Valorisation and Dissemination of Secondary Metallurgy Technology
DissTec

Measurements and models
for on-line control of inclusions in ladle

Filippo Cirilli
Michele De Santis
Antonello Di Donato
Patrizia Miceli

Contents

Scope of the presentation

In the frame of research projects on secondary metallurgy technology a big effort has been addressed to the development of measurements, analysis and models for improving steel quality.

As a consequence of this effort today the secondary steelmaking operations can be accurately designed.

The availability of reliable and powerful techniques for collecting and elaborating information allows on-line and real time control of the operations in ladle.

This presentation shows examples of real industrial applications implemented thanks to the knowledge achieved and the techniques developed in European researches.

The presentation also intends to indicate an outlook to the future developments, toward a continuous monitoring and automated control.

Measurements in ladle

The composition of steel and slag during secondary metallurgy operations in ladle are very crucial factors for reaching the desired final steel quality, in terms of dissolved elements and non-metallic inclusions.

In routine industrial operations steel and slag samples are regularly taken for accurate chemical analysis based on atomic absorption spectrometry, inductively coupled plasma-optical emission spectrometry and x-rays diffraction.

Disadvantages of these techniques are their limit to laboratory use and the necessary sample preparation.

Measurements in ladle

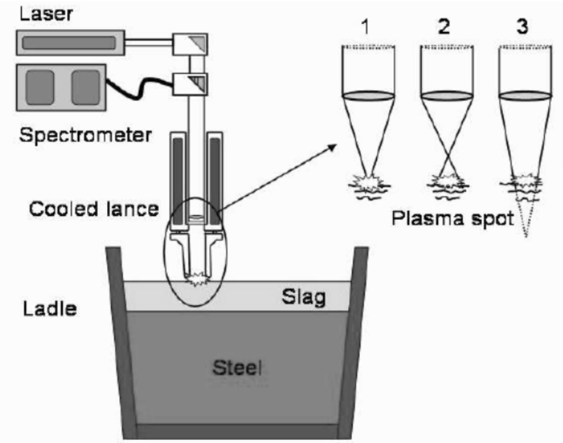
Important objectives of RFCS research have always been to increase the number and quality of data characterizing steel and slag and their availability in short time during the process, empowering the classical routine measurements, such as temperature, dissolved oxygen concentration, steel and slag composition from samples.

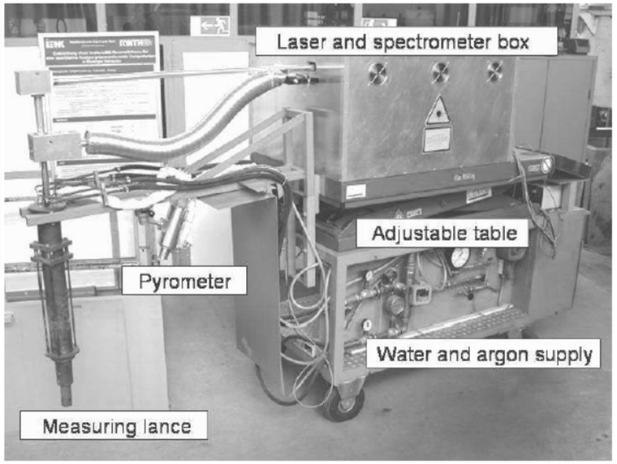
Activities carried out mainly regarded:

1) The development of new techniques for more complete and more rapid analysis of chemical composition of steel and slag
2) The improvement of sampling and analytical technique for more accurate inclusion analysis

Measurements in ladle

Laser based techniques, such as Laser-Induced-Breakdown-Spectroscopy (LIBS), have been investigated for rapid on-line determination of steel and slag composition [1]



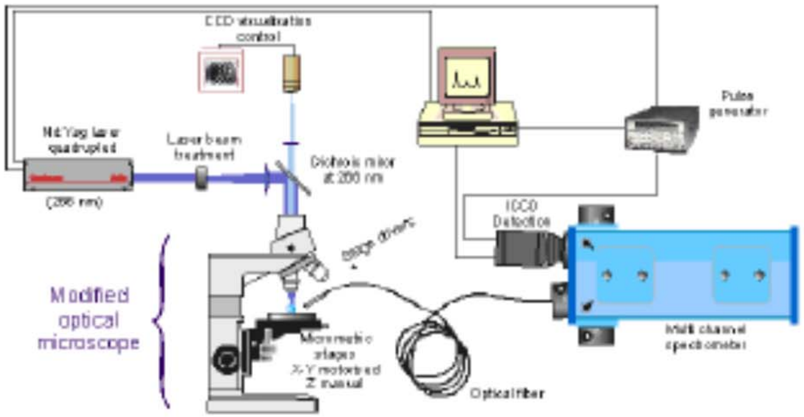


LIBS measurements of slag in ladle
Laboratory testing system and application in real ladle

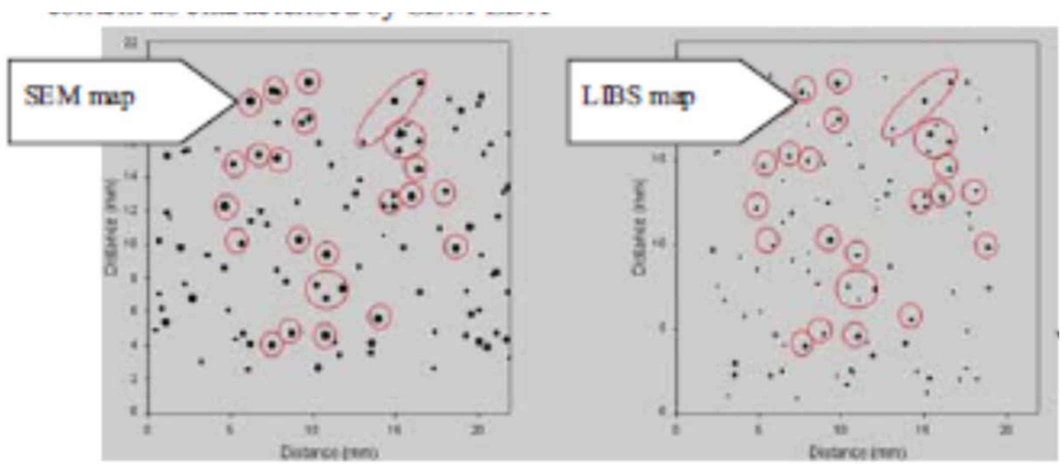
[1] In-situ quick sensing system for measuring of critical components in steelmaking slags (inquiss) - EUR 221018, 2007

Measurements in ladle

LIBS techniques have been also developed for rapid determination of inclusion population [2].



Schematic of a microprobe LIBS system for rapid inclusion analysis

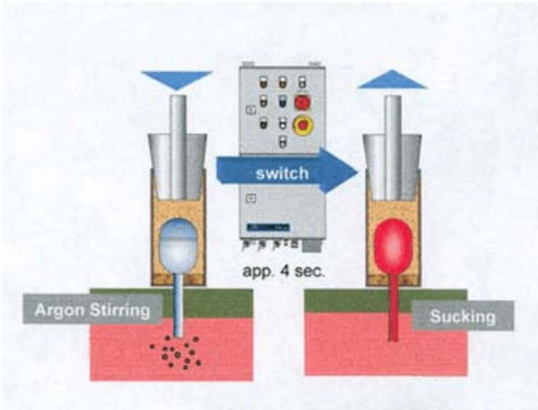


Comparison of SEM and LIBS maps of inclusions

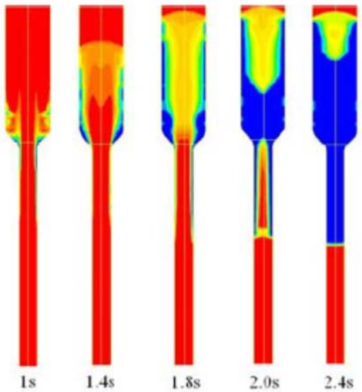
[2] Development of inclusion reference materials and simultaneous determination of metals and non-metallic inclusions by rapid LIBS analysis in steel samples – EUR 24190 2010

Measurements in ladle

To get reliable information optimization of liquid steel sampling methods for inclusion analysis have been studied [4].



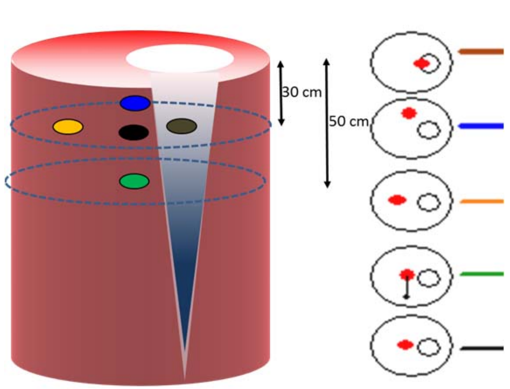
In this project different sampling techniques based on argon injection was experimented, using different shapes of samples, and solidification simulations were performed to define the best operating procedures to avoid crack, hole and inhomogeneity of the sample.

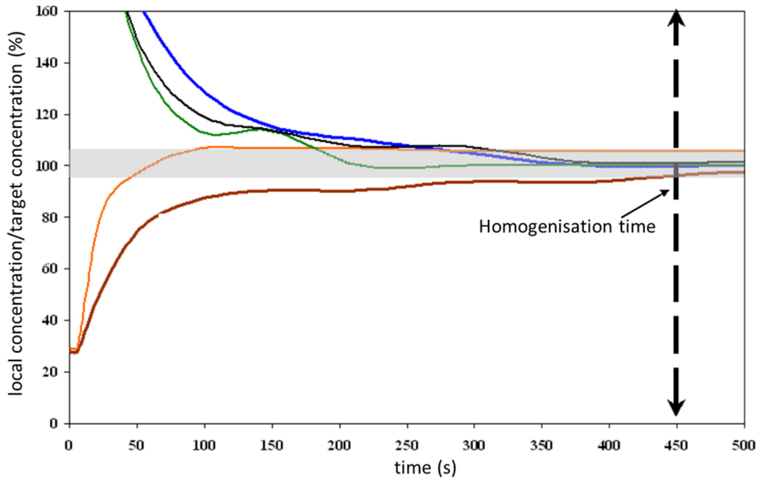


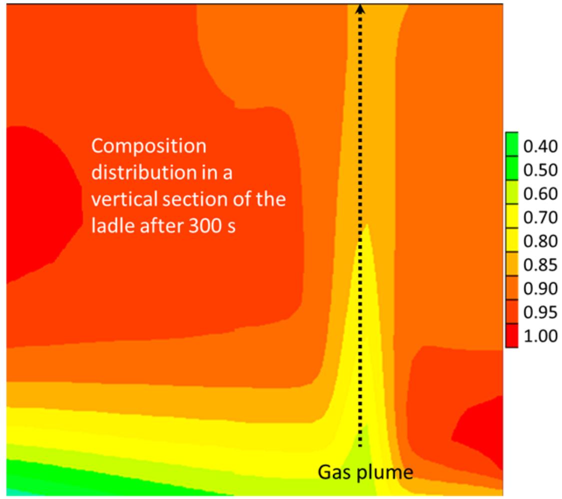
[3] Optimisation of sampling at liquid steel state and correlative inclusion assessment of liquid steel for the improvement of high performance steel grades production process (Sopliqs) – EUR 25039 2012

Measurements in ladle

Measurements in ladles must be accurate and representative.
Efforts were dedicated to improve the quality of the information studying the optimum positioning inside the ladle.







Fluid dynamic simulation of the evolution of steel composition in different positions inside the ladle after alloying operation to individuate the point more representative in the shorter time
(ladle 65 t, 2,4 hrigh, 2.4 diameter, stirring flow rate 50 L/min)

[4] De-oxidation practice and slag ability to trap non-metallic inclusions and their influence on the castability and steel cleanliness - 7210-PR/329, 2005

Monitoring and modelling of the Interaction between slag and steel

Most of the research activities were dedicate to the development of measurements techniques and models of the interaction between steel and slag and consequently on inclusion population.

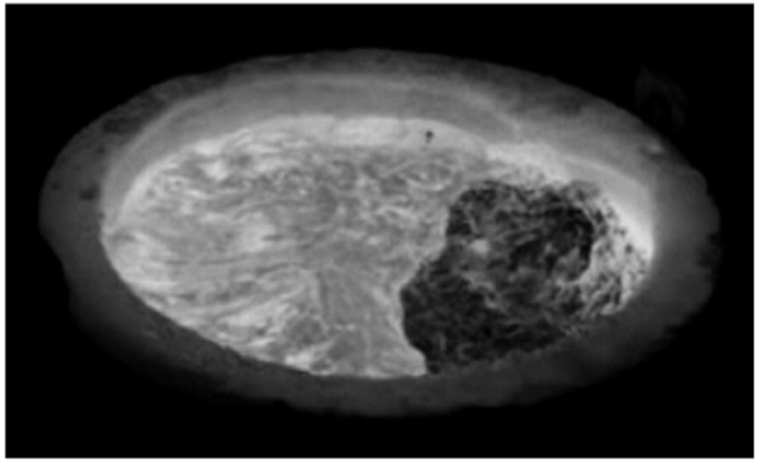
The impact of stirring practices and slag composition on the final steel quality has been investigated in deep.

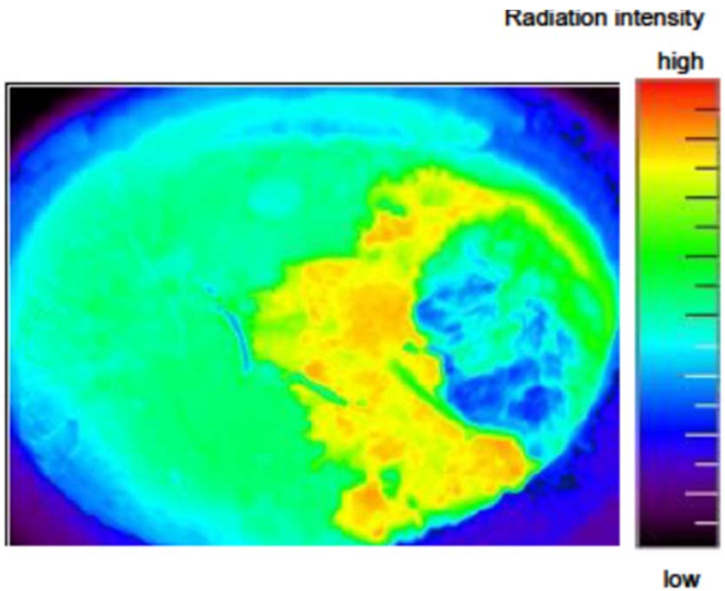
These researches contributed to define guidelines and rules for designing best slag composition that allows:

- To favour inclusion entrapment in slag, minimizing inclusion in the cast steel
- To control inclusions composition, reducing defects and casting problems

Monitoring of the interaction between slag and steel

The impact of slag properties and stirring conditions on slag/steel emulsification has been studied with experimental techniques.



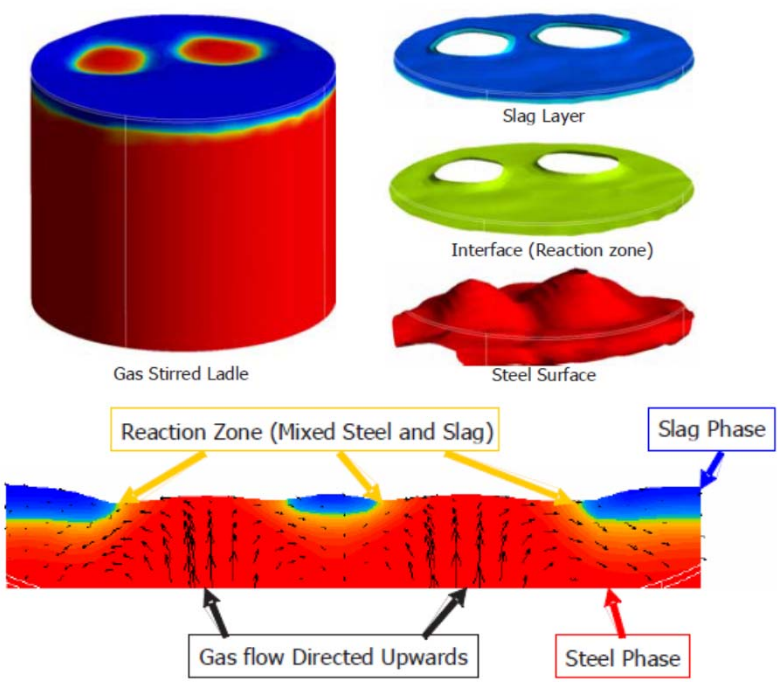


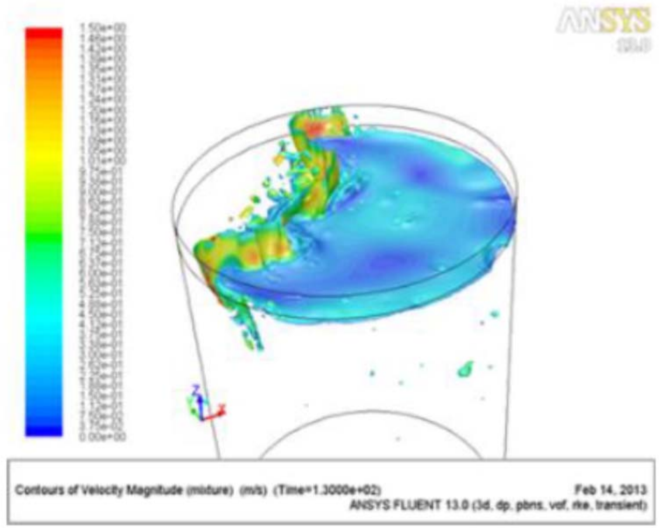
IR Camera images of steel and top slag under stirring conditions

[5] Improvement of ladle stirring to minimise slag emulsification and reoxidation during alloying and rinsing (Stimprove) – EUR 25 0168 2012

Modelling of the interaction between slag and steel

Fluid dynamic modelling has been used to evaluate the interaction between slag and steel and the ability of slag to entrap inclusions.





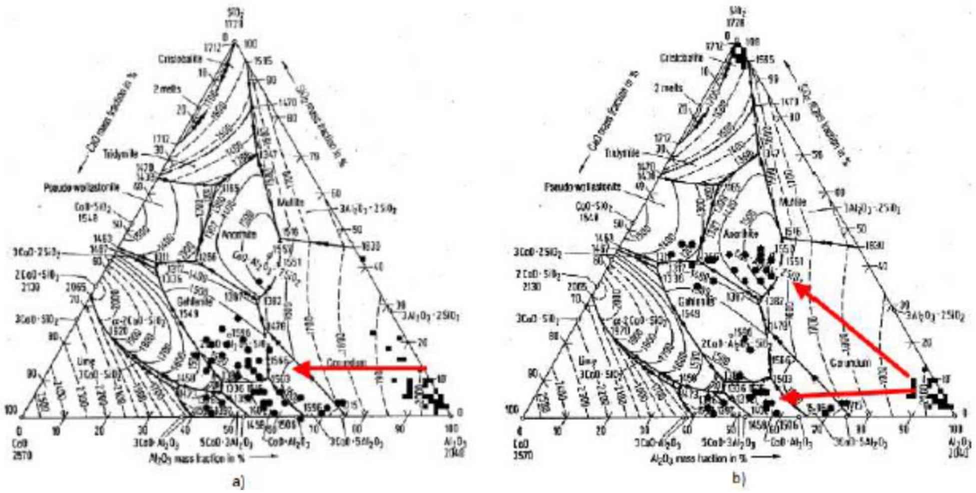
Fluid dynamic simulations of:

- slag and steel interaction (left) [6]
- inclusion entrapment in slag (right) [7]

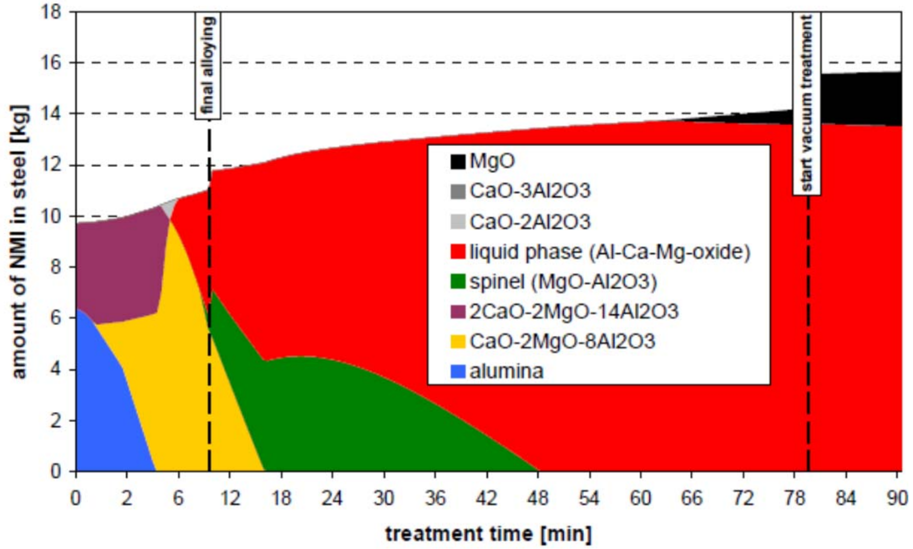
[6] Improved control of inclusion chemistry and steel cleanness in the ladle furnace – 7210-PR-331
[7] Intelligent cleanness controls in secondary steelmaking by advanced off- and online process models (INTCLEANCON) – EUR 27832 - 2014

Modelling of the interaction between slag and steel

The influence of the interaction between slag and steel has been investigated with measurements and thermodynamic modelling.



Evolution of inclusions with two different de-oxidation practices [8]
a) Si together Al
b) and Si before Al



Thermodynamic calculation (FactSage) of the evolution of inclusions during ladle treatment [9]

[8] Prediction of inclusions in the slabs from the process characteristics (PREDINC) – EUR 24992 - 2011
[9] Development of steel grade-related slag systems with low reoxidation potential in ladle and optimised ladle glaze techniques for improving steel cleanliness (STEELCLEANCONTROL) – EUR 25076 - 2013

Inclusion control:
Examples of follow-up in industrial applications

These researches contributed to define guidelines and rules for designing best slag composition that allows:

- To favour inclusion entrapment in slag, minimizing inclusion in the cast steel
- To control inclusions composition, reducing defects and casting problems

As a consequence of this big effort, today the effect of operating conditions and slag composition on inclusion population is well known and sophisticated and accurate models for designing operating practices and for the control operations are available.

The following examples show practical application of the integration of knowledge, mathematical models and on-line measurements for inclusion control.

Example 1
On-line slag adjustment for inclusion control

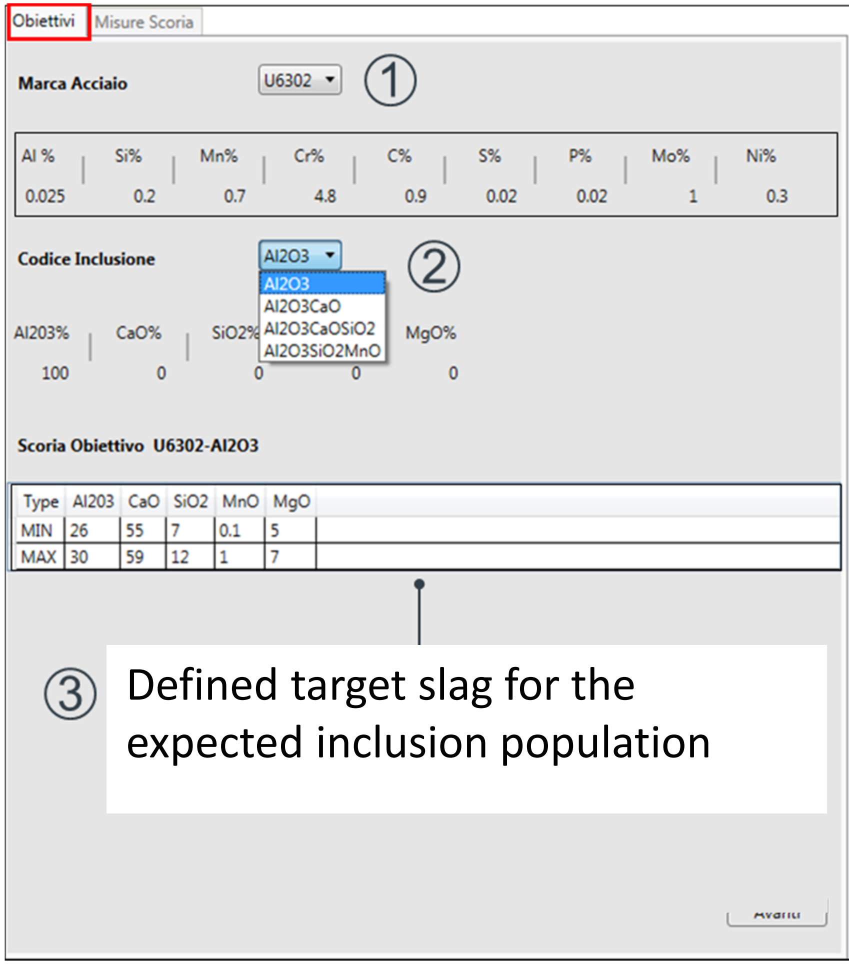
The knowledge gained on slag design for inclusion control has been implemented in industrial control systems.

The system is based on the following steps:

1) From target steel grades and operation sequence performed in ladle the expected inclusion population is individuated
2) For the modification and removal of the expected inclusion population the best slag composition is defined: the target slag composition
3) During secondary steelmaking operations slag samples are taken and analysed
4) From the current slag analysis and target slag composition type and amount of fluxes to add are calculated for obtaining the target slag composition

Example 1
On-line slag adjustment for inclusion control

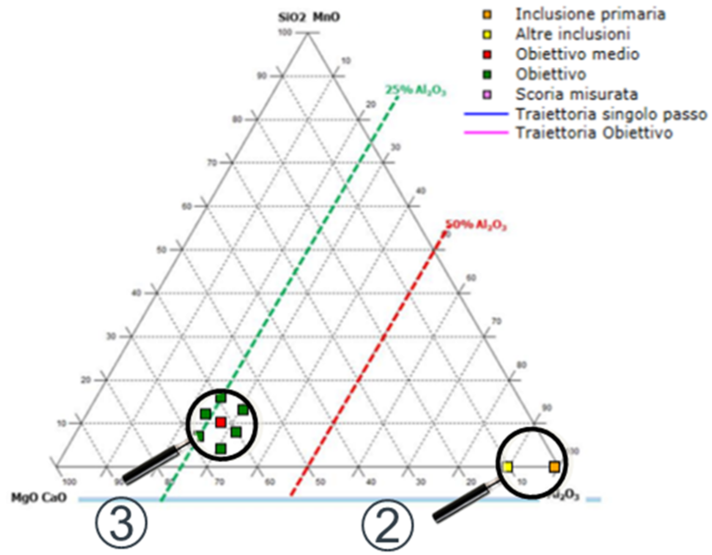
Steps 1 and 2 – Definition of expected inclusion population and target slag



① Selection of the steel grade and expected inclusions

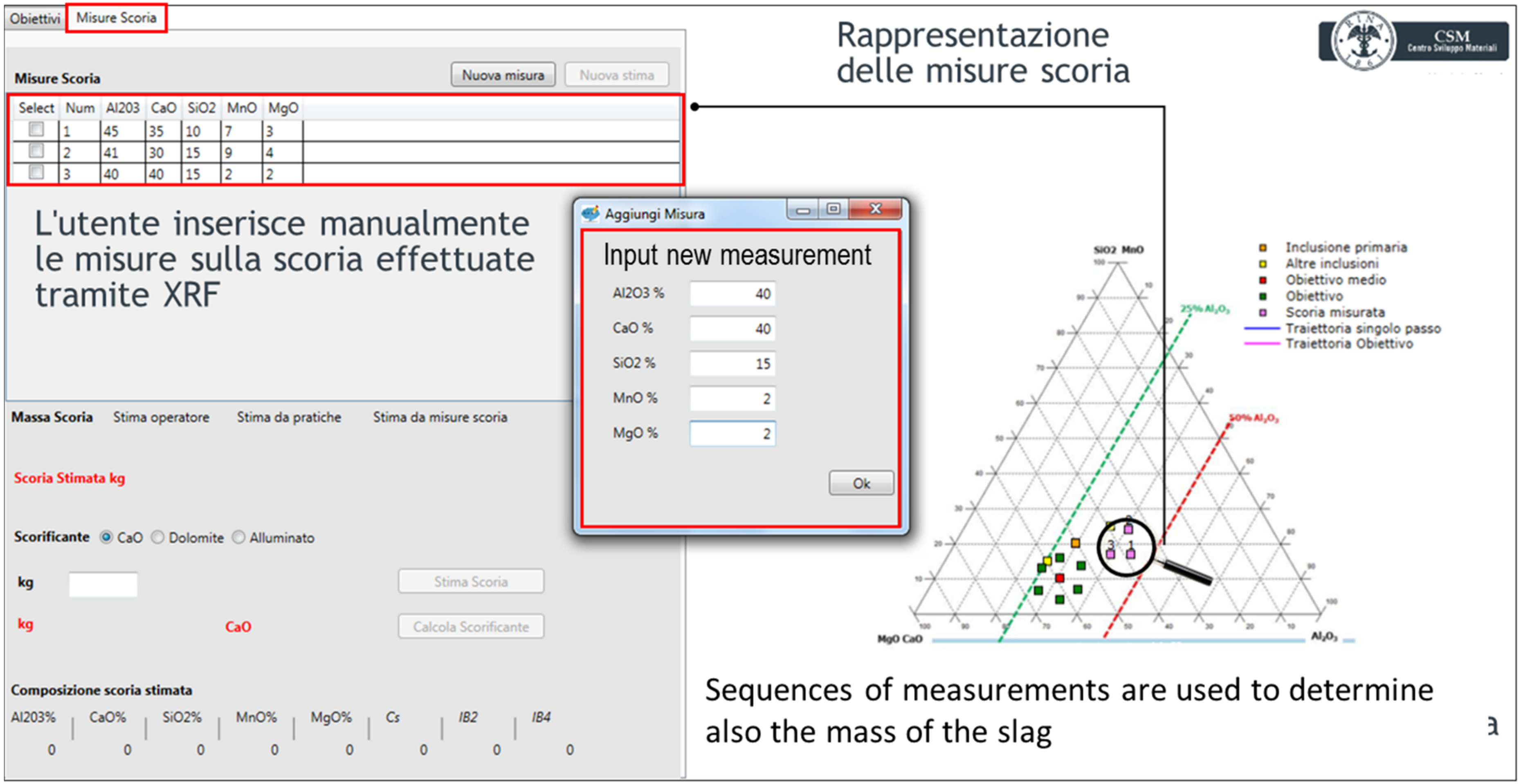
② Expected inclusion population (taking into account planned steelmaking operations)

③ Defined target slag for the expected inclusion population



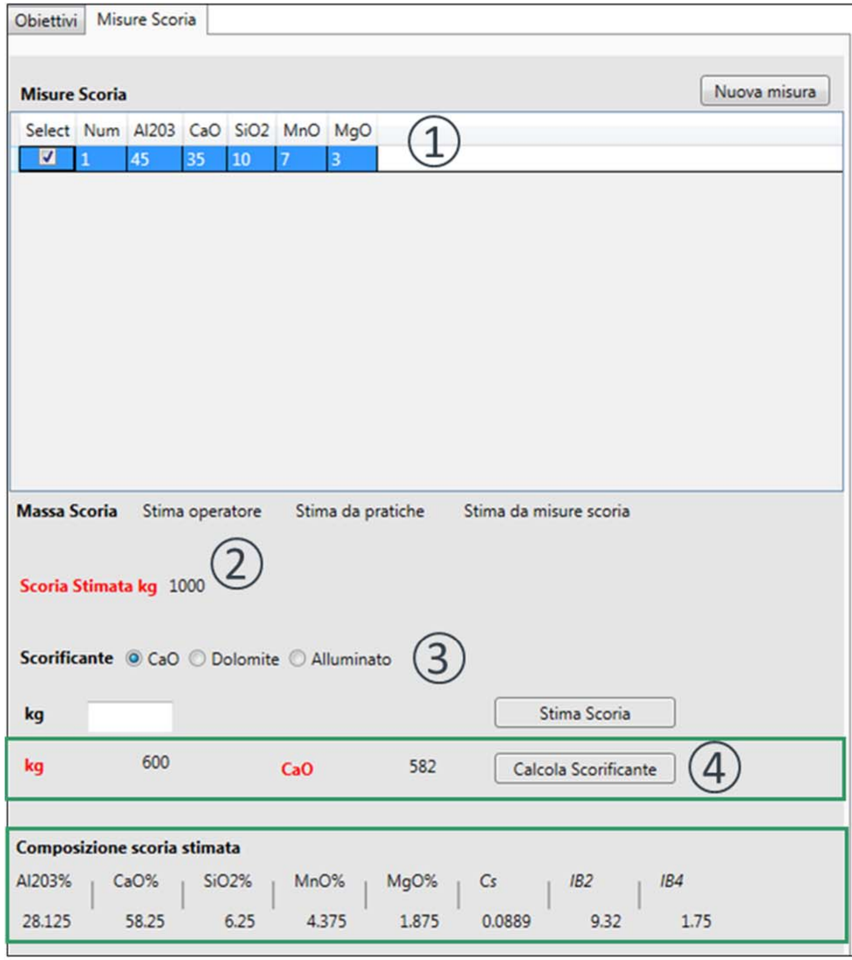
Example 1
On-line slag adjustment for inclusion control

Step 3 – Determination of actual slag composition



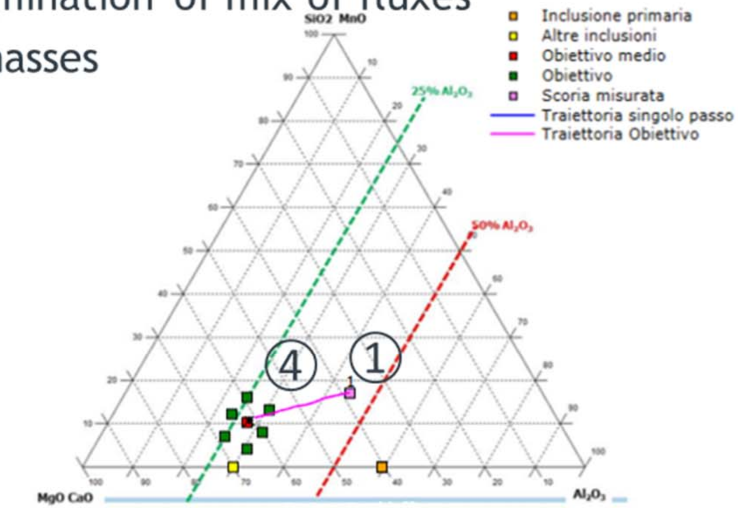
Example 1
On-line slag adjustment for inclusion control

Step 4 – Output: best mix and mass of fluxes to obtain target slag



Steps:

0. Selection of steel grade and expected inlcusions
1. Determination of slag composition
2. Determination of slag mass
3. Determination of mix of fluxes
4. and masses



Output
- mass of fluxes to add
- composition of obtained slag
- technological indexes

Example 2
Control of inclusions in Ca-treated Al-killed steel

In European researches, thermodynamic calculations have been used, for instance, to calculate the effect on inclusion composition of:

- De-oxidation practices with different ferroalloying mixes and order of addition
- Trace elements in steel (e.g. Mg)
- Emulsification of slag
- Re-oxidation of steel from air entrance
- Interaction of steel with refractories

The final composition of the inclusions impacts on both steel quality (formation of defects) and castability (clogging)

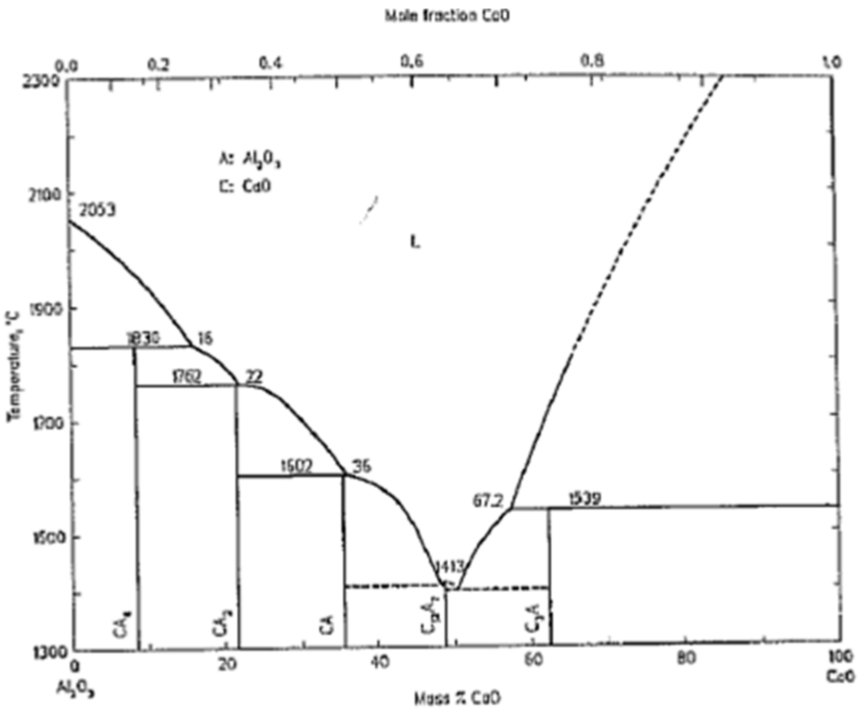
The following examples refer to thermodynamic calculation applied for the classical case of the design of the calcium treatment for inclusion modification.

The aim is to show the benefit of integration of thermodynamic calculation and measurements

Example 2
Control of inclusions in Ca-treated Al-killed steel

The main effect of Calcium treatment on alumina inclusions is well known.

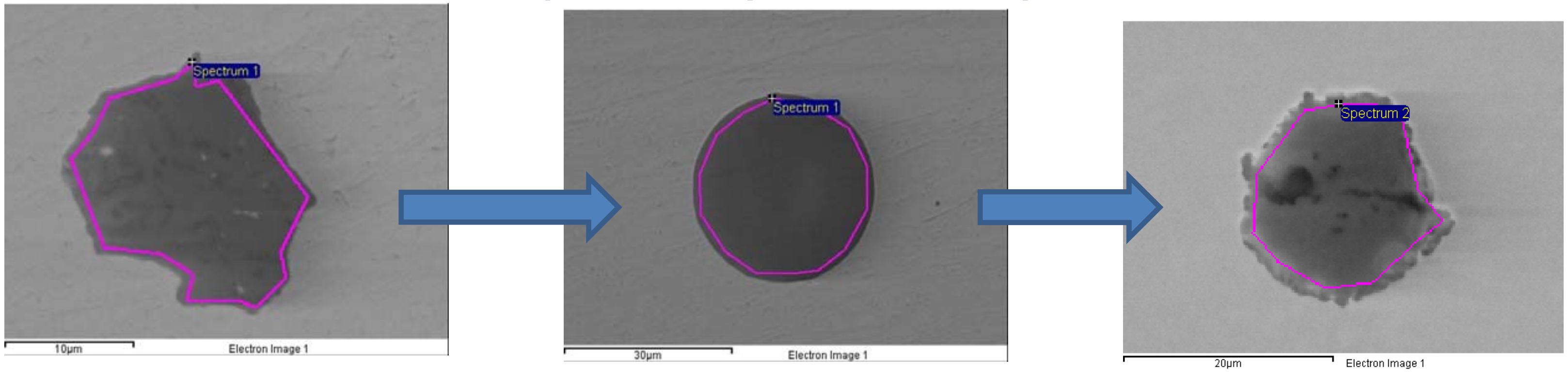
The existence of liquid calcium aluminates is exploited to transform solid alumina into liquid. This technique is useful to prevent nozzle clogging during continuous casting and to favor inclusion coalescence and floatation, to obtain clean steel. The phase diagram shows the compositional range of liquid calcium-aluminate

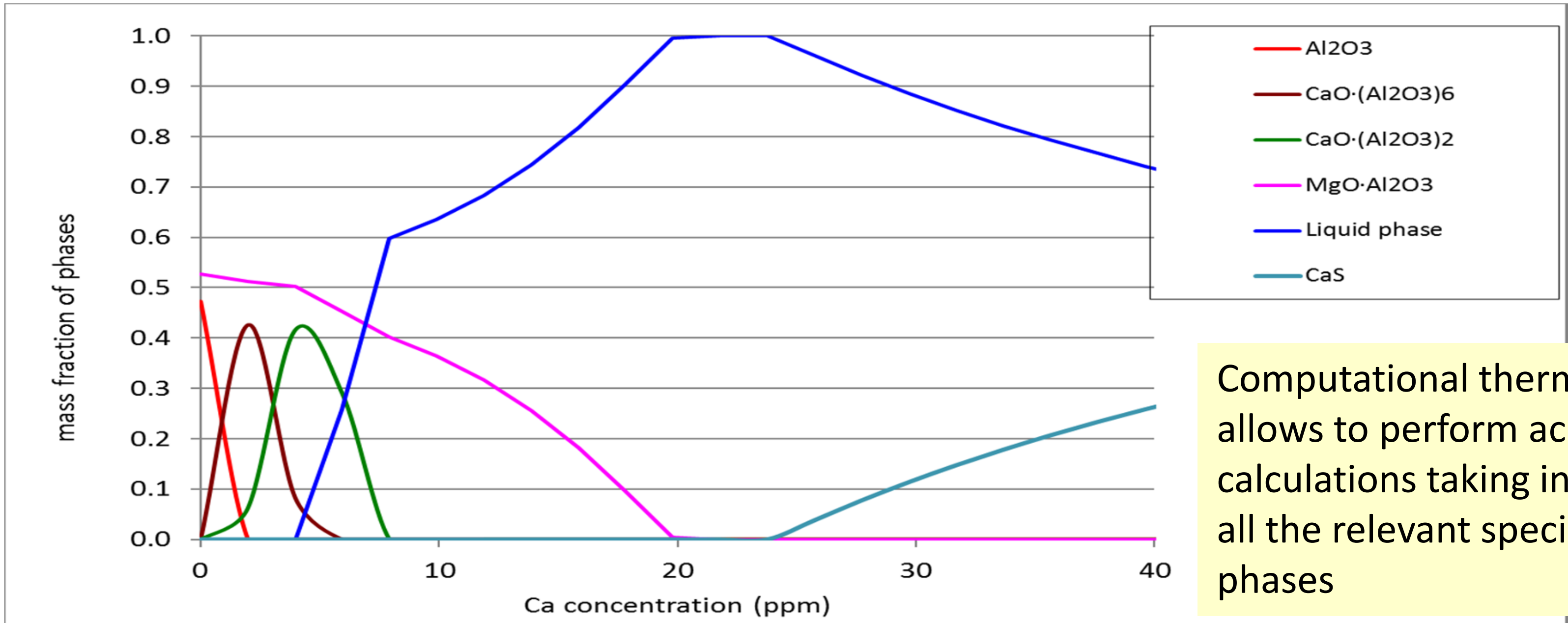


The binary phase diagram of the system Al_2O_3 – CaO is an oversimplified schematization. Useful for didactic purpose, but of scarce usefulness in real application, where the inclusions are much more complex and the presence of trace elements in steel can change dramatically the final result.

Example 2
Control of inclusions in Ca-treated Al-killed steel

solid inclusion (Alumina) — **Liquid inclusion (Calcium Aluminate)** — **Multiphasic inclusion AlCa with CaS**



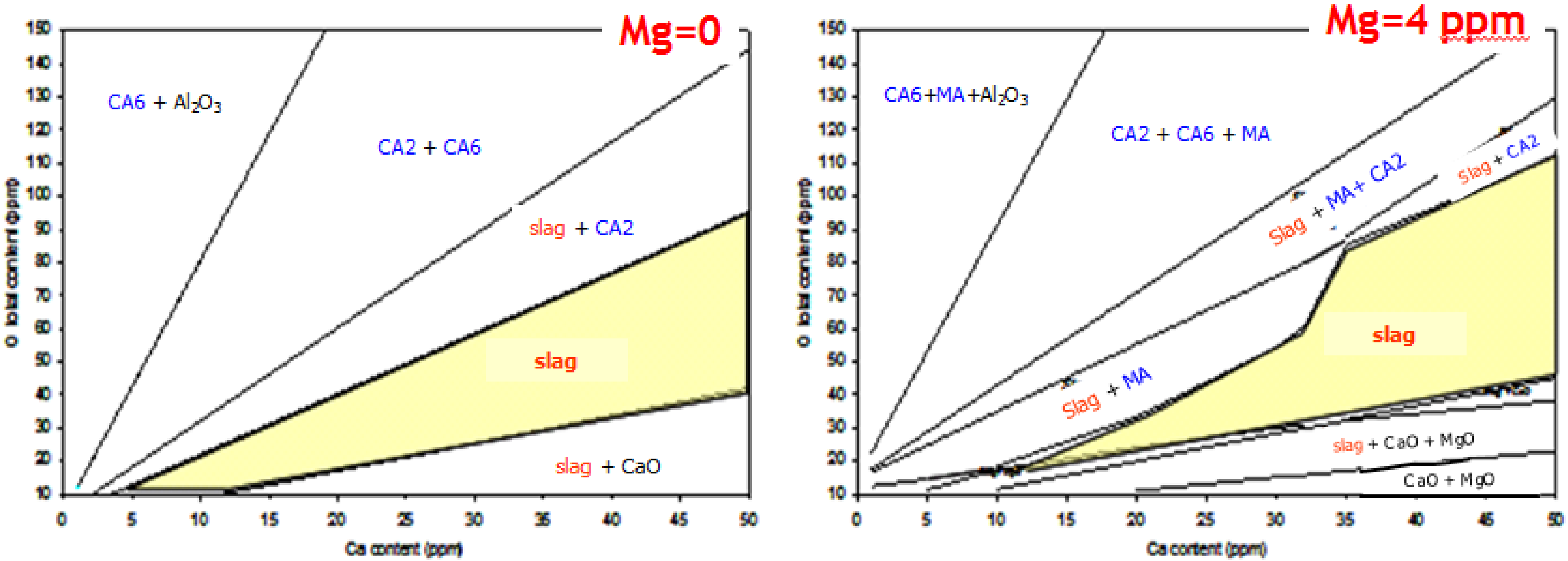


Computational thermodynamics allows to perform accurate calculations taking into account all the relevant species and phases

CSM calculation with Thermocalc®

Example 2
Control of inclusions in Ca-treated Al-killed steel

Influence of Mg on inclusion modification with Calcium



Phases present in inclusions in equilibrium with steel as a function of Ca and O content (total concentration). The calculations have been performed without magnesium (left) and with magnesium (right). The yellow regions indicate liquid inclusions. The presence of magnesium in the system changes completely the concentration of calcium that allows the existence of inclusions in the liquid state.

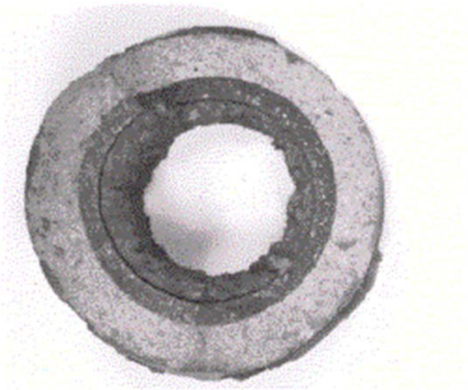
Steel composition: Al = 400 ppm; Si = 100 ppm; Mn = 2500 ppm; **Mg = 0** (left) **4 ppm** (right);

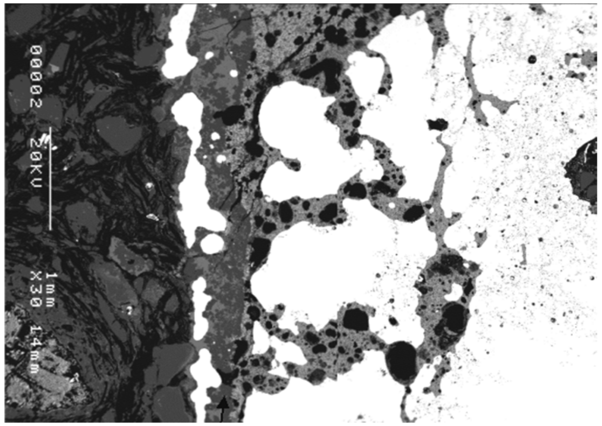
Temperature: 1600°C

Example 2
Control of inclusions in Ca-treated Al-killed steel

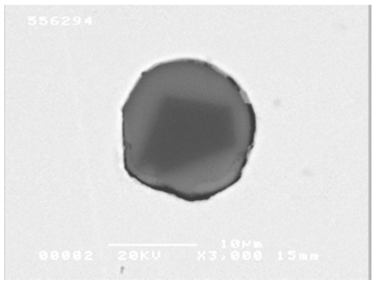
A major objective of Ca-treatment is to reduce the problems of clogging during casting.

For this reason the transformed inclusions must remain liquid also at the casting temperature (to avoid sticking of liquid/solid inclusions to the refractory in continuous casting, such as stopper rod or Submerged Entry Nozzle)





Chemical characteristics of inclusions affect the formation of deposits inside the casting devices



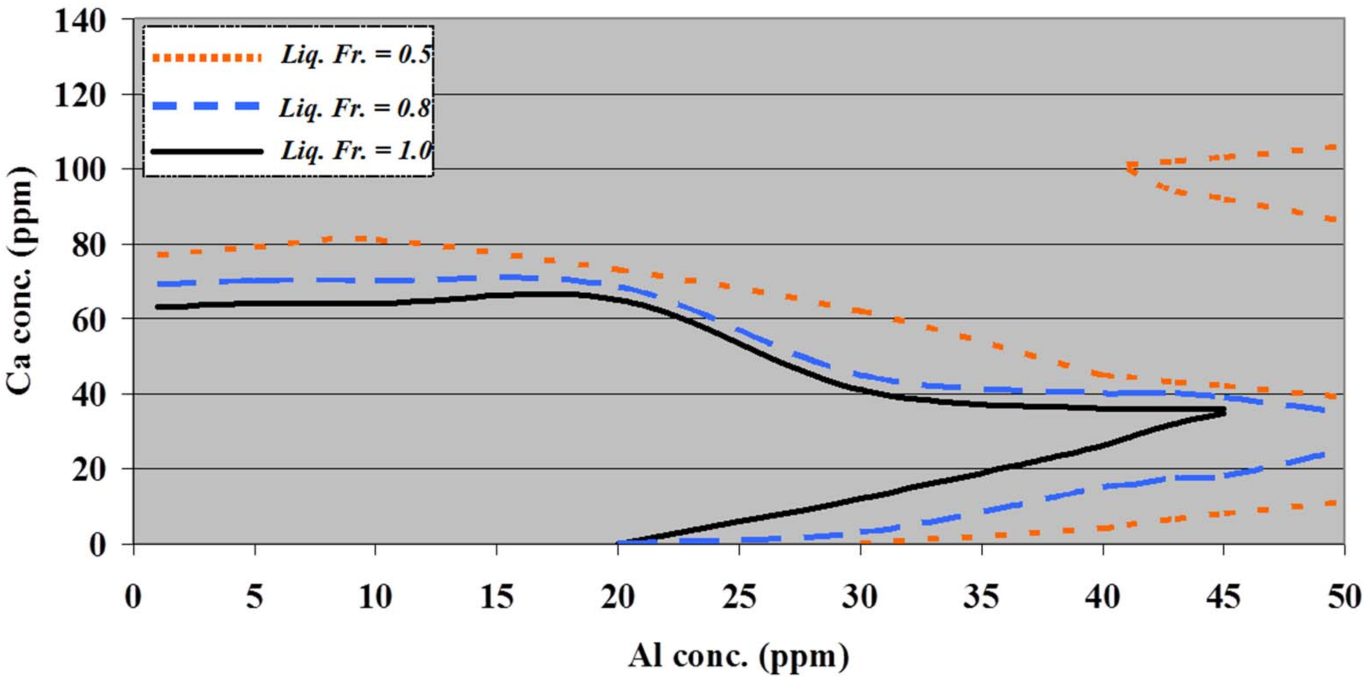
For example inclusions at high Al_2O_3 content and small fraction of a liquid phase (5-10%) tend to give deposits, reducing the steel flow.

Example 2
Control of inclusions in Ca-treated Si-killed steel

To perform accurate calculation is necessary to take into account all the relevant species and for different possible conditions
(for example for different values of Al pollution and total oxygen).
Calculation of liquid fraction of inclusions as a function of Al and Ca concentration at casting temperature

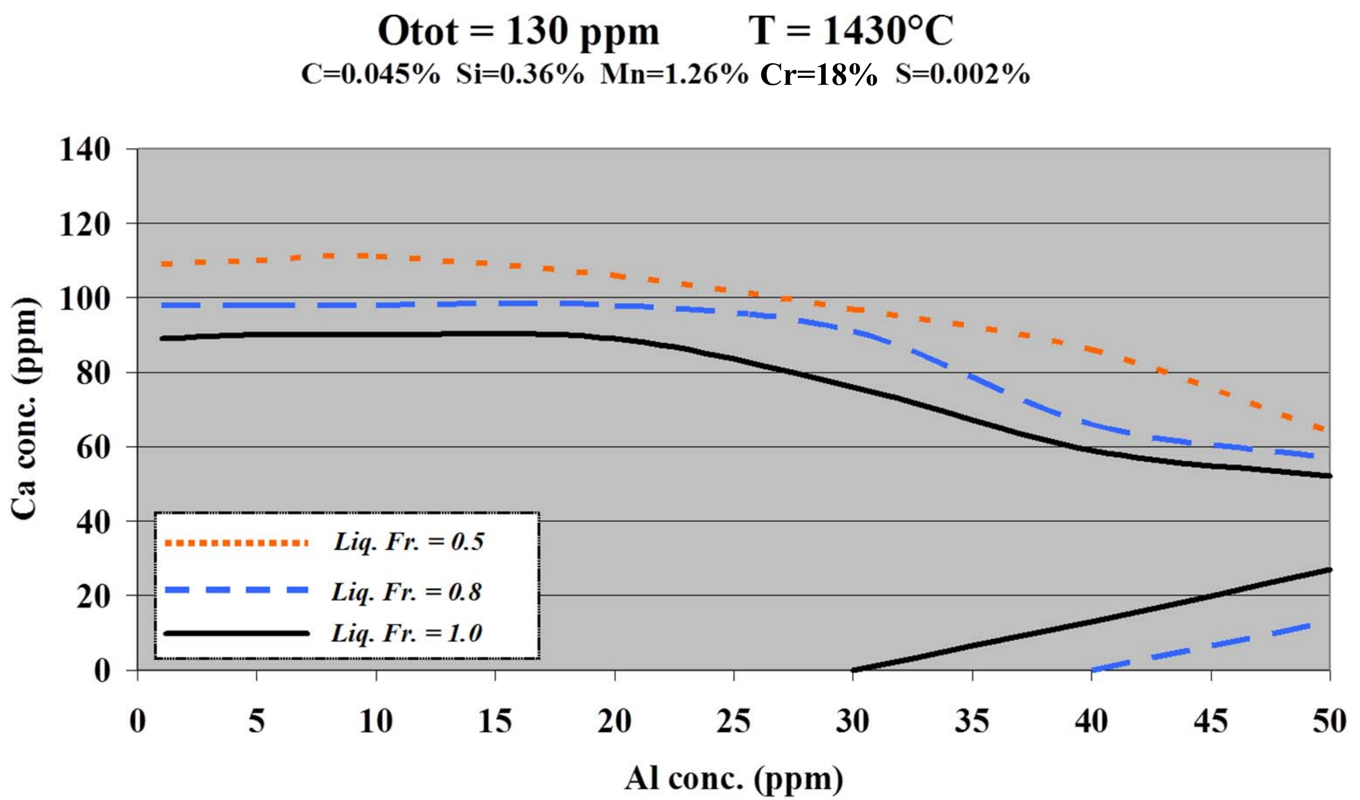
Otot = 100 ppm T = 1430°C

C=0.045% Si=0.36% Mn=1.26% Cr=18% S=0.002%



Example 2
Control of inclusions in Ca-treated Si-killed steel

To perform accurate calculation is necessary to take into account all the relevant species and for different possible conditions
(for example for different values of Al pollution and total oxygen).
Calculation of liquid fraction of inclusions as a function of Al and Ca concentration at casting temperature



BFI
MPI Materials Processing Institute
Excellence in Materials & Process Innovation
CRM GROUP
swerea MEFOS
CSM Centro Sviluppo Materiali

Example 2
Control of inclusions in Ca-treated steel

The previous examples used information not rapidly available on the plant, such as concentration of total oxygen, which is possible to measure only in chemical analysis laboratory with a long procedure.

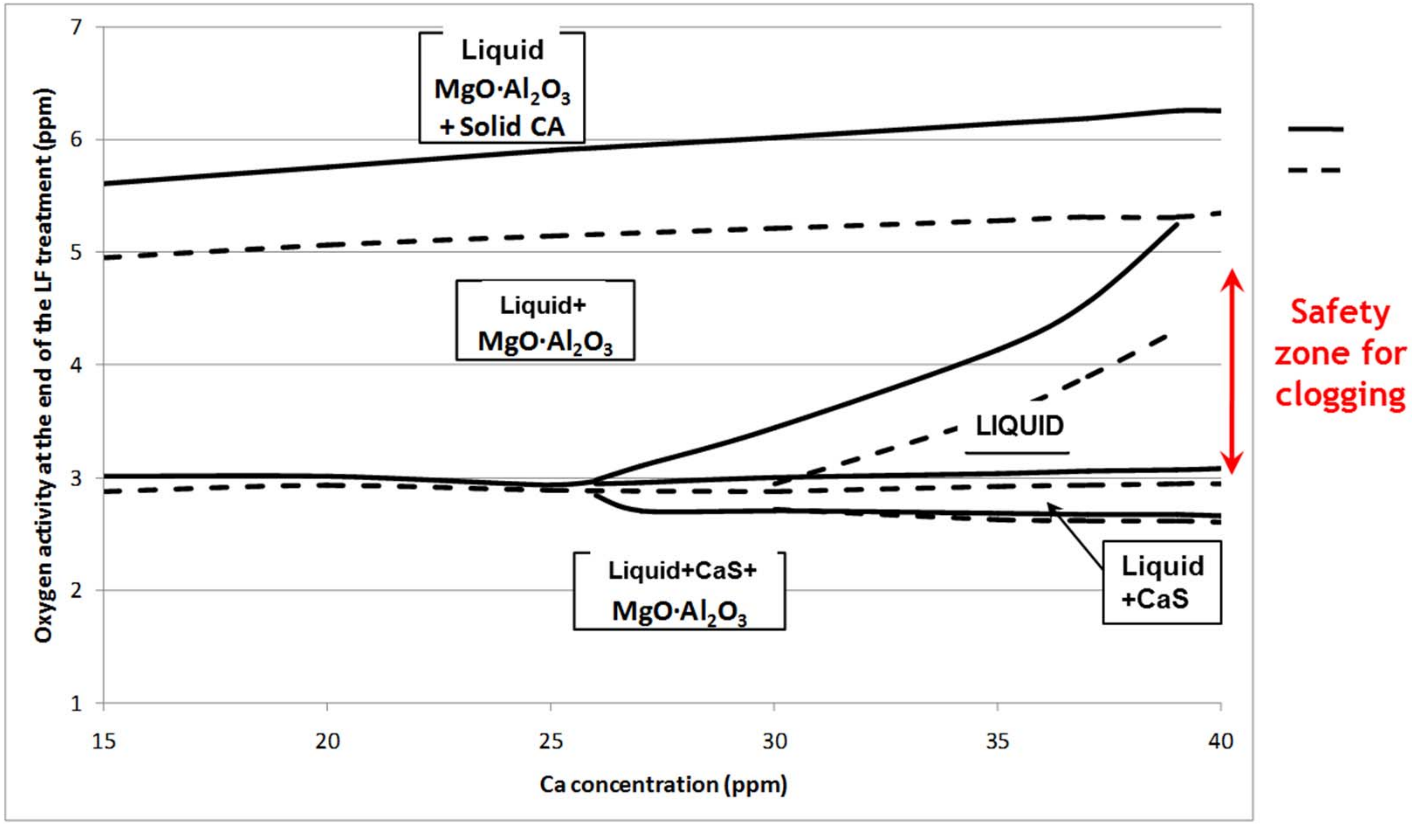
However the same thermodynamic calculations can be performed starting from data available (at least in principle) on-line, such as concentration of dissolved oxygen, concentration of total and dissolved aluminum.

The following two examples show calculations of inclusion composition that use as input quantities available from on-line measurements.

The calculations have been performed to build maps of conditions giving the ranges of safety conditions for casting (no clogging).

Example 2
Control of inclusions in Ca-treated Al-killed steel

Calculation of solid and liquid phases formed in Ca treatment.
Complete liquid inclusions should be obtained to prevent clogging problems



Al = 0.03%
Al = 0.04%

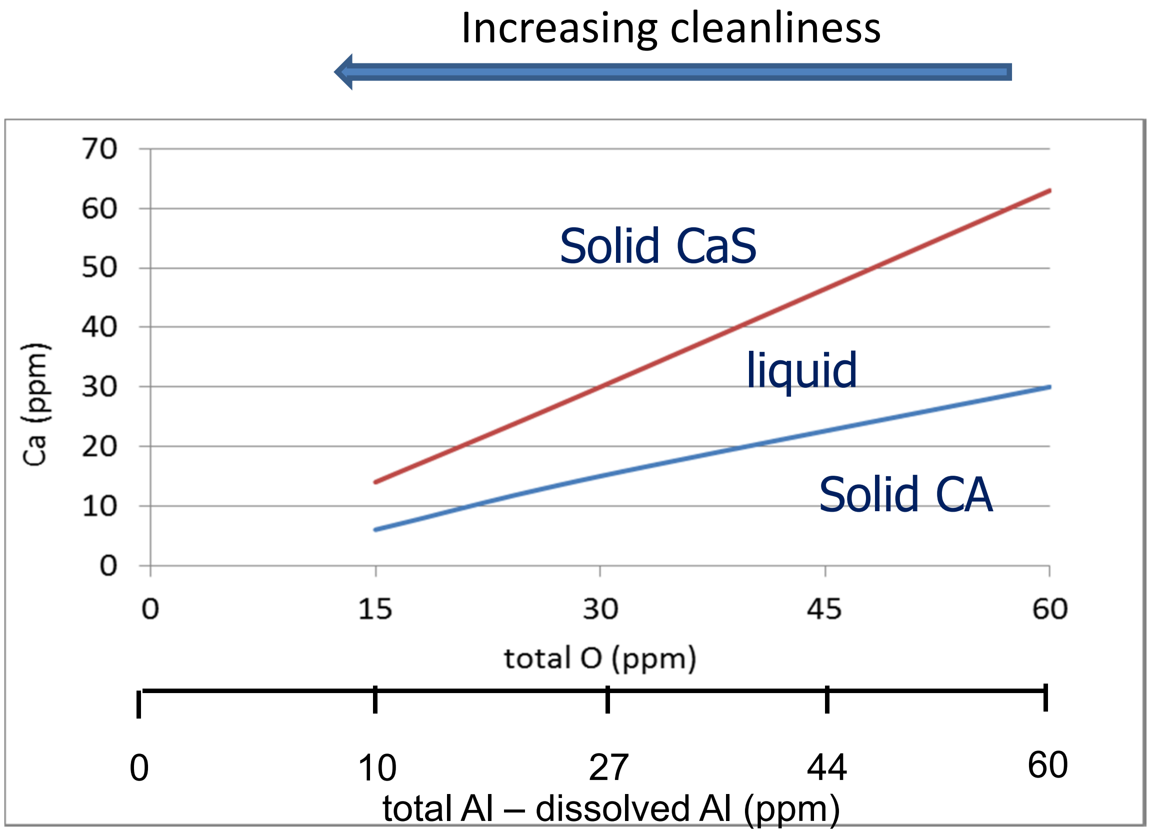
C=0.17%
Si= 0.30%
Mn=1.70%

Mg = 4 ppm
S = 200 ppm

Map of safety and dangerous inclusions calculated as a function of Ca and dissolved oxygen concentration

Example 2
Control of inclusions in Ca-treated Al-killed steel

Calculation of Ca addition from measurements of Al concentration potentially available on-line



Steel: Si 0.45%; Mn 1.5%; Al 300 ppm; S 100 ppm;
Temp 1600 °C

This diagram indicates the windows available for a proper transformation of alumina inclusions into fully liquid aluminates without CaS precipitation in liquid steel. The calculation has been performed starting from concentrations of total and dissolved Aluminum.

It is evident that for low concentration of oxygen the window is narrow, a strict control of Ca addition is necessary.

Example 2
Control of inclusions in Ca-treated Al-killed steel

The classical case of calcium treatment is an example where the integration of thermodynamic calculations with measurements potentially already available on the plants could be used for on-line control of the operation.

In this case measurements of concentration of dissolved oxygen and dissolved aluminium (already possible with the current techniques) can be used to guide the process.

A vision of the future:
continuous measurements and control

The large number and the quality of the European researches on secondary steelmaking technology made available a huge knowledge and validated mathematical techniques.

Many useful measurement methods are already available.
The integration of the measurements with the models constitutes a powerful tool for the design of operating practices and the control of operations.

The control of steel quality with the integration of existing models and existing discontinuous measurements can be already applied in all the secondary steelmaking operations:

- De-oxidation
- De-sulfurization
- De-gassing
- Ca-treatment
- Inclusion floatation
- Control of re-oxidation problem

A great step forward would be the development of continuous measurements

A vision of the future: continuous measurements and control

Continuous or rapid measurements for continuous monitoring and control of inclusion population:

Continuous Temperature

for accurate calculations of steel quality

Continuous Dissolved O, S, Al

for control of inclusion population in de-oxidation, de-sulfurization,

Rapid determination of Ca, Mg, Inclusions number and size

for rapid determination of inclusion population and risk for castability

Some, examples of techniques for these measurements can be found in the European projects.

Further developments in this direction would be desirable

Thank you