

# Cleanness assessment of high quality steels produced by RH treatment

PIERRET Jean-Christophe (CRM)

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Cleanness assessment of high quality steels produced by RH treatment

# Context - Rurhstahl Heraeus (RH) Degasser

- ❖ RH plants are world-wide commonly used for secondary metallurgy processes like e.g. decarburization and alloying. In the recent time, and with regard to provide an optimum steel cleanliness, another important task of the RH process is the **removal of non-metallic inclusions** suspended in the melt.
- ❖ Therefore, a detailed knowledge of the relevant physical mechanisms playing a role during RH treatment is necessary for a better **understanding of the process** and therefore for its further improvement.
- ❖ In addition, possibilities to online measure and/or control the inclusions in the melt and to **predict the quality of the final product** are of high interest.



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# Related RFCS projects

	Project name	Partners	Duration
1	Development of a model for the vacuum circulating process	<u>BFI</u>	01/08/1990 - 31/01/1994
2	Improvement of vacuum circulation plant operation on the basis of the BFI simulation model	BFI, VoestAlpine, <u>CSM</u>	01/07/1995 - 31/12/1998
3	Control of inclusions in RH degassing processes	<u>ArcelorMittal/IRSID</u> , Corus, CRM, BFI	01/07/1997 - 31/12/2000
4	In-line assessment of steel cleanness during the secondary steelmaking processes	<u>ArcelorMittal</u> , Ascometal/CREAS, CRM, Sidenor	01/07/2001 - 31/12/2004
5	Improvement of inclusion flotation during RH treatment	<u>ArcelorMittal</u> , CSM, BFI, RWTH	01/07/2001 - 31/12/2004

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# 1. Development of a model for the vacuum circulating process

- ⌘ Working period: 01/08/1990 - 31/01/1994
- ⌘ Consortium : BFI
- ⌘ Objectives :
  - ⌘ To develop a detailed dynamic model for decarburisation in the RH process, which can be used for on-line monitoring of the process behaviour.
- ⌘ Main results :
  - ⌘ Process model was provided as simulation model with validation by industrial process data.
  - ⌘ On-line application was possible, but not foreseen within the project.

## 2. Improvement of vacuum circulation plant operation on the basis of the BFI simulation model

- ⌘ Working period: 01/07/1995 - 31/12/1998
- ⌘ Consortium : BFI, VoestAlpine, CSM
- ⌘ Objectives :
  - ⌘ To improve vacuum circulation plant operation on the basis of the BFI simulation model in order to include further metallurgical operations such as stirring, gas input, oxygen blowing, dehydrogenation and nitrogen control and to improve process control of RH plants.
- ⌘ Main results :
  - ⌘ Dynamic process model for description of degassing and temperature behaviour during RH process

### 3. Control of inclusions in RH degassing processes

- ⌘ Working period: 01/07/1997 - 31/12/2000
- ⌘ Consortium : IRSID, Corus, CRM, BFI
- ⌘ Objectives :
  - ⌘ Industrial goal: to improve the control of inclusions during the RH degassing process.
  - ⌘ R&D goal : to develop modelling techniques, to test and to validate them with steel plant measurements and/or physical modelling in order to provide validated tools for evaluation of liquid steel temperature, composition and cleanness in the RH degasser process.
- ⌘ Main results :
  - ⌘ 2D/3D CFD modelling of RH degassing process
  - ⌘ Development of reagent injection procedure and validation of efficiency
  - ⌘ Development of new methods for samples characterisation

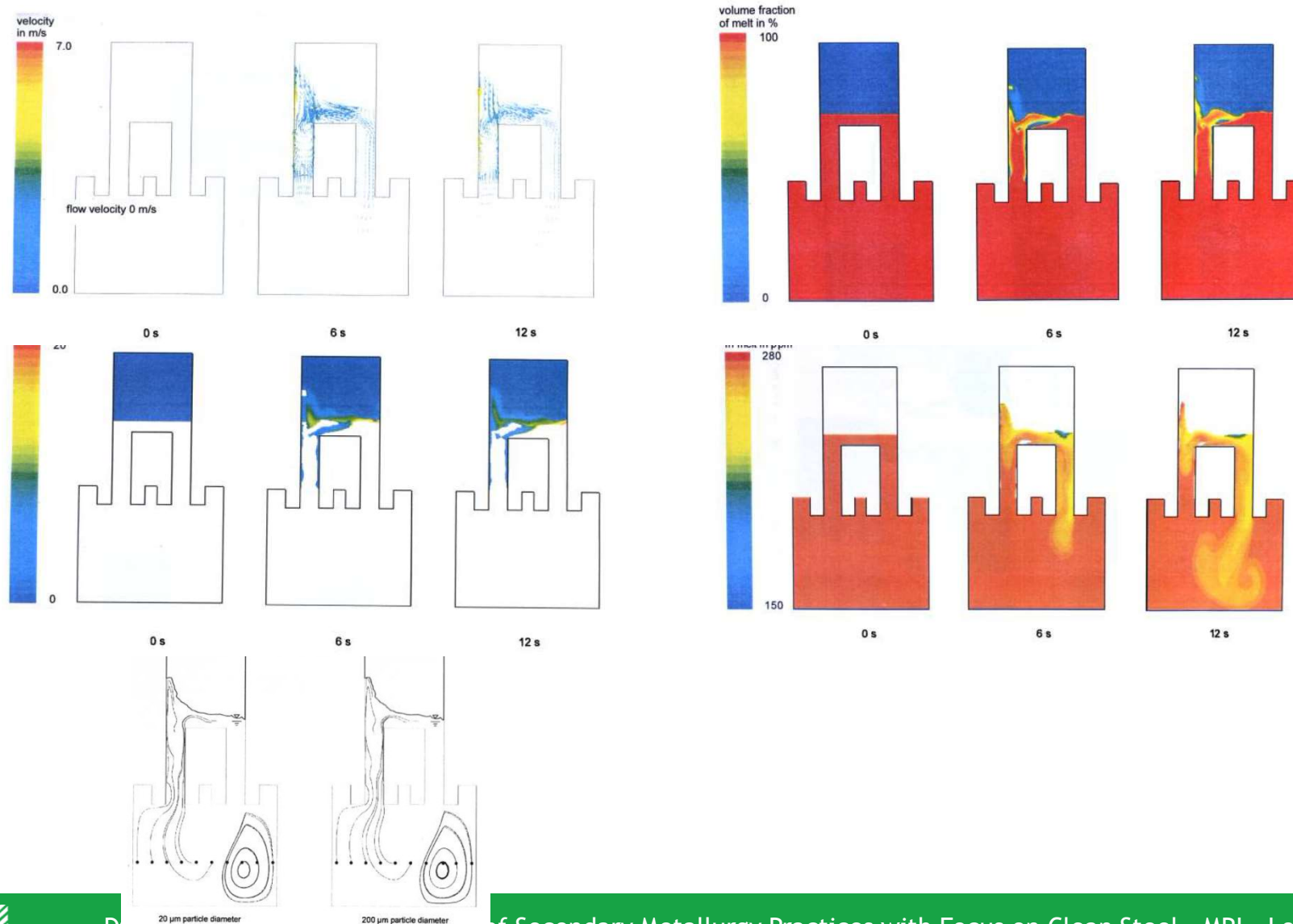
### 3. Control of inclusions in RH degassing processes - CFD modelling

- ⌘ Modelling techniques for the evaluation of the RH vacuum degasser performance with respect to steel cleanliness have been developed, tested and validated.
- ⌘ Numerical simulation computations and physical model trials have been performed with special attention paid to the dynamic interactions between the three phases melt, gas and non-metallic inclusions.
- ⌘ 2D and 3D simulations, time-dependent flow and time-dependent heat transfer, movement of the free surface, decarburisation and separation of non-metallic inclusions.
- ⌘ 2D simulations have shown that it is possible to take into account several of the physical mechanisms simultaneously in one numerical computation in order to account for coupled physical effects.
- ⌘ CFD code has therefor been applied to 3D set-up but CPU time was (at that time) exorbitant. Therefore, the relevant physical mechanisms have been investigated separately.

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### 3. Control of inclusions in RH degassing processes - CFD modelling



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### 3. Control of inclusions in RH degassing processes - physical modelling

- ⌘ Physical model investigations have been conducted with regard to flow visualisation, residence time analysis and simulation of decarburisation.
- ⌘ Numerical and experimental results obtained have been compared among each other and to operational data found in the literature with good agreement for what concern flow visualisation and residence time.
- ⌘ For decarburisation, a factor 2 in the reaction rate was observed.

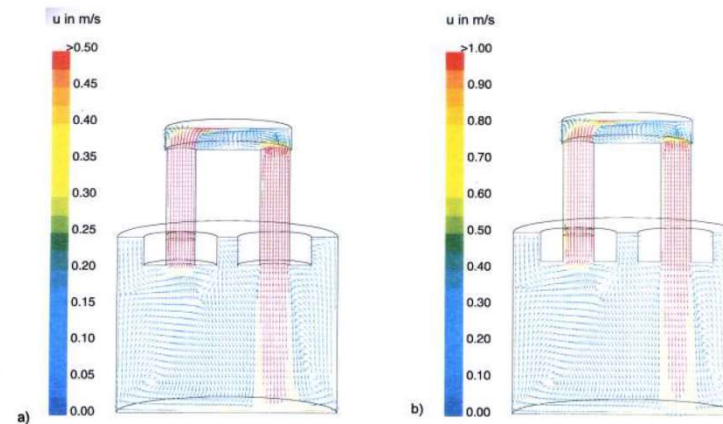
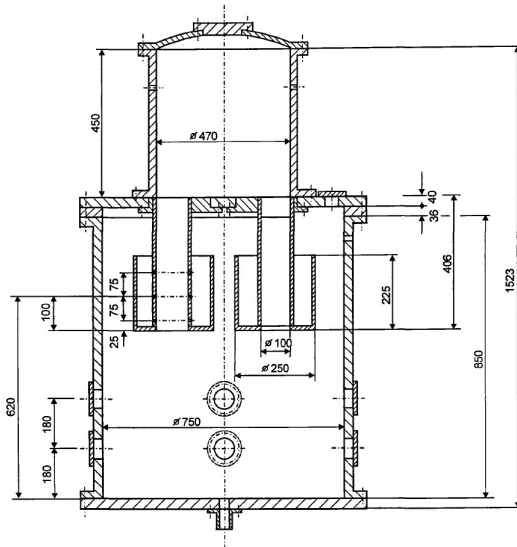


Figure 1.12: Comparison of numerically computed melt velocities for the symmetry plane of the RH set-up (3D-simulation)  
a) physical RH model  
b) RH plant

### 3. Control of inclusions in RH degassing processes - Injection of reagent powder

- ⌘ The possibility to modify and to better control the melt composition and the inclusions content by a reactive powder injection during the RH treatment has been investigated.
- ⌘ Follow-up of industrial trials realized in the RH installation equipped for injecting reagent powder during the vacuum treatment. Several distinct RH conditions have been fully characterized.
- ⌘ Objectives:
  - ⌘ Reduction of the steel sulphur and nitrogen level
  - ⌘ Delivery of highly clean steel

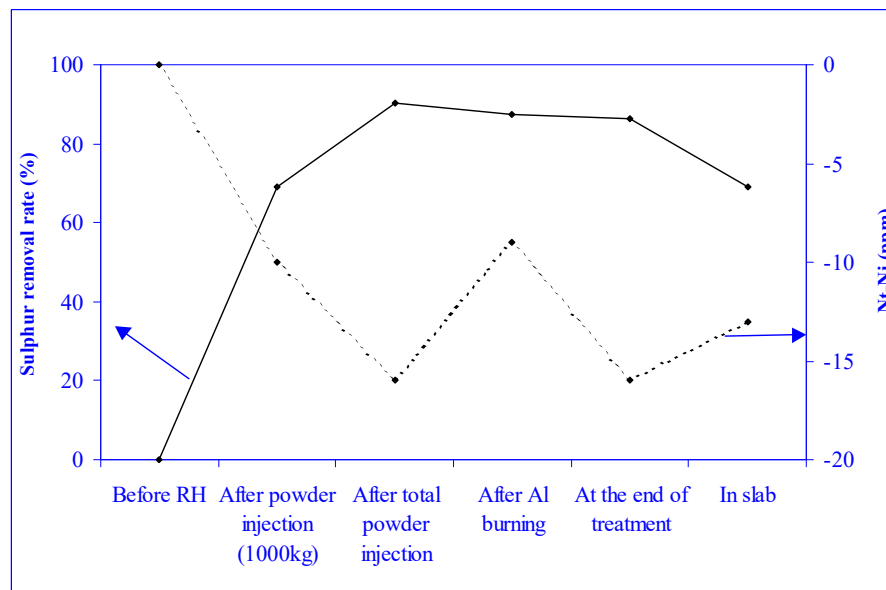
### 3. Control of inclusions in RH degassing processes - Injection of reagent powder

#### ⌘ Before RH treatment

- ⌘ Deslagging of the ladle and deposit of a new basic top slag
- ⌘ Increasing of the aluminium content(0,15%)

#### ⌘ During RH treatment

- ⌘ Injection through the lance of the reagent powder( max .100 Kg/min)
- ⌘ Burnt off of the excess of Al content
- ⌘ Addition of ferroalloys for a fine tuning of the aimed steel composition.



### 3. Control of inclusions in RH degassing processes - Assessment of steel cleanness

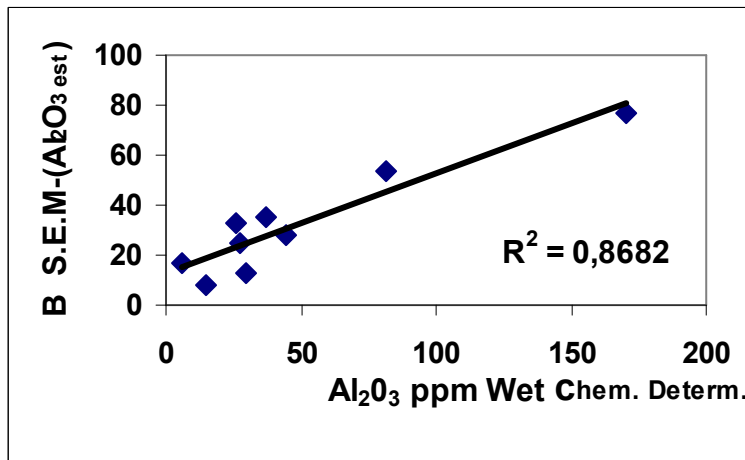
- ⌘ Besides the conventional optical and electronic image analysis techniques, new procedures, based on SEM and on PDAOES, have been developed in order to characterise these samples.
- ⌘ S.E.M. (Scanning Electron Microscopy - analysis time = 8h)
  - ⌘ For sorting in semi-quantitative approach the inclusion content by size and composition
- ⌘ P.D.A.O.E.S. ( Pulses Distribution Analysis Optical Emission Spectrometry - analysis time=5min)
  - ⌘ By using spark by spark discrimination, setting up of a method to quickly obtain information about the inclusion content

### 3. Control of inclusions in RH degassing processes - SEM procedure methodology

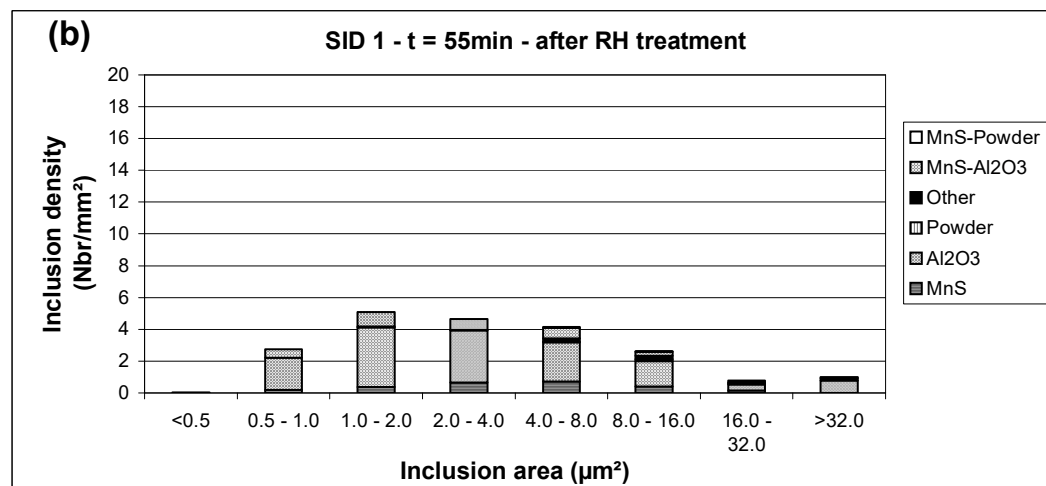
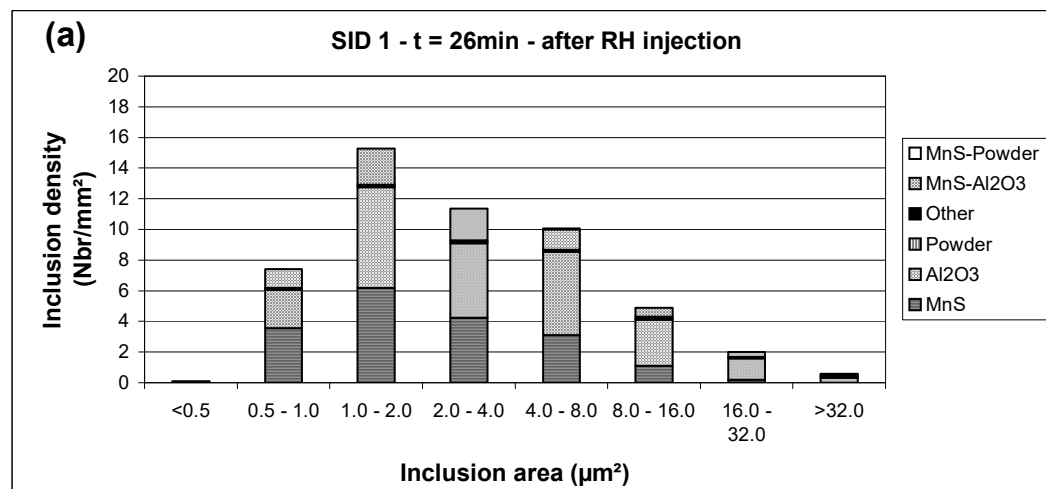
- ⌘ Through an automatic method, a large surface of the steel sample was scanned with an electron beam. Each inclusion detected was characterised in size (area, shape factor, diameter...) and in composition (content in Al, Mn, S, O, Ti, Ca, Na, K...)
- ⌘ For getting sufficient statistical representability a minimum of 1000 particles should be analysed. Depending on the inclusion density, 300 to 3000 frames had to be investigated (total area 10-15mm<sup>2</sup>).
- ⌘ The results must be evaluated and processed adequately for avoiding false interpretations and artefacts due to risks of interactions between the metallic matrix, the non metallic and the metallic particles and precipitates.

### 3. Control of inclusions in RH degassing processes - SEM procedure validation

- ⌘ Image analysis was also carried out by using optical microscopy coupled to an image analyser.
  - ⌘ Reproducibility tests have been performed in order to compare the information obtained by Image Analysis and by SEM. In terms of reproducibility, the order of magnitude was equivalent.
  - ⌘ Except the particles characterised by a very low area ( $<1 \mu\text{m}^2$ ) the comparison of the size distribution of the particles are comparable for the two methods
- ⌘ Measured composition by SEM was compared to wet chemical determination



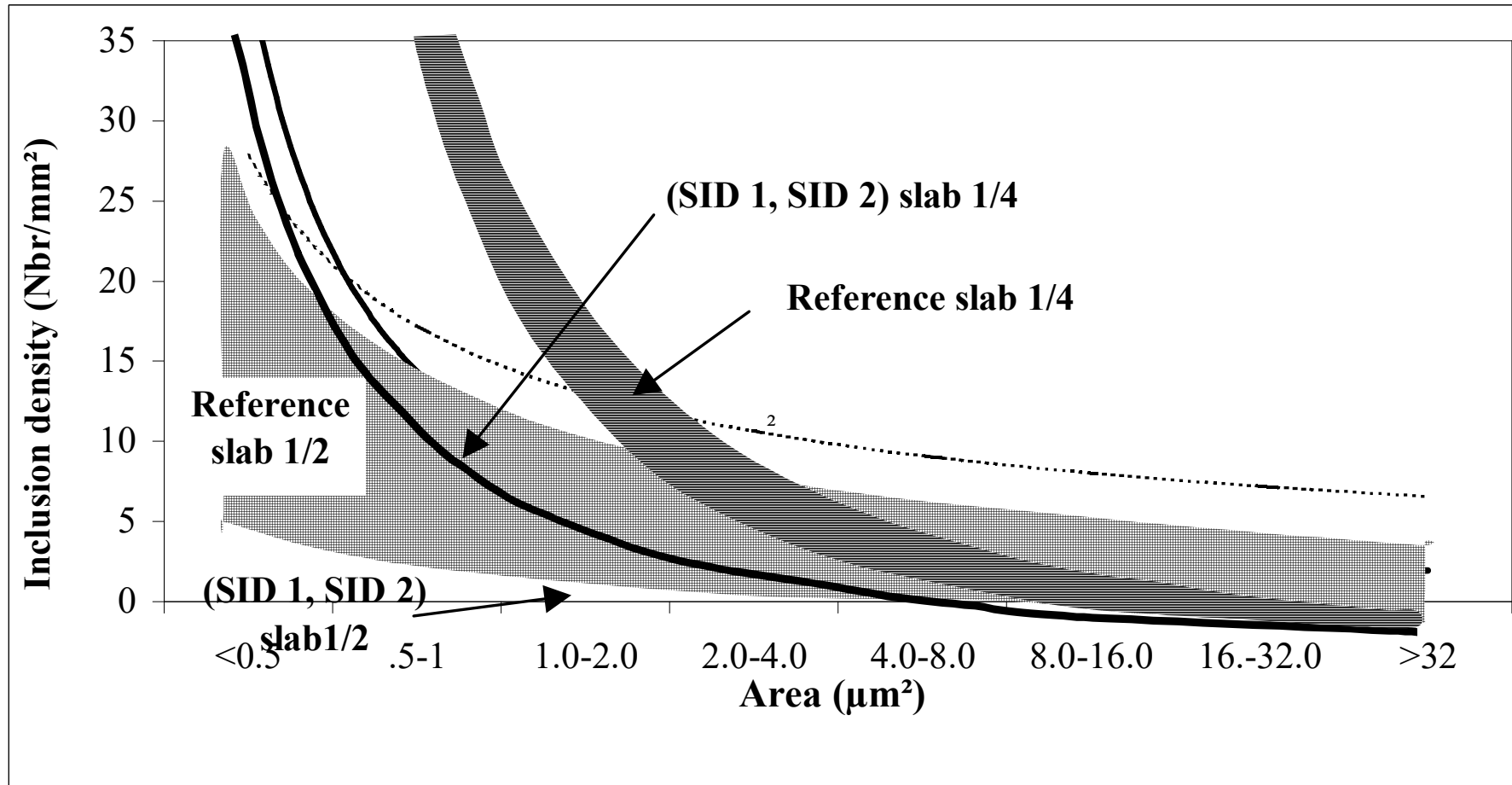
### 3. Control of inclusions in RH degassing processes - SEM procedure use



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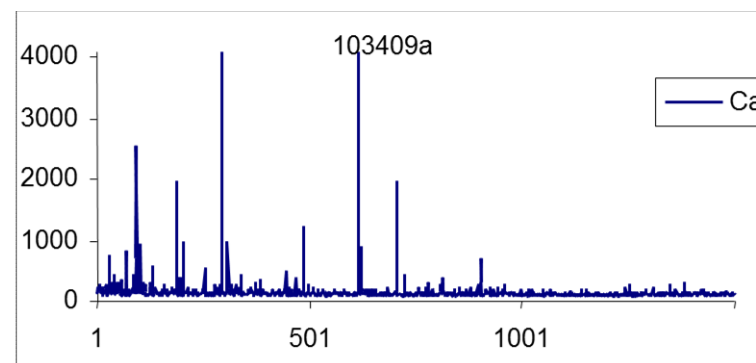
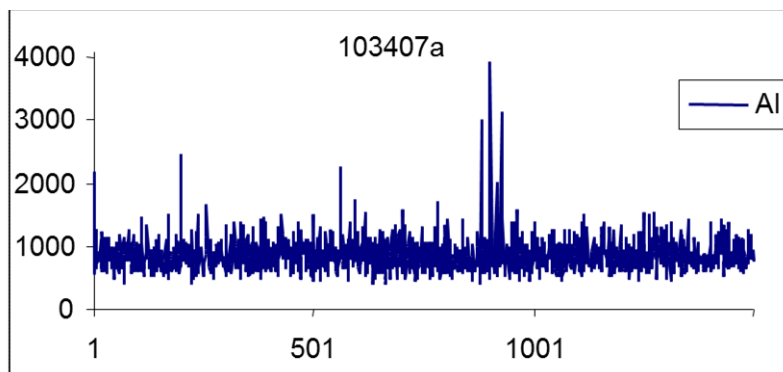
### 3. Control of inclusions in RH degassing processes - SEM procedure results



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### 3. Control of inclusions in RH degassing processes - PDAOES methodology

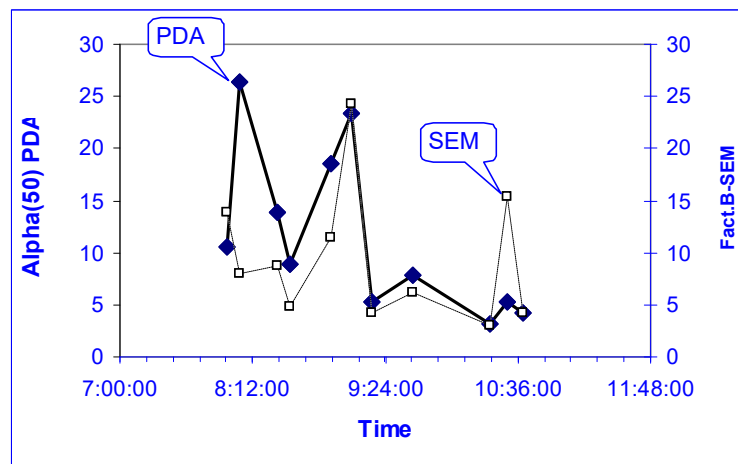
- ⌘ Measurement method is based on emission spectrometry with spark discrimination.
- ⌘ For each element, the pulse by pulse screening make possible the recording of emission intensity.
- ⌘ Because of the high concentration of elements included in inclusions, the variation in the pulse height is particularly marked for discharges adjacent to non-metallic inclusions. Consequently, it is possible to characterise the precipitation content.
- ⌘ The software used to deconvolute the intensity raw data is mainly based on the numbering of “abnormal” sparks intensities.



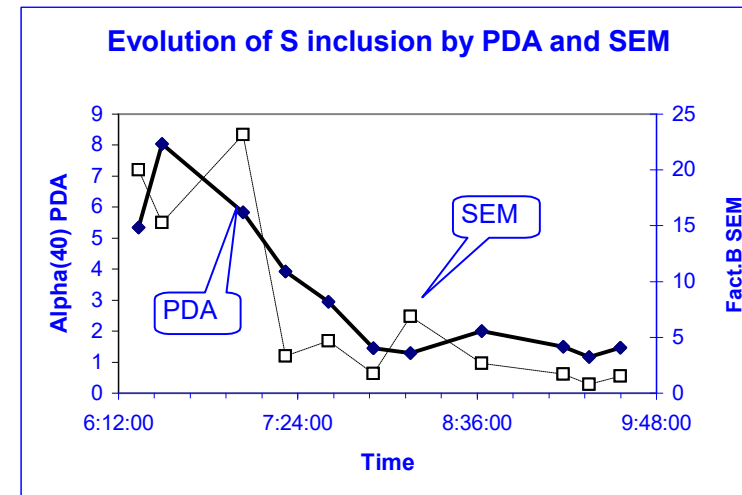
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### 3. Control of inclusions in RH degassing processes - SEM / PDAOES comparison

- ⌘ Good coherence of the relationship obtained between PDA signal perturbation of aluminium, calcium oxide and sulfur and the factor B determined by SEM, opening a possibility with the PDAOES method to develop a fast in line assessment of the steel cleanness.



Al inclusions



S inclusions

### 3. Control of inclusions in RH degassing processes - Conclusions

- ⌘ It is possible to take into account several of the physical mechanisms simultaneously in one numerical computation in order to account for coupled physical effects but when applied to 3D set-up the CPU time was (at that time) exorbitant.
- ⌘ It is possible to reduce significantly the sulphur and the nitrogen content of the steel melt by adjusting the injection conditions of a reagent mixture.
- ⌘ PDA-OES technique could provide an opportunity to assess in real time the inclusions content of samples extracted during the process but due to possible microstructure and particle diameter effects, the effective possibilities of the PDA methodology still need to be better defined.

## 4. In-line assessment of steel cleanness during the secondary steelmaking processes

- ⌘ Working period: 01/07/2001 - 31/12/2004
- ⌘ Consortium : ArcelorMittal, Ascometal/CREAS, CRM, Sidenor
- ⌘ Objectives :
  - ⌘ Development of a special device for the simultaneous sampling from the shallow top slag and underlying liquid steel during the RH process,
  - ⌘ Development of a statistical model and simulation to improve traditional process results,
  - ⌘ The definition of a cleanness index aimed to predict the quality at different step of the process and of the final products.
- ⌘ Main results :
  - ⌘ Development of new sampling method.
  - ⌘ Tool for steel cleanness in-line assessment during secondary steelmaking process based on PDA-OES measurement.
  - ⌘ Preindustrial demonstration, in real time, during one shift.

## 4. In-line assessment of steel cleanness during the secondary steelmaking processes - Background

### ⌘ Various grades of steel

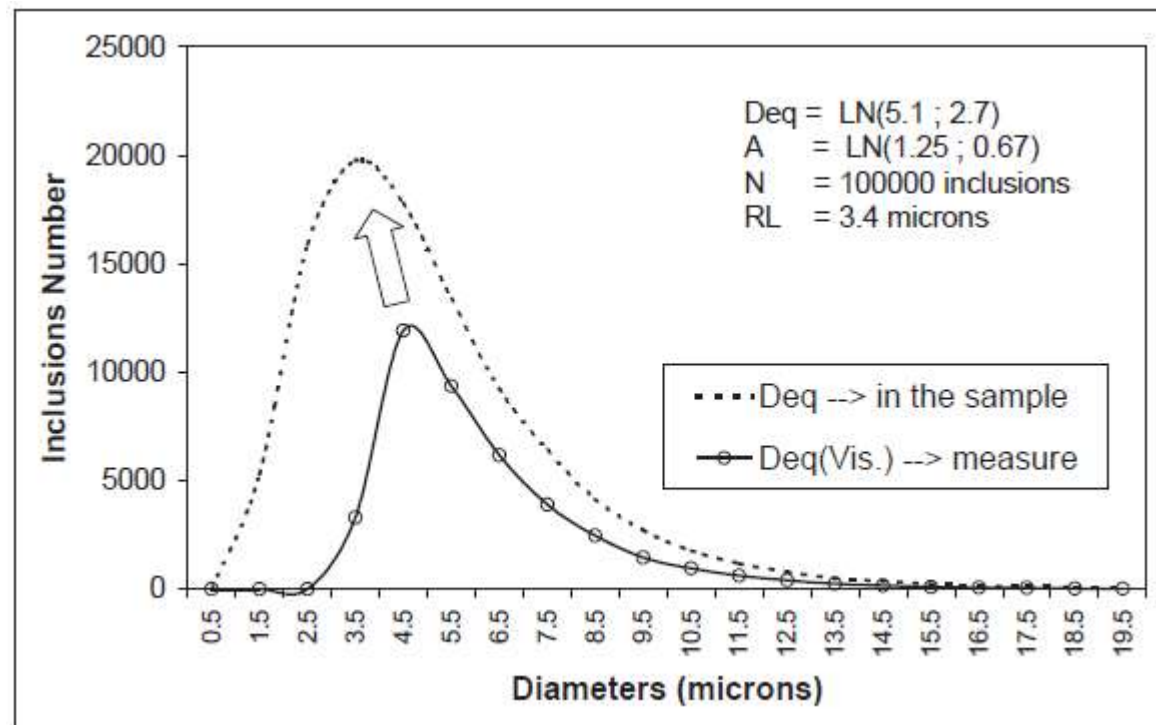
Origin	Grade	Sample	Chemical analysis																
			C	Si	Mn	P	S	Cu	Ni	Cr	Mo	V	Ti	Al	As	Sn	Nb	B	Co
Sidenor	57SiCr6D	wire 35 mm	558	1308	647	11	2	122	86	621	32	4	29	6	5	7	3	<1	6
CRM	3613	flat 4 mm	2	5	109	11	8	16	21	20	2	3	64	31	2	1	<1	<1	3
Ascometal	100Cr6	bar 80 mm	942	223	326	7	6	197	111	1444	31	3	1	25	8	11	2	<1	8

The chemical analyses were obtained on a spectrometer. The unit of the data is 0.001.

- ⌘ At the starting of the research, a lot of developments have been already carried out in the field if PDAOES applied to the cleanness determination.
- ⌘ According to the measure conditions a manual method, with a control threshold, and a manual selection of the inclusions are necessary.

## 4. In-line assessment of steel cleanliness during the secondary steelmaking processes - Background

- ⌘ A model was developed to determine the real size of inclusion population according to the resolution limit and cutting factor:

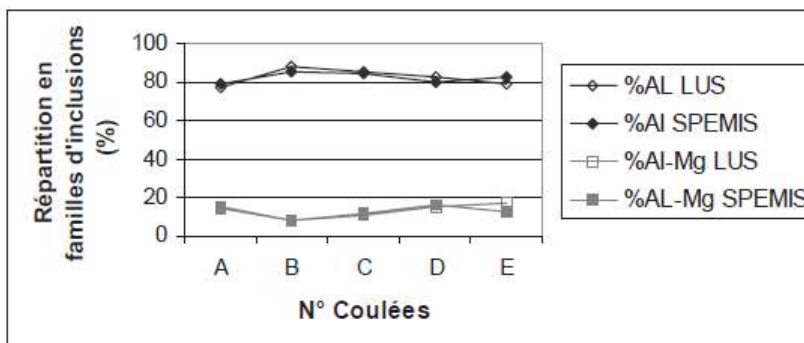
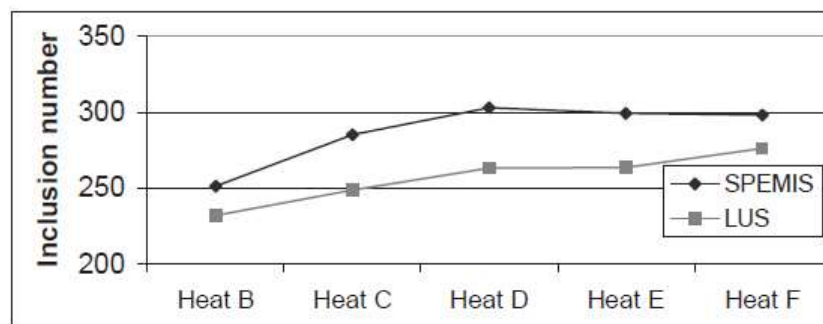


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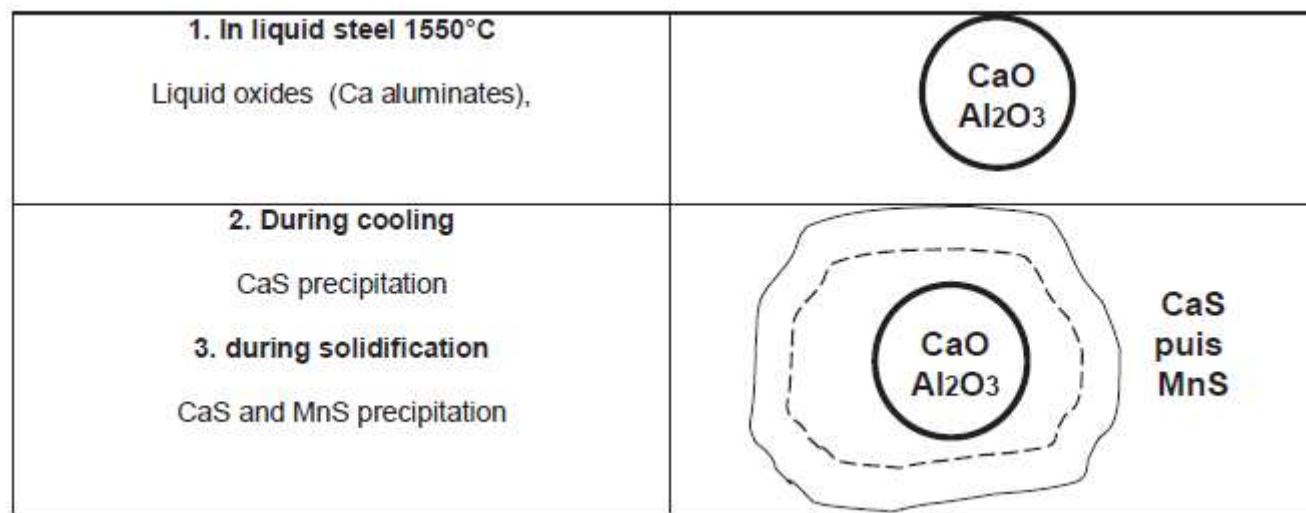
## 4. In-line assessment of steel cleanliness during the secondary steelmaking processes - Steel samplers

- ⌘ PDA-OES analysis is able to detect the number and nature of inclusions such as alumina, Al-Mg, Al-Ca, Al-Mg-Ca etc.
- ⌘ The LUS samples are generally exclusively used for specific trials. For routine production control (automatic preparation), the lollipop sample is used.
- ⌘ The classification of heats by cleanliness order for both LUS and lollipop samples shows an equivalent classification
- ⌘ The number of detected inclusions on LUS samples is systematically less than measured on lollipop samples
- ⌘ Lollipop samples can be used to characterise cleanliness by PDA-OES



## 4. In-line assessment of steel cleanness during the secondary steelmaking processes - Steel samplers, cooling impact

- ⌘ Fast cooling of sample permits to know the real oxide composition of liquid steel.
- ⌘ Low cooling speed gives a correct idea of the nature of inclusions in the final product and of its cleanness



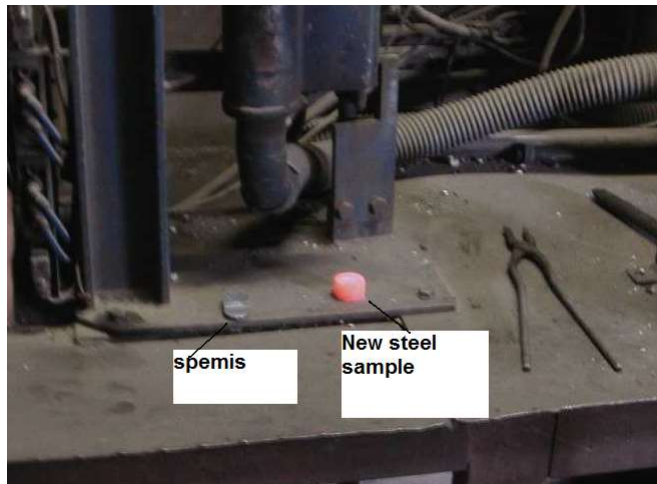
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## 4. In-line assessment of steel cleanness during the secondary steelmaking processes - combined sampling

- ⌘ New sampling technique was developed with aim to allow a fast slag analysis (+/-3 min) on the plant floor. The natural first step to this development is to devise a method for obtaining directly glassy bead taken simultaneously with the steel samples.
- ⌘ Sampling procedure have been carried out at various steps of the RH process (before the vacuum treatment, after the decarburisation and after the addition of Ferro-alloys) as well as on continuous casting.
- ⌘ Samples, corresponding to the previous heats, have also been cut after Hot Rolling process in order to qualify the final products.
- ⌘ Steel samples gave homogeneous and reproducible measurements allowing to monitor the evolution of the inclusion density and composition.
- ⌘ At the opposite, it seems very difficult to obtain useful information from slag analysis for defining a process cleanness Index.

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#### 4. In-line assessment of steel cleanliness during the secondary steelmaking processes - combined sampling



Steel and slag samples

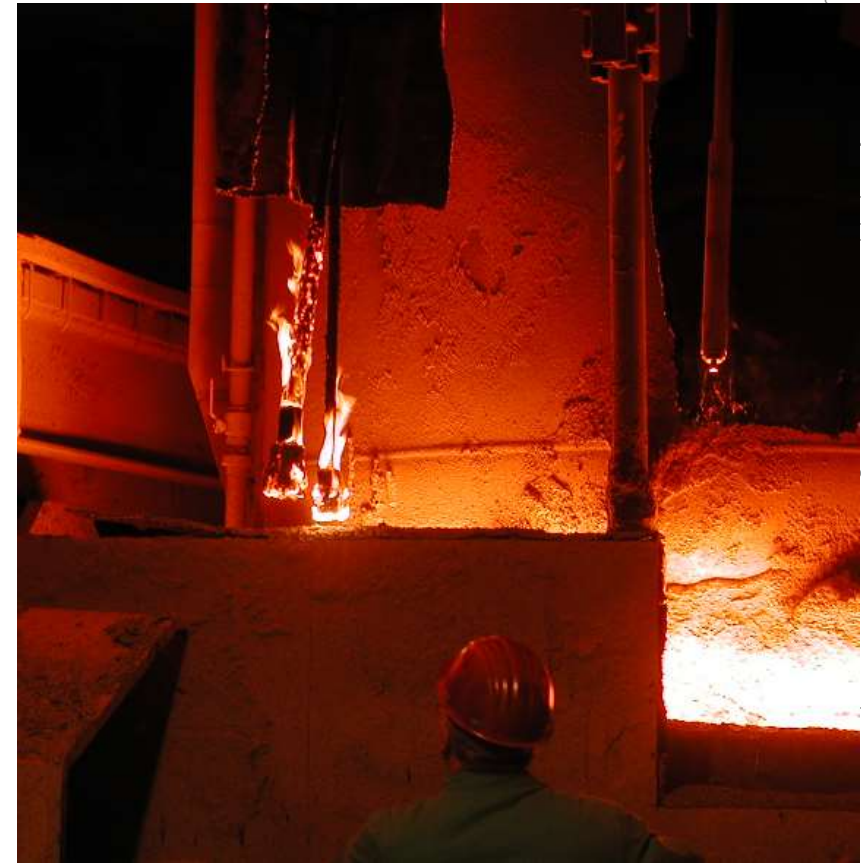
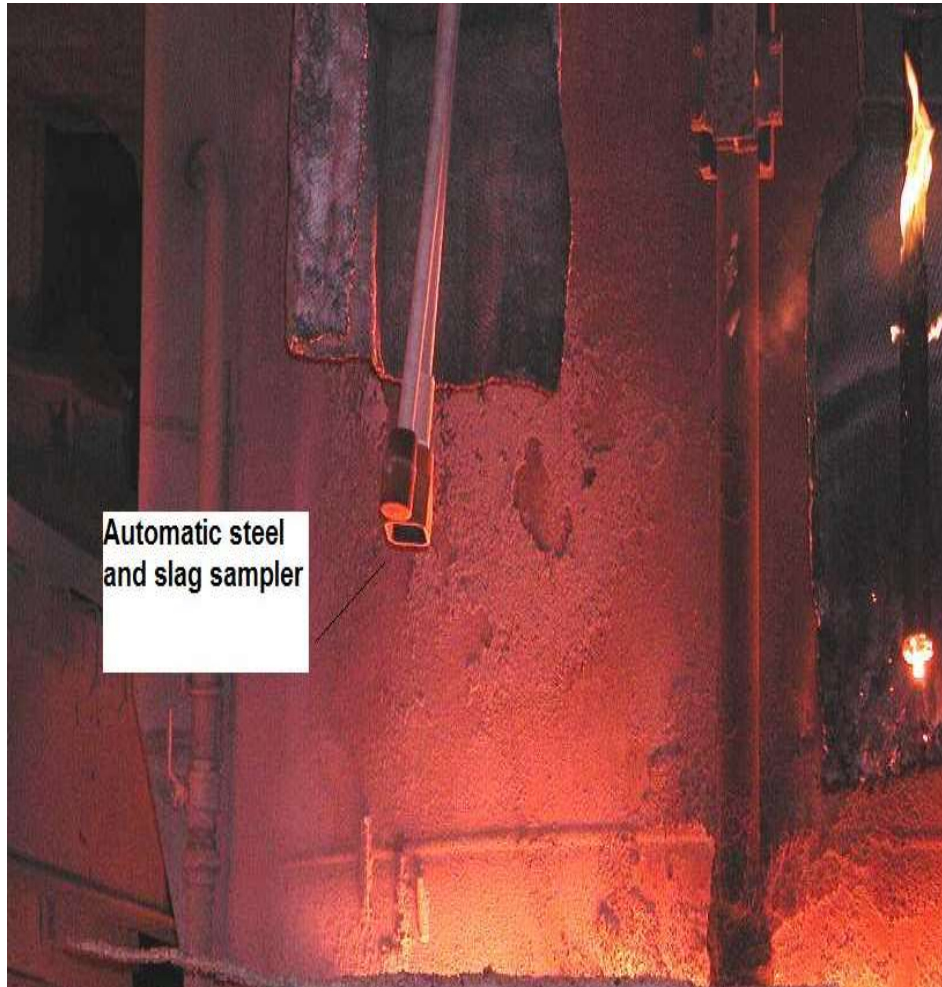


Combined steel and slag sampler

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#### 4. In-line assessment of steel cleanness during the secondary steelmaking processes - combined sampling



Automatic slag and steel sampling

## 4. In-line assessment of steel cleanness during the secondary steelmaking processes - Statistic & modelling with PDAOES

- ⌘ The three different steel grades were characterised by PDA-OES.
- ⌘ Possible chemical inclusion compositions were taken into account.
- ⌘ A peak with an intensity greater than the limit level corresponding to the matrix peak intensity is considered to be an inclusion.
- ⌘ Results obtained by PDA-OES were compared with image analysis results for the same samples. Significant differences (not always great) were found. These differences could be explained by:
  - ⌘ The size of the analysed volume
  - ⌘ Diameter and density measurement errors
  - ⌘ Inclusion dispersion in the sample is not homogeneous
  - ⌘ The detection limit might be incorrect
  - ⌘ One peak is not always one inclusion and one inclusion on the analysed area is not always one peak
  - ⌘ The number of sparks (5) is insufficient
  - ⌘ The spark conditions on the spectrometer
- ⌘ It appears that PDA-OES can be effective when the inclusion density is ranging from 0.5 to 5 /mm<sup>2</sup>.

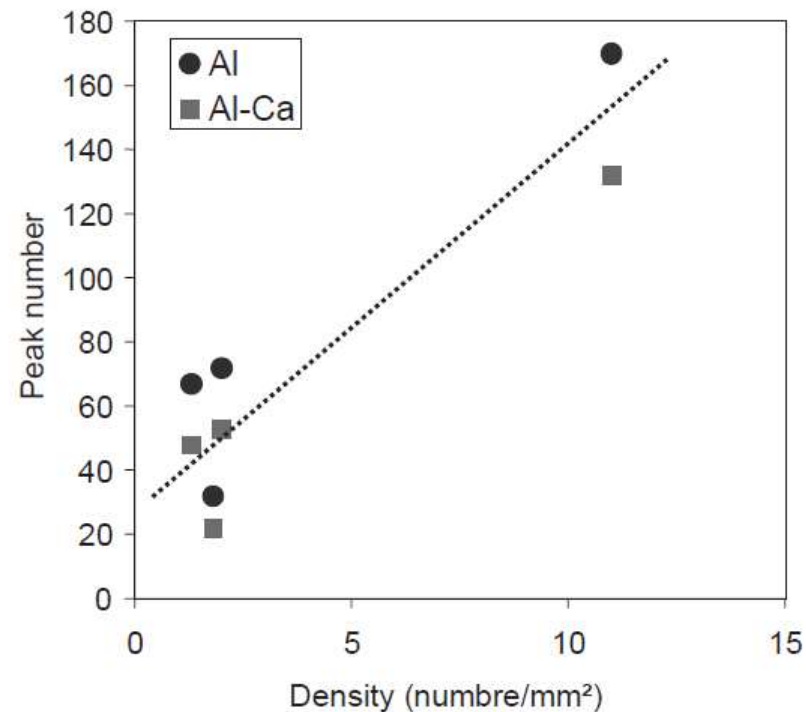
## 4. In-line assessment of steel cleanness during the secondary steelmaking processes - Assessment of cleanness index

- ⌘ When considering the final product quality, from the cleanness point of view, care must be taken.
  - ⌘ Depending on the final product (flat or round), inhomogeneity in inclusion content can be observed.
  - ⌘ For certain flat products, an inclusion content gradient has been observed as a function of the depth of the analysed surface.
  - ⌘ Even more, comparison of results on final product obtained by classical methods (IA) and by PDA-OES differs.
- ⌘ All these facts makes difficult to predict a cleanness value for the final product from the liquid steel sampling.
- ⌘ But proposed methodology allows quickly evaluating the effectiveness of the secondary metallurgy operations regarding inclusion removal from the liquid steel, and thus, to early detecting if the final product is susceptible of presenting cleanness problems.



## 4. In-line assessment of steel cleanness during the secondary steelmaking processes - Assessment of cleanness index

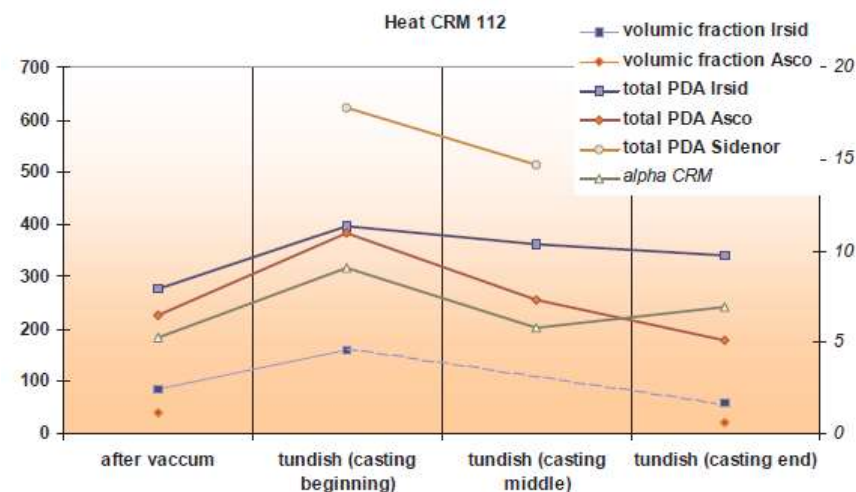
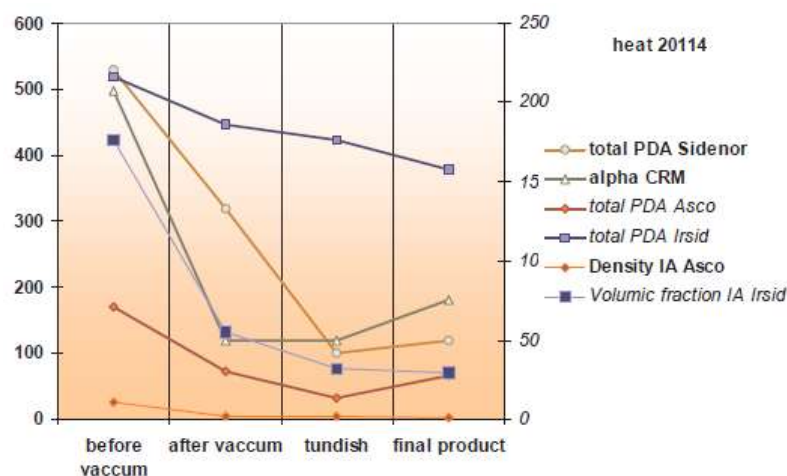
- ⌘ Inclusion density has been measured by image analysis on several lollipop samples taken at different process steps and compared with PDA-OES measurements.
- ⌘ Correlation was fairly good.



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## 4. In-line assessment of steel cleanness during the secondary steelmaking processes - Conclusion: Assessment of cleanness index

- ⌘ Two PDA based indexes (total inclusion peaks and alpha factor) which are able to qualify the cleanness along the process have been developed
- ⌘ These indexes have the same evolution and are in agreement with the expected cleanness along the process
- ⌘ Indexes have similar variations as the density or volume fraction measured by image analysis
- ⌘ These indexes allow some control of the process but do not predict the final product cleanness



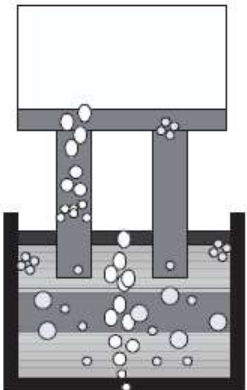
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## 5. Improvement of inclusion flotation during RH treatment

- ⌘ Working period: 01/07/2001 - 31/12/2004
- ⌘ Consortium : ArcelorMittal, BFI, CSM, RWTH
- ⌘ Objectives :
  - ⌘ To develop a validated model of prediction of the evolution of the inclusion population during RH treatment,
  - ⌘ To evaluate the effect of ladle slag composition on the capture of inclusions,
  - ⌘ To propose an optimised flotation process at the RH unit.
- ⌘ Main results :
  - ⌘ Various models taking into account the important features of the process.
  - ⌘ Recommendation for optimised process.

## 5. Improvement of inclusion flotation during RH treatment - Framework

- ⌘ 2 types of steel grade:
  - ⌘ a stainless steel grade, de-oxidized with Si-Mn, with a relatively low initial content of inclusions. These inclusions are mainly liquid,
  - ⌘ an Al-killed carbon grade, with a relatively high initial content of solid inclusions, that tend to cluster.

Inputs	Industrial assessment Water modeling Numerical simulation Mathematical model	Outputs
RH configuration and lift gas injection Inclusion types: small oxides, clusters, large oxides Bottom ladle bubbling Boundary conditions for inclusion entrapment Clustering laws		Main driving phenomena Best circulation rate Best treatment time Inclusion final population Kinetics of inclusion removal Use or not of ladle bottom bubbling Impact of slag layer

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## 5. Improvement of inclusion flotation during RH treatment - approaches

Parameters	Tools	Partner	Specifications	Variations / remarks
Flow visualization	Water model	BFI	Color injection, video recording	Variation of melt circulation rate and ladle bottom plug
Multiphase behavior	Water model	BFI	Two phase PIV	Principle feasibility
Circulation rate	Numerical model	Arcelor Res.		Vary lift gas flow rate
	Water model	BFI	LDA measurement	Variation of lift gas nozzle number and lift gas flow rate
Mixing in RH	Water model	RWTH-IEHK	Steady-state, focus on vacuum vessel	Effect of level of liquid metal in the vacuum vessel
Flow field	Numerical model	CSM	3D, steady state	Only ladle
		BFI	3D, steady state	Water model and plant set-up
		Arcelor Res.	3D	Vary lift gas flow rate to get turbulence par. in sub-boxes
Mixing behavior	Water model	BFI	Color concentration probes	Variation of melt circulation rate and ladle bottom plug
Alloying and mixing	Numerical model	BFI	3D, time-dependent	Variation of ladle bottom plug (plant)
Thermal behavior at top slag and ladle wall	Numerical model	BFI	3D, time-dependent	With / without use of ladle bottom plug (plant)
Inclusion separation at top slag only	Water model	BFI CSM	Turbidity probes Qualitative eval.	Variation of circulation rate, ladle bottom plug and stirring gas flow rate, with and without simulated slag
Inclusion separation	Numerical model	BFI	3D, time-dependent	Variation of melt circulation rate and ladle bottom plug (plant)
Inclusion aggregation as cluster and their separation at top slag and ladle and RH wall	Mathematical model	Arcelor Res.	Fractal dimension of cluster; multi sub-reactor model of aggregation	With variable boundary condition at ladle slag and wall, variable initial oxygen content and 2 RH configurations
	Mathematical model	RWTH-IEHK	One-reactor simplified model of aggregation	Supposed exponential time-decrease of inclusion content
Agglomeration of liquid inclusion and their separation	Mathematical model	CSM	One-reactor simplified model of aggregation	Low content of large-size inclusions
Inclusion separation in real RH	Industrial data	Arcelor Res.	Time-dependent	With variable ladle slag, initial oxygen content and 2 RH configurations Al de-oxidized steels
	Industrial data	CSM	Time-dependent	Si-Mn de-oxidized steels

Fluid flow

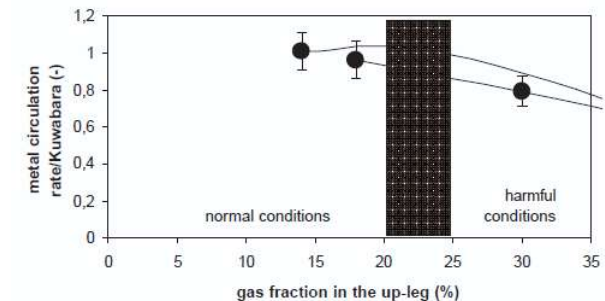
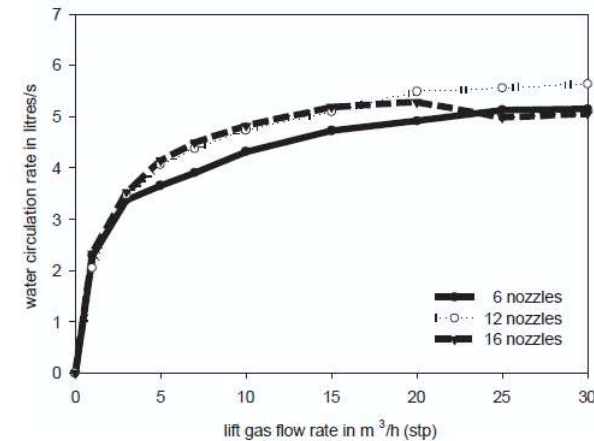
Mixing alloying

Inclusion separation rate

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## 5. Improvement of inclusion flotation during RH treatment - Fluid flow

- ⌘ Experimental model trials using non-intrusive Laser Doppler Anemometry measurements (LDA) have been carried out in order to investigate the influence of varying the total lift gas nozzle number as well as the lift gas flow rate.
- ⌘ An increased melt circulation rate lets expect better RH performance with regard to chemical reactions and homogenization.
- ⌘ Several calculations with the Fluent code were made to calculate the recirculation metal rate in the RH legs.



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## 5. Improvement of inclusion flotation during RH treatment - Alloying time

- ⌘ Flow visualization trials and mixing-time measurements have been performed in the physical model, along with numerical time-dependent mixing behaviour simulation computations for the full 3D set-up of the industrial RH plant.
- ⌘ Both experimental and numerical approaches show that the use of a plug in the ladle helps to minimize the mixing-time after alloy addition, as compared to the conventional RH set-up. With both approaches, an improvement of about 10 % has been determined. But this also increase snorkel wear.

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## 5. Improvement of inclusion flotation during RH treatment - Non-metallic inclusion separation behaviour

- ⌘ The modelling of inclusion separation was performed through the following approaches:
  - ⌘ A first model supposing an exponential time-decrease of inclusion content. Effect of metal height in the vacuum vessel on inclusion floatation,
  - ⌘ Numerical simulations (CFD) without inclusion aggregation: effect of slag and metal circulation rate,
  - ⌘ Numerical modelling without inclusion aggregation: exponential time-decrease of inclusion content. Effect of circulation rate and ladle bubbling,
  - ⌘ Numerical and mathematical modelling with inclusion aggregation of low content of large-size inclusions,
  - ⌘ Numerical and mathematical modelling with inclusion aggregation for high content of small inclusions: effect of fractal dimension and entrapment at walls.

## 5. Improvement of inclusion flotation during RH treatment - Conclusion : Recommendation for an optimized process

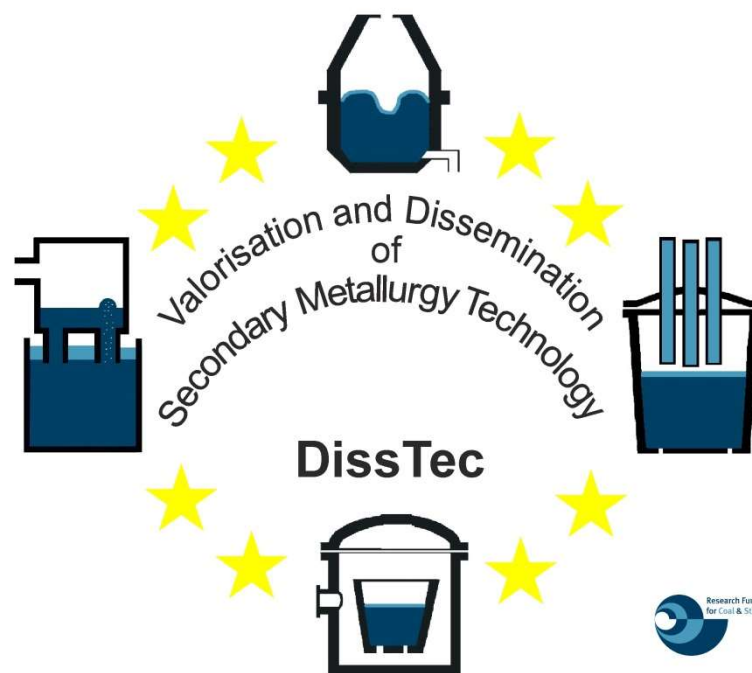
Project final proposal is a two-phases process:

- ⌘ A first intensive metal recirculation for decarburization, alloy melting and mixing and inclusion aggregation,
  - ⌘ High circulation rate to improve inclusion aggregation and entrapment at the walls. This is achieved by using all possible nozzles and by increasing the flow rate. Nevertheless it is useless to inject a too high lift gas flow rate, a maximum 25% gas fraction is enough.
  - ⌘ The additional use of plugs is possible, but does not seem favourable, as operational trials have shown that it also increases the snorkel wear.
  - ⌘ A high enough initial inclusion content ( $> 300$  ppm) is better to promote clustering and inclusion removal.
- ⌘ A second phase favouring inclusion floatation without re-entrainment.
  - ⌘ Melt circulation rate could be limited by reducing the lift gas flow rate, so that a more quiescent melt surface in the ladle is achieved,
  - ⌘ If plugs have been used in the first treatment step, they could be deactivated.

The use of de-oxidized and liquid ladle slag is of course needed, although it was not possible to confirm precisely optimum composition.

# Conclusion

- ❖ Several European projects dealing with steel cleanliness assessment at RH degasser have been performed,
- ❖ They focused on different topics :
  - ❖ Modelling of the process
  - ❖ Direct enhancement of steel cleanliness
  - ❖ Development of new sampling methods
  - ❖ Development of sample analysis techniques
  - ❖ Calculation of cleanliness indexes to control the process
  - ❖ Proposal of optimised process pattern
- ❖ Experience gained in a project is often valorised in a further one,
- ❖ Some of these development are now state of the art, other have been used in the specific pilot plants but none were useless or failed.



## Cleanness assessment of high quality steels produced by RH treatment

PIERRET Jean-Christophe (CRM)

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