculations, general steelplant experience, and comprehensive operational tests. The new method reduces the failure rate from casting problems to less than one quarter. Sequence casting of Al killed grades with high sulfur contents is then no longer a problem.

Metallurgische Prozeßtechnik zur Verbesserung der Vergießbarkeit von Stählen mit erhöhtem Schwefelgehalt. Das störungsfreie Vergießen Al-desoxidierter Stahlschmelzen mit erhöhtem Schwefelgehalt erfordert in der Sekundärmetallurgie eine kontrollierte Ca-Zugabe, um den Effekt des Tauchrohr-Cloggings zu vermeiden. Bei zu geringer Dosierung tritt Clogging durch das Absetzen von hochschmelzenden Kalkaluminaten ein, bei zu hoher Dosierung durch Ausfällung von Calciumsulfid. Aufgrund thermodynamischer Berechnungen, allgemeiner Stahlwerkserfahrung und umfangreicher Betriebsversuche wird eine Verfahrenstechnik zur zielgerechten Ca-Dosierung entwickelt. Die neue Methode verringert die Ausfallrate infolge Gießstörungen auf weniger als ein Viertel. Das Sequenzgießen Al-beruhigter Stahlsorten mit erhöhtem Schwefelgehalt stellt kein Problem mehr dar.

Only steels with excellent oxide cleanliness are suitable for the production of seamless precision tubes. The sulfur contents of these grades are in the 200 to 300 ppm range, in order to improve their machining properties. Special provisions in metallurgical process engineering are necessary to be sure to attain the required properties in the steel with certainty: Controlled and monitored deoxidation of the crude steel heats with Al, alloying with C, Si, Mn, S and, where appropriate, with Cr and V, to narrow composition specifications, Ca treatment in secondary metallurgy, shrouded casting using the argon pressure box method and optimized flow control in the caster's tundish. These provisions are in common use, but in the present case necessitate strict monitoring of operations as a whole.

The basic problem in operating technology can, however, be found in assuring the castability of the heats by means of calcium treatment in secondary metallurgy. If Ca input is too low, the tundish nozzles become blocked due to the deposition of high melting-point calcium aluminates; if it is too high, the same effect occurs as a result of the deposition of calcium sulfide. In both cases, it is generally necessary to abort the cast and return the remaining liquid steel in the ladle to the converter shop.

In the continuous-casting plant, the function of the submerged entry nozzles is monitored by means of the so-called casting scrolls. **Figure 1** shows a typical example: The cast remains normal during casting of the first heat of the sequence. With casting speed unchanged, the mould filling level and stopper opening also remain stably constant. Typical problems soon occur at the start of casting of

the second heat. Casting speed drops as a result of gradual blockage of the submerged entry nozzles, mould filling level becomes unsteady and cannot be brought under adequate control even with the stopper wide open. In this case, the remainder left in the ladle amounted to some 70 t of steel. The depositions in the nozzle consisted of alumina, high-melting-point calcium aluminates and, to a lesser

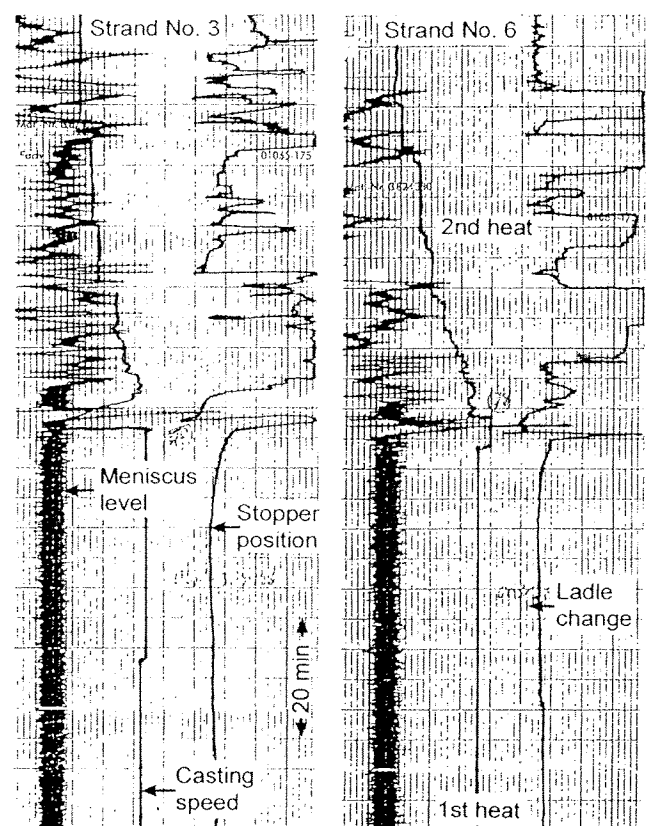


Figure 1. Casting records, strands no. 3 and 6, for a two-heat sequence

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extent, spinel. The cause of the casting problems was therefore too low addition of Ca during the secondary metallurgical treatment of the heat.

The consequences of inadequate Ca adjustment become visible immediately during continuous casting, can no longer be corrected, and therefore result in significant losses of production and disruptions to operation. Since chemical analysis figures are known not to constitute guiding variables, it is therefore necessary to provide process-engineering information in order to ensure precise charging of every heat with calcium. This publication outlines methods to effectively avoid the deposition of oxides or sulfides in the submerged entry nozzles and thus stabilizing production even in sequence casting. The investigations and developments have been performed at Hüttenwerke Krupp Mannesmann GmbH (HKM) in Duisburg. The methodology is therefore oriented around the metallurgical process engineering practiced there.

Secondary metallurgy and casting technology for the production of clean heats

On the basis of standardized operating practice, the 250 t crude steel converter heats are pre-deoxidized at tapping using 400 kg Al drops and pre-alloyed with corresponding additive elements. Secondary metallurgical treatment in the basic-lined steel teeming ladles starts as early as converter tapping on the steel transfer car in the form of bottom bubbling of the teeming ladle with argon at a maximum gas flow of $3 \text{ m}^3 \text{ (STP) min}^{-1}$.

After transfer of the teeming ladle to the secondary metallurgy treatment stand, the level of deoxidation, target composition and target temperature are generally adjusted in two treatment stages with a high bubbling gas flow. The quantities of additives are selected on the basis of the values obtained from chemical analysis of samples of the steel. The upper analysis limits are targeted for Al and S content, while casting temperature is $1550 \text{ }^\circ\text{C}$, with $1535 \text{ }^\circ\text{C}$ as the lower limit. Re-formation of the alumina inclusions remaining in the heat to calcium aluminates is performed at the end of secondary metallurgical treatment by means of mechanical injection of 80 kg of CaSi filled wire. The flow of bubbling gas is reduced during this procedure to $0.1 \text{ m}^3 \text{ (STP)/min}$ and bubbling plug in order to promote the removal of the $\text{Al}_2\text{O}_3/\text{CaO}$ inclusion droplets formed and to avoid any emulsification of the top slag.

The clean heats are cast on two round casters of 5 and 6 strands respectively, in formats of 177 to 406 mm diameter [1; 2]. The casting time may be as much as three hours in the case of small formats. The 30 t tundishes are equipped with Ar flushed pressure boxes to ensure shrouded casting [3; 4]. The walls of these take the form of perforated walls (baffles) in order to improve flow control and removal of inclusions [5]. Uncontrolled slag carry-over from the teeming ladle is avoided by means of an automatic slag detection system [6]. Particular attention is directed to the fact that extremely narrow entry nozzles of only 22 mm internal diameter are used for the casting of clean heats. This provision on the one hand avoids the detrimental

gradient agglomeration of inclusions in the SEN (submerged entry nozzle) [7] and, on the other hand, is an assurance of the required quality in terms of oxide cleanliness [8].

Clean heats with sulfur contents of between 100 and 300 ppm make up around 15 % of HKM's total production. At the start of the metallurgical development work, heat rejection rate due to inadequate castability was 17.7 %. This figure fell to 3.6 % following introduction of the new metallurgical process technology; the main reason for the remaining rejections was excessively low casting temperature [9].

Castability and inclusion types in the steel heat

Oxygen contents in the crude steel after the converter process are between 500 and 1500 ppm. During converter tapping and subsequent ladle treatment, oxygen is fixed as Al_2O_3 by means of the addition of aluminium as a deoxidation agent. The finely dispersed precipitation products are subject to coarsening processes as a result of agglomeration. The cause of this effect can be found in particle collisions caused by Brownian motion, drift in turbulent shear flows, and gravity segregation [10; 11]. Only inclusions of below approximately $20 \text{ }\mu\text{m}$ generally remain in the form of suspended particles in the heat at the end of the secondary metallurgy treatment. During continuous casting, these micro-inclusions accumulate on the inner walls of the SENs and block them (clogging) [12...14]. The most effective method of preventing depositions of alumina in the SENs is treatment of the heat with calcium [15; 16]. Calcium is normally injected into the heat in the form of CaSi powder or in the form of filled wire [17; 18].

The addition of calcium is intended to convert the alumina particles to liquid calcium aluminates. The $\text{CaO}-\text{Al}_2\text{O}_3$ phase diagram is shown for this purpose in figure 2 [19]. Starting from pure alumina, with a melting point of $2053 \text{ }^\circ\text{C}$, the melting point of the inclusions is lowered by means of formation of calcium aluminates. Liquid calcium aluminates occur in the range of CaO mole fractions of 60 to 70 at casting temperatures of between 1500 and 1560

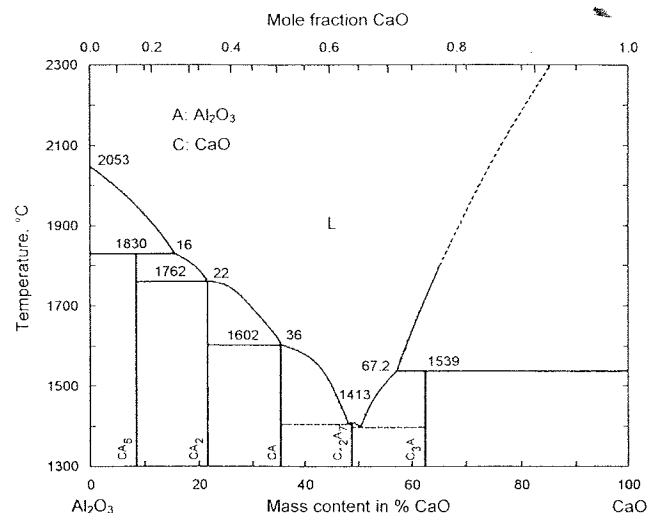


Figure 2. Binary phase diagram $\text{CaO}-\text{Al}_2\text{O}_3$ [19]

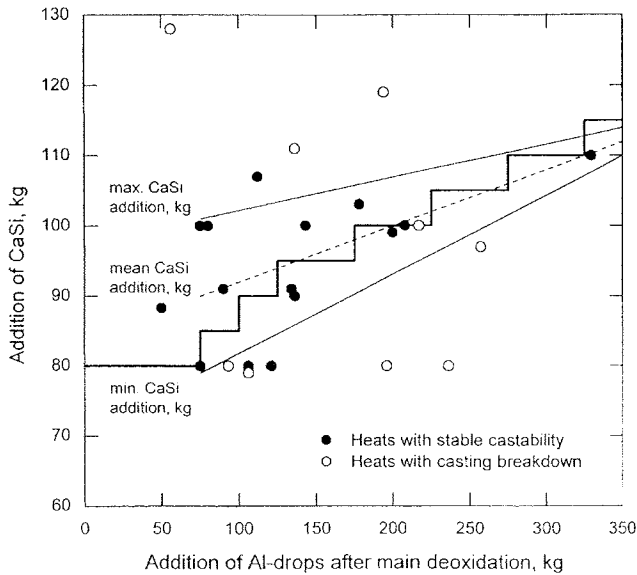


Figure 5. CaSi addition as a function of Al consumption after main deoxidation for heats with elevated S content

The reliably applicable activity plots traceable to Rein and Chipman [25] are used here.

The saturation curves for a range of temperatures for equilibrium between solid CaS and the aluminate with CaO mole fractions of 60 % are shown in figure 4. In accordance with the specification, CaS activity is equal to one along these equilibrium curves. Above the curve, solid CaS is precipitated from the liquid calcium aluminate. Below the curve it remains dissolved in the aluminate. The metallurgical and operational technology freedom of action for high sulfur contents in the heat increases as casting temperature rises and Al contents fall. At a casting temperature of 1550 °C and Al mass contents of 0.02 %, the limit on the basis of these calculations is approximately 200 ppm sulfur. It should be noted that this quantity decreases as the CaO contents of the calcium aluminate increase [9].

Development of a working procedure for addition of CaSi

The results of the thermodynamic calculations in the previous section are highly informative with respect to the potentials and limitations of Ca treatment of Al-deoxidized heats. However, they do not solve the operational problem of what information should be used for input of Ca in order to produce low-melting-point calcium aluminates. The following working hypothesis has proven on the basis of the metallurgical process engineering at HKM to be effective for the solution of this problem: During the main deoxidation process with a constant 400 kg Al at converter tapping, the alumina precipitated is largely deposited in the ladle slag as a result of the high pouring turbulence. This is not true to the same extent of the subsequent deoxidation cycle at the secondary metallurgy treatment stage. Due to the significantly lower level of energy dissipation in the ladle bubbling process, significant quantities of Al₂O₃ remain in the heat in the form of suspended particles [25]. In accordance with the working hypothesis, the quantity of

Al₂O₃ at the end of secondary metallurgical treatment is proportionate to the quantity of Al added to the heat for adjustment of target composition. Input of Ca should therefore be performed on the basis of Al consumption in secondary metallurgy.

The data records of a large number of heats were evaluated on the basis of this concept and elaborated into an operating procedure. The result is shown in figure 5. Heats with a trouble-free cast are shown in the form of solid circles and heats with disrupted casts as a hollow circle. The information leads directly to a working procedure with an adequate tolerance width: Starting from a minimum quantity of 80 kg CaSi, the input amount increases step by step as Al consumption rises, to reach a value of 115 kg CaSi at 350 kg Al. High-melting-point calcium aluminates form below the minimum input, calcium sulfide above the maximum input.

The effects of Ca treatment in accordance with this procedure were apparent, for example, in casting of a two-ladle sequence. 330 kg of Al were consumed for the first heat in secondary metallurgy, and 200 kg Al in the second heat. In accordance with the operating procedure in figure 5, addition of CaSi was 110 and 100 kg, respectively. The casting scrolls shown in figure 6 document the trouble-free casting of this sequence.

Further factors influencing the casting behaviour of heats

The new method of controlled addition of CaSi to improve the castability of heats with high sulfur contents has

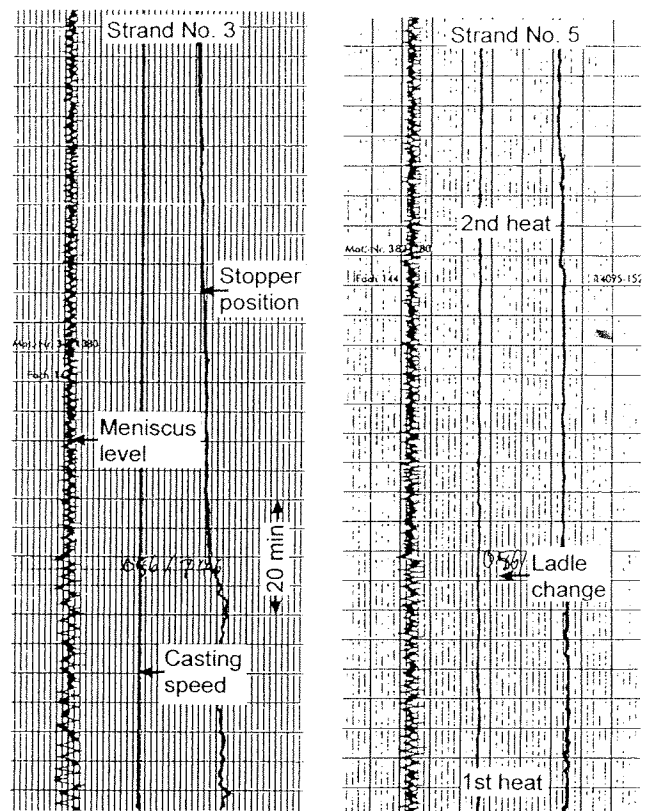
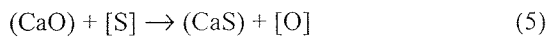


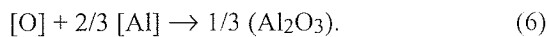
Figure 6. Casting records, strands no. 3 and 5, for a two-heat sequence

proven its value extremely well in most operational practices. Despite adherence to the operating procedure, individual cases involving serious disruptions to casting, the cause of which could not initially be explained, nonetheless occurred. The submerged entry nozzles became completely blocked during casting of a single heat, for instance, with the result that it was not possible to cast 48 t of steel. The depositions consisted of the Al_2O_3 , $CaO \cdot 2Al_2O_3$, $CaO \cdot 6 Al_2O_3$ and $MgO \cdot Al_2O_3$ phases. Input of Ca in accordance with the procedure was obviously not adequate in this case.

The results of regular temperature measurements of this heat and those of the chemical analysis of the samples taken are summarized in **table 1**. All the data are within the normal range, but the pronounced desulfurization of the heat during secondary metallurgical treatment under a comparatively high-calcium top slag is conspicuous in this case and also in all similar cases. The desulfurization reaction in accordance with



supplies the heat with oxygen, which, with aluminium, is precipitated in the form of alumina, in the follow-up reaction



Desulfurization results in continuous and probably high enrichment of the heat with alumina [26], for the reformation of which the planned quantity of CaSi is obviously not sufficient.

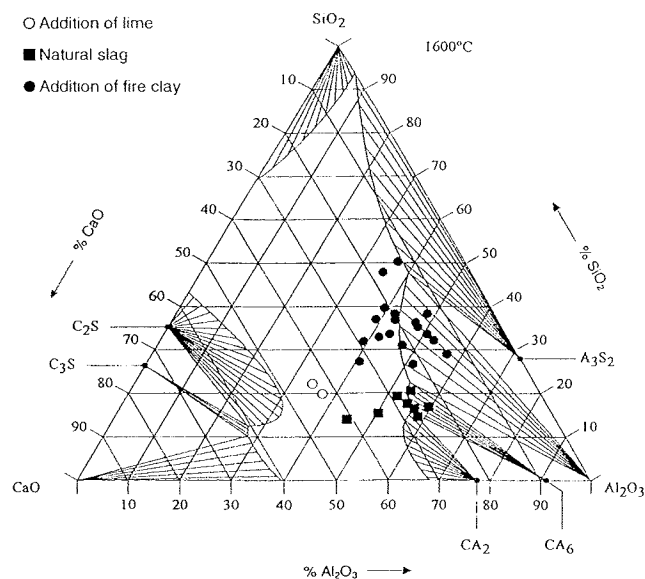


Figure 7. Slag composition in the CaO - CaS - Al_2O_3 ternary phase diagram at $1600^\circ C$ for addition of various slag components during tapping

Table 1. Characteristic casting and treatment data for a single heat

| Temperature, °C | converter measurement | | | | | | | | |
|---------------------------------|-------------------------|------|------|--------|--------|------|------|-------|--------|
| | 1722 | | | | | | | | |
| | 1st ladle measurement | | | | | | | | |
| | 1678 | | | | | | | | |
| | 2nd ladle measurement | | | | | | | | |
| | 1618 | | | | | | | | |
| | 3rd ladle measurement | | | | | | | | |
| | 1604 | | | | | | | | |
| | 4th ladle measurement | | | | | | | | |
| | 1585 | | | | | | | | |
| | 1st tundish measurement | | | | | | | | |
| | 1558 | | | | | | | | |
| | 2nd tundish measurement | | | | | | | | |
| | 1555 | | | | | | | | |
| | 3rd tundish measurement | | | | | | | | |
| | 1551 | | | | | | | | |
| | 4th tundish measurement | | | | | | | | |
| | 1548 | | | | | | | | |
| composition, mass contents in % | C | Si | Mn | S | Al | Cr | V | Nb | Ca |
| converter sample | 0.017 | 0.00 | 0.08 | 0.0502 | --- | 0.03 | 0.00 | 0.002 | --- |
| 1st ladle sample | 0.157 | 0.19 | 1.51 | 0.0394 | 0.0110 | 0.05 | 0.12 | 0.030 | 0.0004 |
| 2nd ladle sample | 0.190 | 0.27 | 1.52 | 0.0296 | 0.0100 | 0.05 | 0.14 | 0.030 | 0.0004 |
| 3rd ladle sample | 0.200 | 0.33 | 1.53 | 0.0267 | 0.0200 | 0.05 | 0.16 | 0.031 | 0.0031 |
| 1st tundish sample | 0.200 | 0.31 | 1.52 | 0.0268 | 0.0180 | 0.05 | 0.15 | 0.030 | 0.0013 |
| 2nd tundish sample | 0.198 | 0.31 | 1.53 | 0.0277 | 0.0180 | 0.05 | 0.15 | 0.030 | 0.0014 |

The experience and discoveries discussed above resulted in 800 kg of granulated fireclay (mass contents of 60 % SiO_2 , 32 % Al_2O_3) being added to the ladle slags in a second process modification. As can be seen from **figure 7**, the SiO_2 mass content of the slag rises to around 35 % as a result of this change. The mode of operation using granulated fireclay as a slag component agent produced stable sulfur contents in the heat during secondary metallurgical treatment and, as a consequence, a further improvement in castability.

In heats with elevated CaSi additions, casting temperatures below $1550^\circ C$ have a particularly disadvantageous effect. This situation is illustrated in **figure 8**. The first

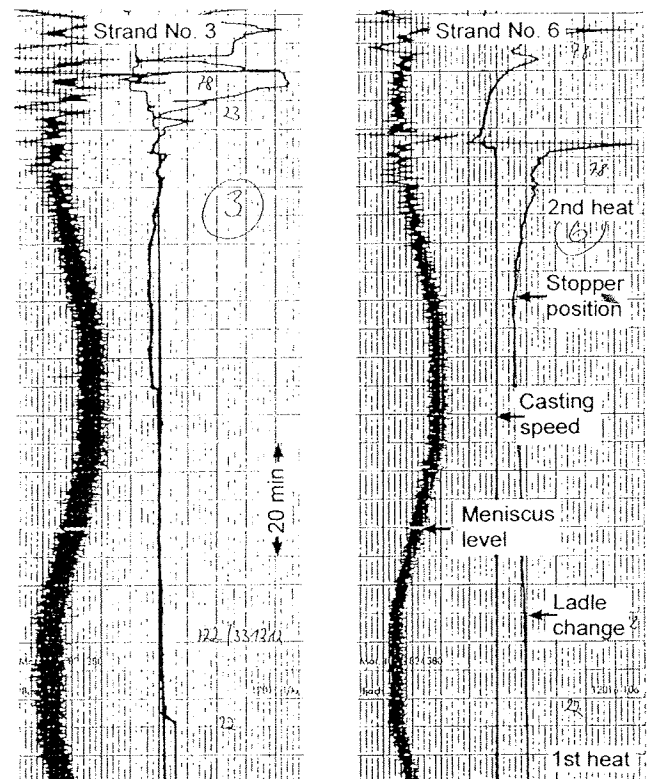


Figure 8. Casting records, strands no. 3 and 6, for a two-heat sequence

heat in the sequence exhibited a normal trouble-free cast. During the second heat, the SENs became increasingly clogged, with the result that a 20 t remainder of steel could not be cast. A tundish nozzle was recovered after the end of casting and the shell of depositions examined under a scanning electron microscope. **Figure 9** shows on the left the structure of a fracture in the deposition shell and, on the right, the results of energy-dispersive X-ray spectral analysis (EDS). These investigations document the fact that the rounded particles consist of CaS, the deposition mass of calcium aluminates. The disruption to casting can therefore be attributed to the precipitation of calcium sulfide. It can be seen from the heat data in **table 2** that the casting temperatures after half casting time for the second charge had dropped below the 1550 °C limit and were, ultimately, only 1528 °C. Otherwise, both heats exhibit sulfur and aluminium contents close to the upper specification limit. Interestingly enough, the excess input of just on 40 kg CaSi for the second heat is scarcely noticeable in the figures for the Ca contents analyzed. This information is therefore not useful for control of secondary metallurgy process engineering.

The third process modification was implemented as a result of this experience: The lower casting temperature limit was raised to 1550 °C and the target analysis was made the

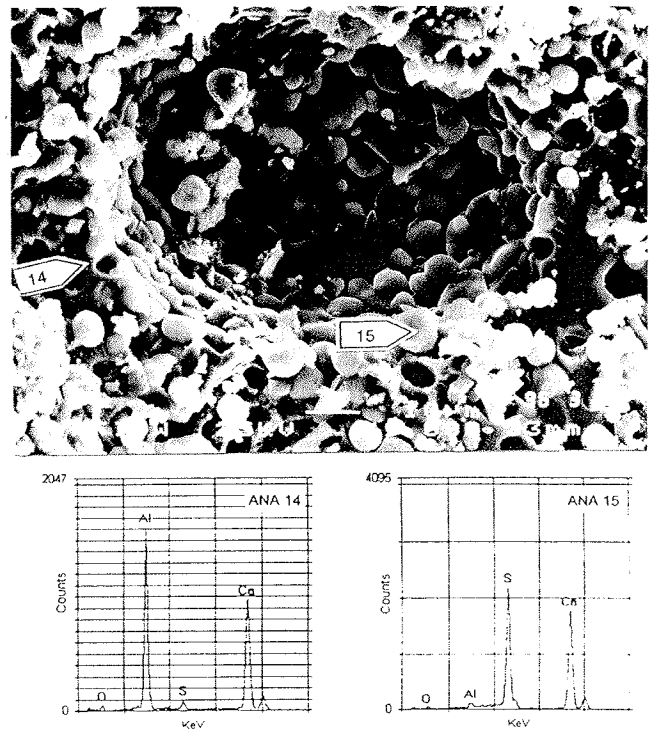


Figure 9. Morphology and chemical composition of deposits in the SEN

Table 2. Characteristic casting and treatment data for a two-heat: sequence

| Temperature, °C | | converter measurement | | 1st heat | | 2nd heat | | | | |
|---------------------------------|--------------------|--------------------------|-----------------------|----------|--------|----------|------|------|-------|--------|
| | | 1st ladle measurement | 2nd ladle measurement | 1647 | 1617 | 1724 | 1715 | | | |
| | | 3rd ladle measurement | 1583 | 1608 | | | | | | |
| | | 4th ladle measurement | 1585 | 1586 | | | | | | |
| | | 5th ladle measurement | 1582 | 1583 | | | | | | |
| | | 1st tundish measurement | 1546 | 1548 | | | | | | |
| | | 2nd tundish measurement | 1541 | 1554 | | | | | | |
| | | 3rd tundish measurement | 1552 | 1554 | | | | | | |
| | | 4th tundish measurement | 1552 | 1555 | | | | | | |
| | | 5th tundish measurement | 1555 | 1552 | | | | | | |
| | | 6th tundish measurement | 1551 | 1545 | | | | | | |
| | | 7th tundish measurement | --- | 1538 | | | | | | |
| | | 8th tundish measurement | --- | 1530 | | | | | | |
| | | 9th tundish measurement | --- | 1528 | | | | | | |
| | | 10th tundish measurement | --- | 1530 | | | | | | |
| composition, mass contents in % | | C | Si | Mn | S | Al | Cr | V | Nb | Ca |
| 1st heat | converter sample | 0.017 | 0.00 | 0.13 | 0.0080 | --- | 0.03 | 0.00 | 0.002 | --- |
| | 1st ladle sample | 0.153 | 0.25 | 1.32 | 0.0247 | 0.0450 | 0.04 | 0.12 | 0.035 | 0.0004 |
| | 2nd ladle sample | 0.173 | 0.26 | 1.49 | 0.0219 | 0.0300 | 0.04 | 0.13 | 0.034 | 0.0004 |
| | 3rd ladle sample | 0.202 | 0.27 | 1.53 | 0.0305 | 0.0310 | 0.04 | 0.16 | 0.036 | 0.0004 |
| | 4th ladle sample | 0.201 | 0.29 | 1.53 | 0.0308 | 0.0320 | 0.04 | 0.16 | 0.036 | 0.0030 |
| | tundish sample | 0.205 | 0.30 | 1.53 | 0.0295 | 0.0290 | 0.04 | 0.16 | 0.036 | 0.0015 |
| 2nd heat | converter sample | 0.020 | 0.00 | 0.14 | 0.0184 | --- | 0.05 | 0.00 | 0.002 | --- |
| | 1st ladle sample | 0.151 | 0.24 | 1.34 | 0.0162 | 0.0190 | 0.06 | 0.13 | 0.031 | 0.0004 |
| | 2nd ladle sample | 0.186 | 0.26 | 1.50 | 0.0267 | 0.0340 | 0.07 | 0.14 | 0.032 | 0.0004 |
| | 3rd ladle sample | 0.202 | 0.28 | 1.53 | 0.0244 | 0.0320 | 0.07 | 0.15 | 0.032 | 0.0045 |
| | 4th ladle sample | 0.197 | 0.28 | 1.53 | 0.0264 | 0.0320 | 0.07 | 0.15 | 0.032 | 0.0036 |
| | 1st tundish sample | 0.208 | 0.29 | 1.55 | 0.0252 | 0.0290 | 0.07 | 0.16 | 0.033 | 0.0021 |
| | 2nd tundish sample | 0.220 | 0.29 | 1.55 | 0.0265 | 0.0270 | 0.07 | 0.16 | 0.034 | 0.0016 |

lower limit for [S] and [Al] contents. These provisions accord entirely with the results of the thermodynamic calculations from figure 4.

Figure 10 summarizes the overall result of this metallurgical development work: It was possible to lower rejections caused by casting problems with steel heats with elevated sulfur contents from the original 17.7 to 3.6 %. Some residual improvements remain possible by means of strict adherence to the raised casting temperature.

Conclusions

Comprehensive metallurgical developments have been performed at HKM for the improvement of the castability of Al-deoxidized steel heats with high sulfur contents. The operating problem consists of controlling calcium charging of the heats in such a way that the alumina inclusions are liquefied without exceeding the failure limit for calcium sulfide.

Thermodynamic calculations, special tests and operating experi-

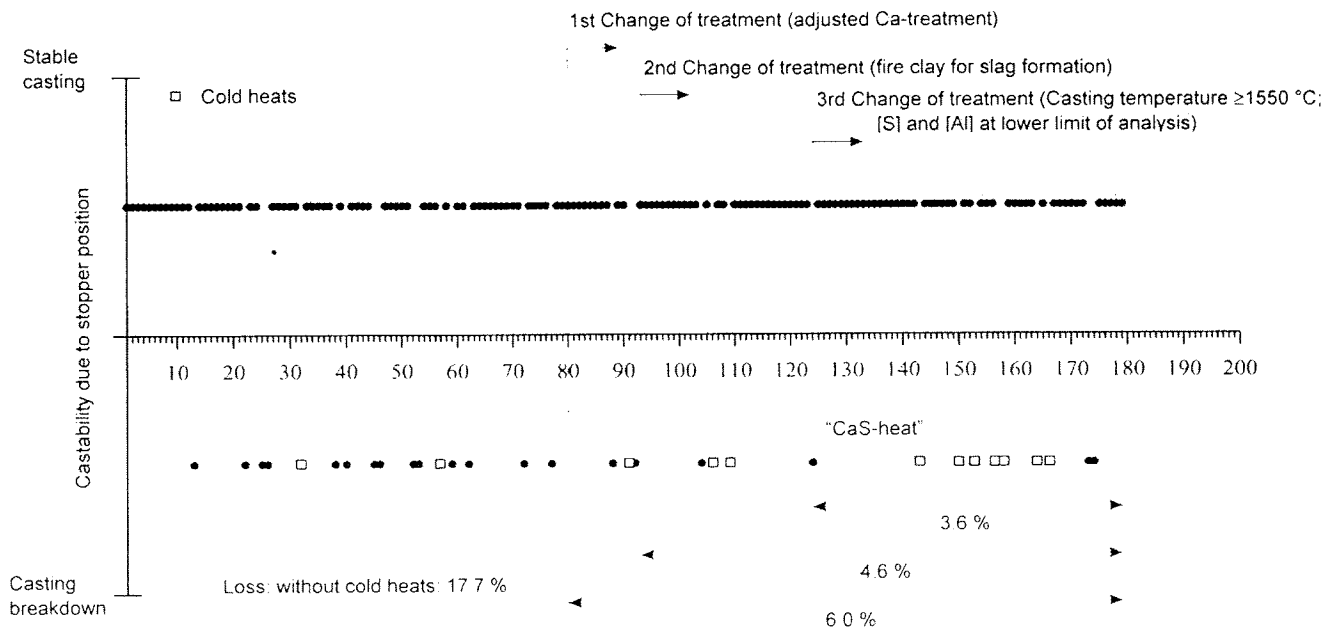


Figure 10. Development of the castability of clean steel heats

ence resulted in a new process concept which reduced the failure rate caused by casting problems by more than a factor of 4. Sequence casting of such grades no longer presents a problem.

The new process concept involves:

- input of CaSi in accordance with Al consumption during secondary metallurgical treatment;
- lowering of the ladle slag's desulfurization capacity via the addition of SiO₂-sources;
- adjustment of casting temperature to 1550 °C or higher;
- limitation of sulfur and aluminium contents to the values of the lower target analysis.

The heats which were cast without difficulties in all cases exhibited good oxide cleanness.

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