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MCL

M2CC METALLURGY • MATERIALS CLEANNESS & CASTING

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RHI

PRIMETALS TECHNOLOGIES

Metallurgie

Untersuchung des Auflöseverhaltens oxidischer Einschlüsse in CaO-Al_2O_3-SiO_2-MgO Schlacken mittels Hochtemperatur-Laser-Scanning Konfokalmikroskopie

S.K. Michelic[1], J.Goriupp[2], S. Feichtinger[3], Y.-B. Kang[4], C.Bernhard[1] and J.Schenk[1]

[1] **Lehrstuhl für Eisen-und Stahlmetallurgie, Montanuniversität Leoben, Österreich**

[2] voestalpine Schienen GmbH, Leoben, Österreich

[3] Swiss Steel AG, Emmenbrücke, Schweiz

[4] Pohang University of Science and Technology, (POSTECH), Pohang, Korea

ALMA MATER LEOBIENSIS

2. November 2015

MONTAN UNIVERSITÄT

FERROUS METALLURGY MONTANUNIVERSITAET

◆ Outline

- ◇ Introduction „Inclusion Metallurgy“
- ◇ HT- Laser-Scanning Confocal Microscopy
- ◇ Inclusion dissolution in slags
  - Experimental procedure and evaluation
  - Examples
- ◇ Summary

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Ferrous Metallurgy: Research

Primary Metallurgy and Metallurgical Processes
(Johannes Schenk, Head of Department and Chair of Metallurgy)

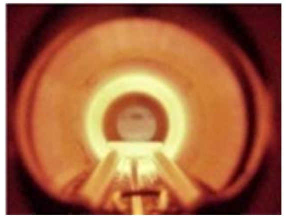
Metallurgy and Materials Science of Steel Cleanness and Casting
(Christian Bernhard)

Facts

2 Professors, 1 Professor emeritus,
1 Assistance Professor, 4 Lecturers,
15 Graduated Researchers
18 Students,
8 Technicians and Office Management

Annual turn-over in research projects 2013: 1.9 Mio. Euro

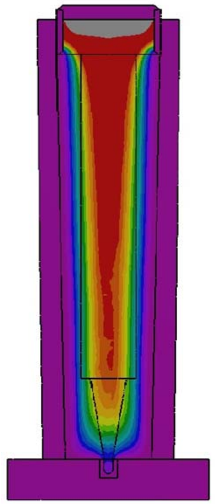
M2CC: Current research fields



- **Inclusion metallurgy**

- Thermodynamics and kinetics of phase transformations in steel

- Defect formation in casting and welding processes



- Computational continuous casting

- Ingot casting and new processes for new steel grades

Inclusion Metallurgy: Research Topics

- **Relation between micro- and meso cleanness**
  - Reaction and interaction within the system steel/slag/refractory
  - Influence of defined alloying elements on the clogging tendency in steels
  - Experimental simulation of the inclusion behavior

- **Specific adjustment of (sub)-micro cleanness**
  - Adjustment of specific inclusion type and size in the steel matrix
  - „Oxide Metallurgy“ – Inclusions as nucleation site for acicular ferrite

- **Further development of characterization methods**
  - Definition of clear limits of detection methods (e.g. size spectrum)
  - Combination of several methods (SEM-EDX, OES-PDA, Extraction)
  - Evaluation and testing of possible new characterization methods

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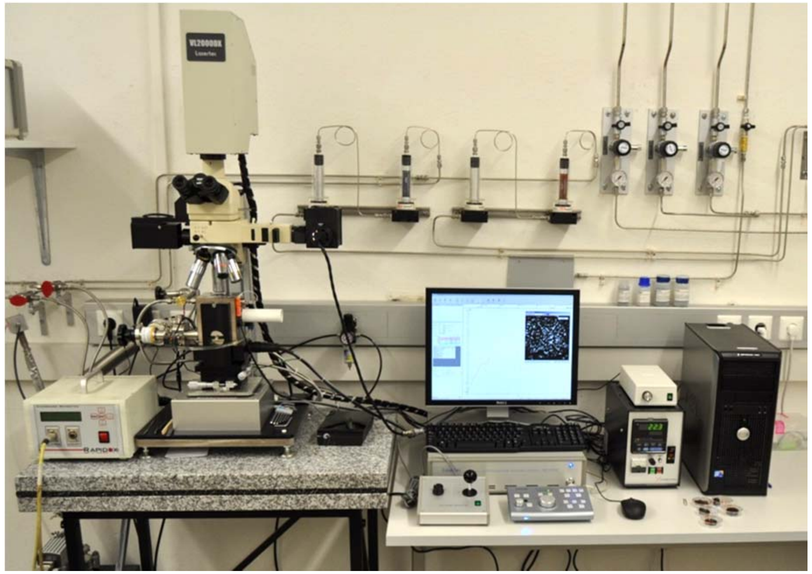
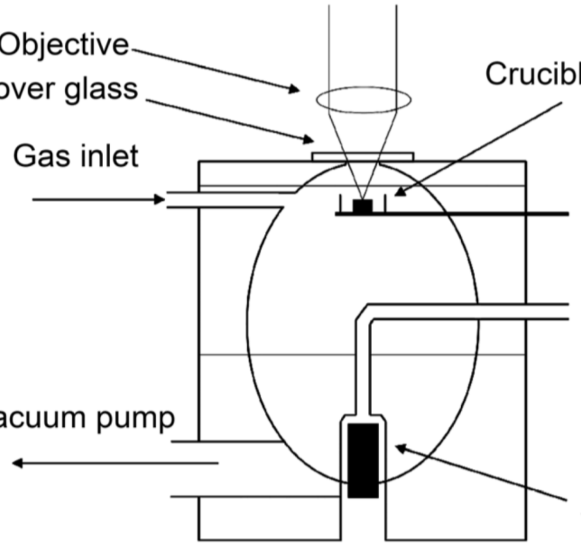
◇ **Summary**

The Method: HT-LSCM

Laser **S**canning **C**onfocal **M**icroscope attached to a **H**igh-**T**emperature Furnace



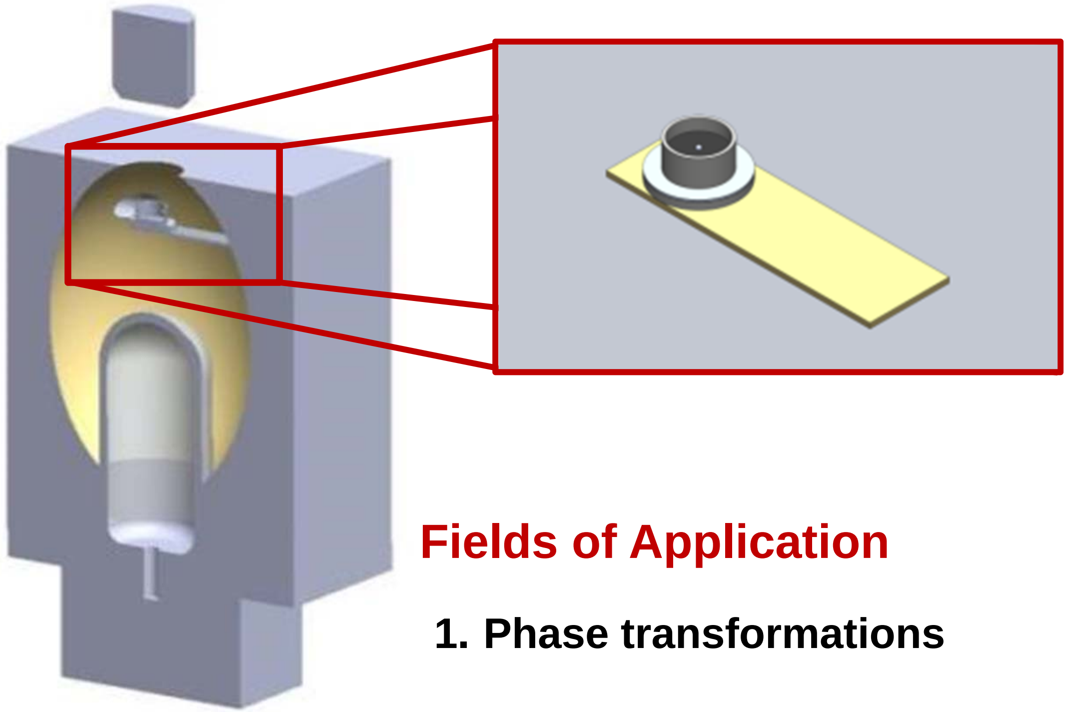


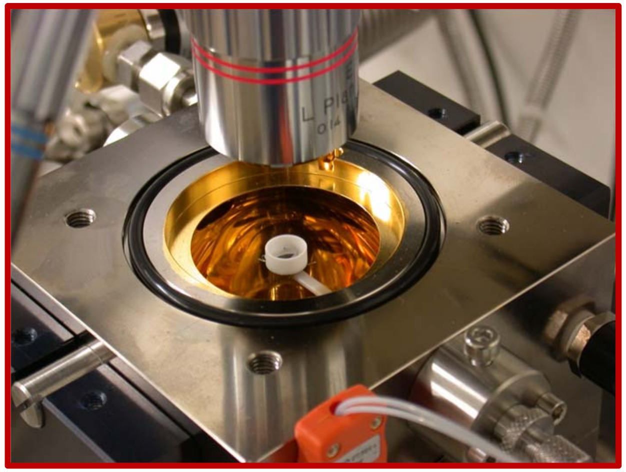
Key data:

- Light source: Laser with a wave length of 408 nm
- Highest temperature is 1700 °C (short time) with a maximum heating rate of 1200 °C/min
- use of oxidizing or reducing atmosphere and vacuum down to 10^{-8} bar possible
- Additional furnace for optimum control of residual oxygen
- Very high scan rates (15 frames/sec at highest resolution)

Source:*Bernhard*, *C.,* et al.:. BHM 156, 5, (2011), 161-167.

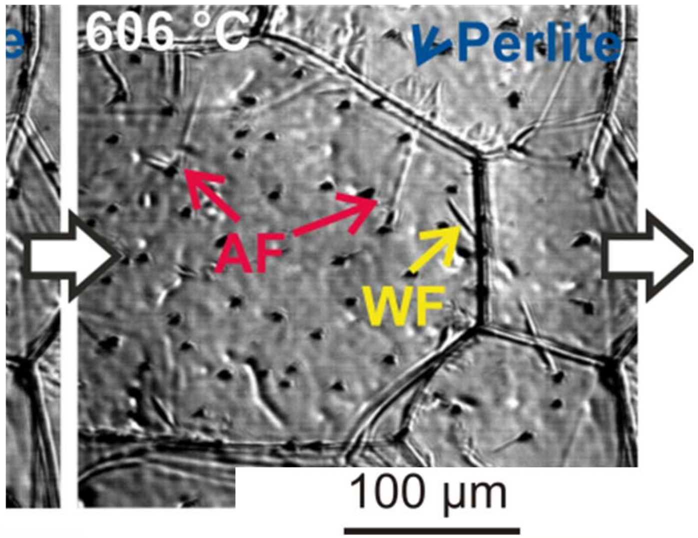
The Method: HT-LSCM





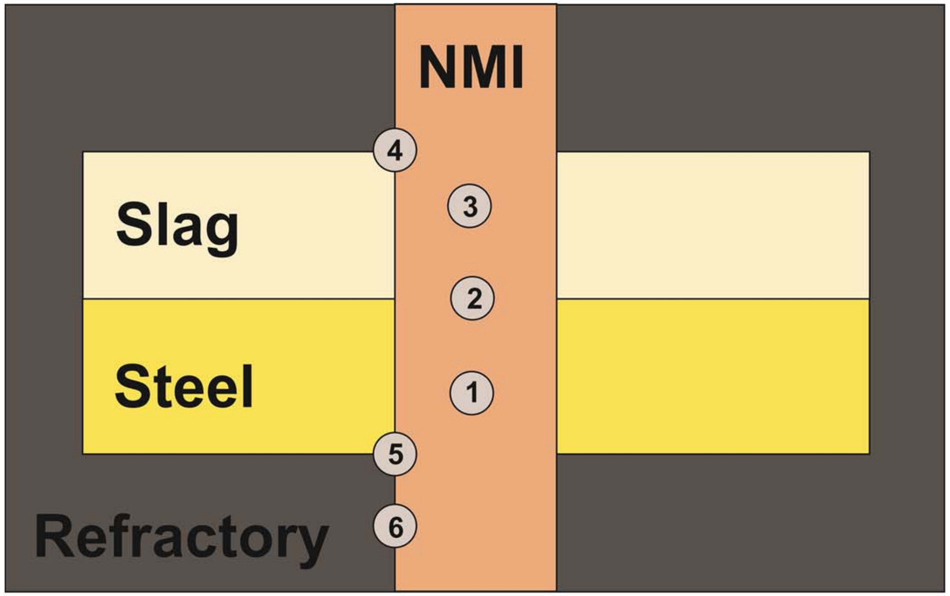
Fields of Application

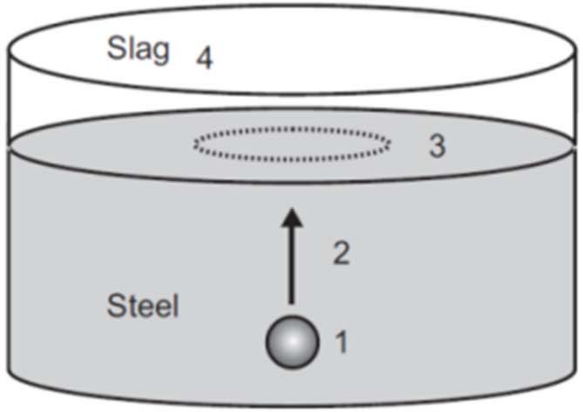
1. Phase transformations
2. Austenite grain growth
3. Reactions of inclusions
in the system steel/slag/refractory



Source: D. Loder et al.: MS&T 2014, Pittsburgh, USA.

HT-LSCM: System steel-slag-refractory

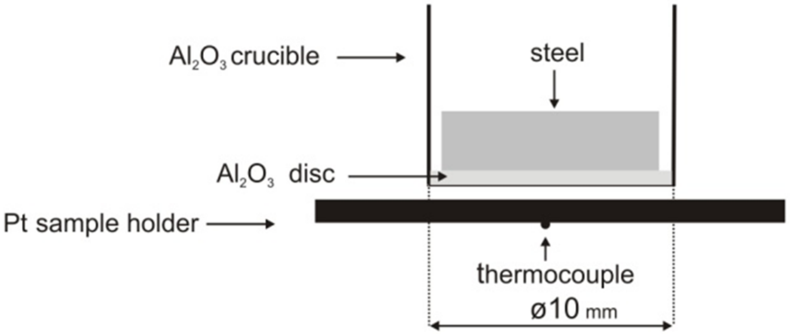


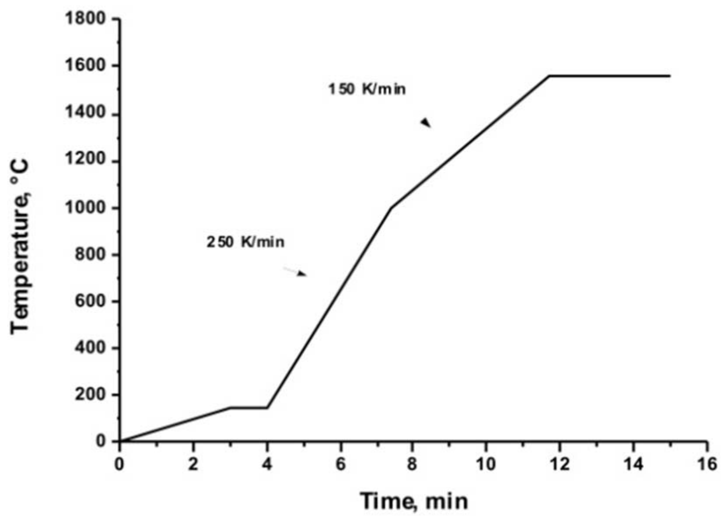


1. Agglomeration of Inclusions in the liquid steel
2. Transportation of the inclusion to the steel-slag interface
3. Inclusion dissolution in the slag
4. Reaction of an inclusion at the slag-refractory interface
5. Reaction of an inclusion at the steel-refractory interface
6. Inclusion deposit on the refractory material

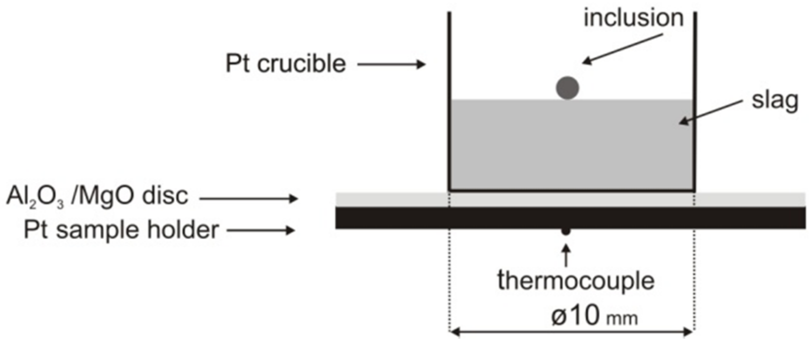
HT-LSCM: Types of Experiments

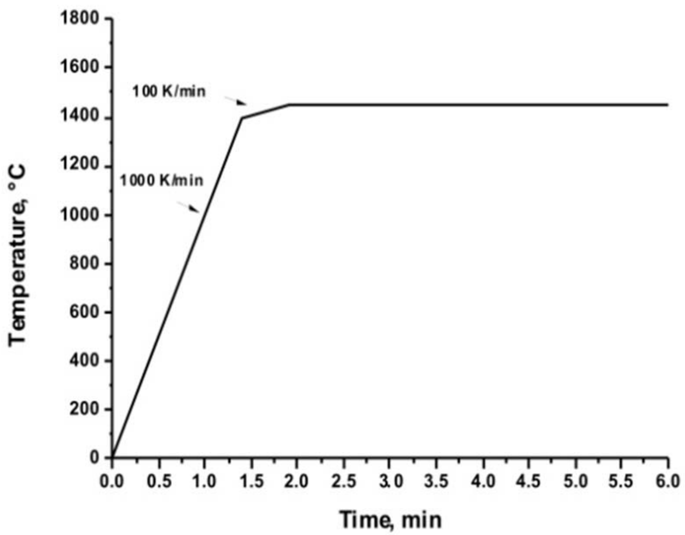
TYPE 1
Inclusion agglomeration in the liquid steel





TYPE 2
Inclusion dissolution in the slag





METALLURGY • MATERIALS CLEANNESS & CASTING

Metallurgie

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Inclusion Dissolution in a Slag

POSTECH GIFT 철강대학원 Graduate Institute of Ferrous Technology

Starting point:

Research cooperation with the Graduate Institute of Ferrous Technology in Korea in 2012

Background: SiO_2 inclusions as a result of Si/Mn deoxidation in order to avoid non-deformable Al_2O_3 inclusions in wire steels for demanding applications (e.g. tire-cord)

journal

J. Am. Ceram. Soc., 1–10 (2013)
DOI: 10.1111/jace.12665

In Situ **Observation of the Dissolution of SiO_2 Particles in $CaO–Al_2O_3–SiO_2$ Slags and Mathematical Analysis of its Dissolution Pattern**

Stefan Feichtinger,[‡] Susanne K. Michelic,[‡] Youn-Bae Kang,[§,†] and Christian Bernhard[‡]

[‡]Chair of Ferrous Metallurgy, Montanuniversitaet Leoben, Leoben A-8700, Austria

[§]Graduate Institute of Ferrous Technology, Pohang University of Science and Technology (POSTECH), Pohang, Kyungbuk 790-784, Korea

Inclusion Dissolution in a Slag

Slag	Inclusion
Chemistry Viscosity Density $T_{Liquidus}$	Chemical Composition Density Size and Shape Purity Crystal structure

1. **Thermodynamic Considerations (**Calculation of c_{sat}, a, η, ρ)
2. **Dissolution Experiment** (incl. slag pretreatment, Temp. Referencing)
3. **Evaluation and Interpretation** (dissolution time and mechanism, Calc. of D)

Required Preconditions

- ◇ perfectly shaped round particles
- ◇ possibly high heating rate
- ◇ particle weight is less than 0.01% of the slag weight
- ◇ Transparent slag
- ◇ Inert atmosphere

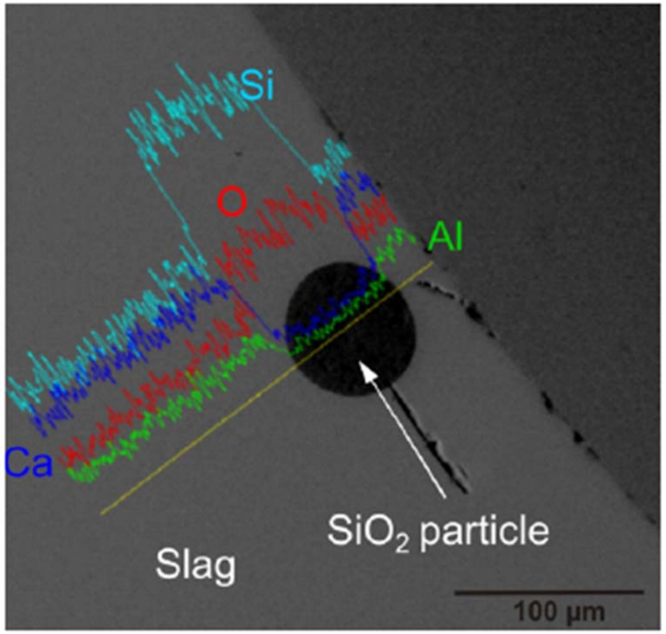


Fig. 4. Micrograph of a SiO_2 spherical particle dissolving into a slag composed of $CaO–SiO_2–Al_2O_3$ at 1450°C and a SEM-EDS line scan showing a qualitative comparison between the contents of Si, O, Ca, and Al.

- ◇ No formation of a reaction layer between slag and inclusions

Source: Feichtinger et al.: Journal of American Ceramic Society, 2014.

Experimental Procedure

Slag pre-treatment

- Mixing of oxide/carbonate powder + premelting in electrical furnace
- Crushing + determination of chemical composition using XRF
- Premelting of slag powder in HAT-LSCM platinum crucibles in electrical furnace
- Premelting and rapid cooling of slag in HT-LSCM

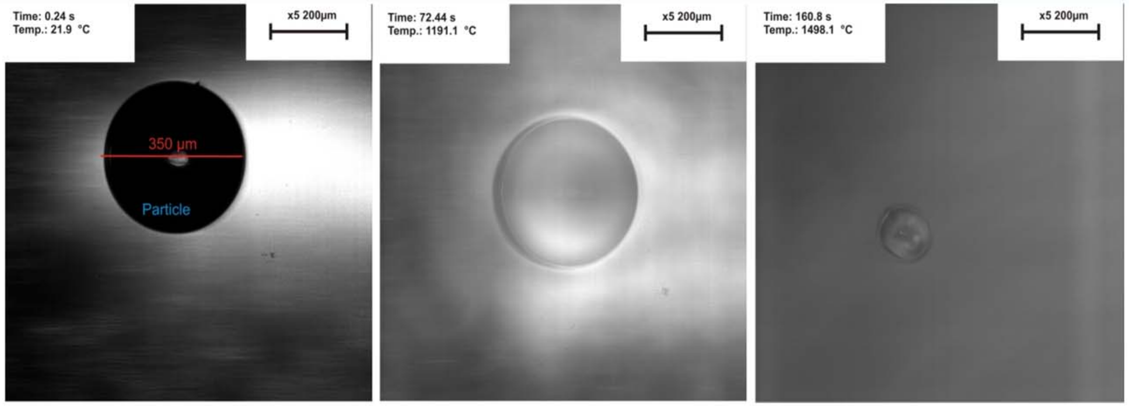
→ **HT-LSCM dissolution experiments**

- Temperature measurement adjustment
- Conducting of dissolution experiments

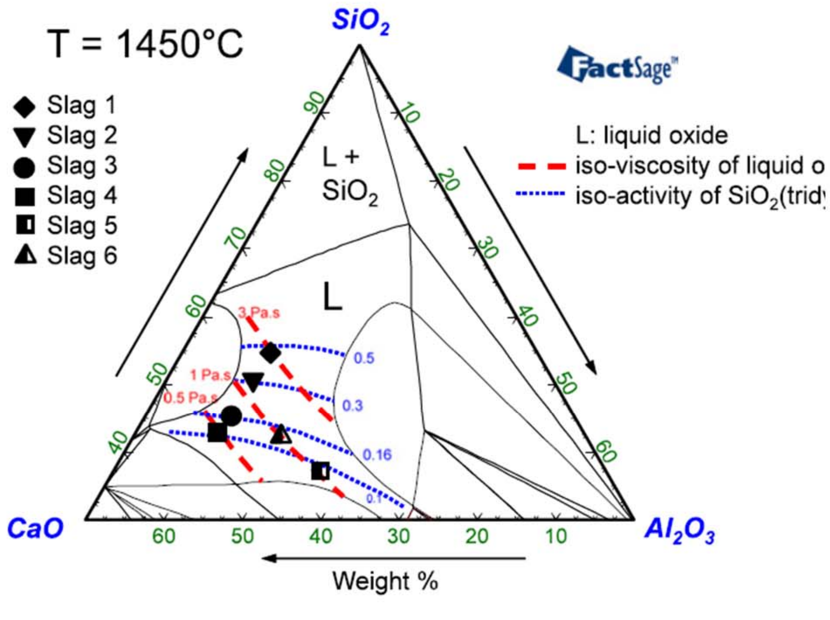
→ **Data Evaluation**

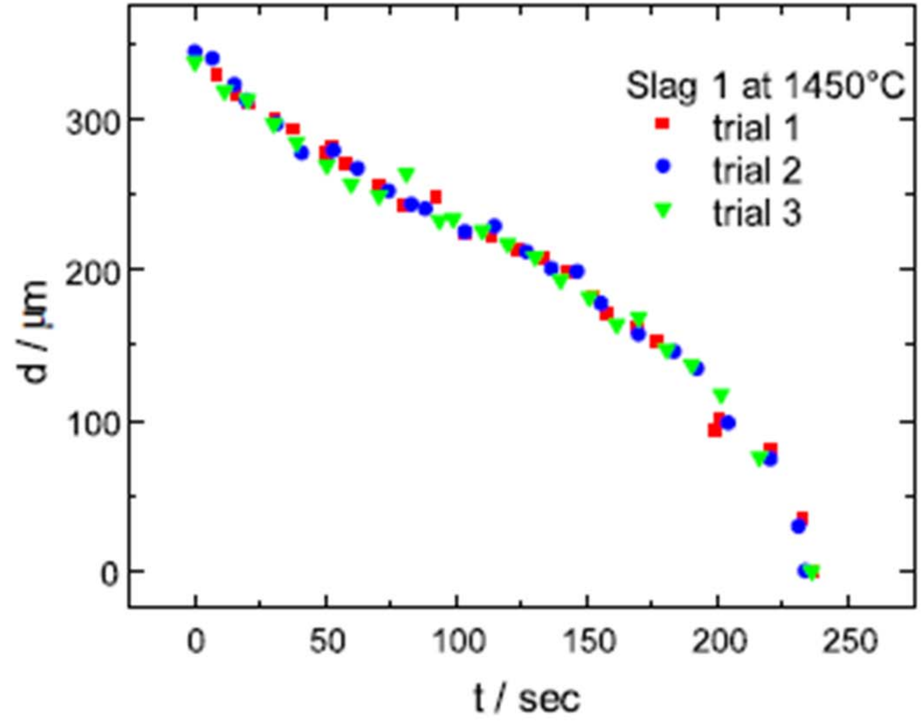
- Extraction of images using VirtualDub
- Measurement of particle size using ImageJ
- Comparison of experimental dissolution diagrams with different models

Data Evaluation



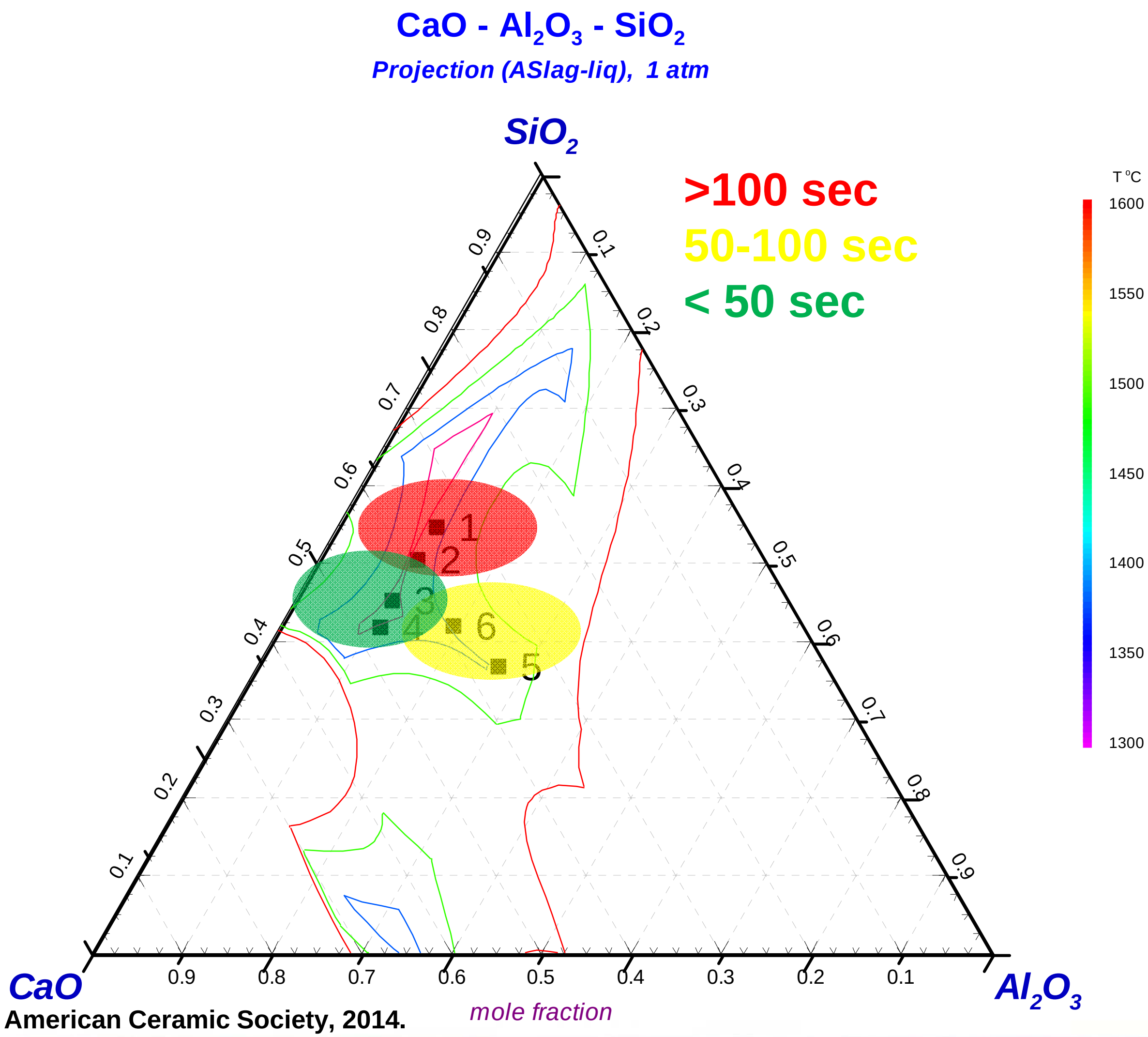
SiO_2 dissolution in CaO-Al_2O_3-SiO_2 slags





Source: Feichtinger et al.: Journal of American Ceramic Society, 2014.

Data Evaluation: SiO_2 dissolution in $CaO-Al_2O_3-SiO_2$ slags



Source: Feichtinger et al.: Journal of American Ceramic Society, 2014.

Interpretation by kinetic models

Analytical Solution

Shrinking Core Model

Reaction Rate Controlled

$$t = \frac{\rho_{incl}}{k \cdot (c_{sat} - c_0)} \cdot (R_0 - R)$$

$$\frac{R}{R_0} = (1 - \frac{t}{\tau})$$

Mass Transfer Controlled

$$t = \frac{\rho_{incl}}{2 \cdot D \cdot (c_{sat} - c_0)} \cdot (R_0{}^2 - R^2)$$

$$\frac{R}{R_0} = (1 - \frac{t}{\tau})^{1/2}$$

Numerical Solution

Diffusion in a stagnant fluid

$$J = -D \cdot \frac{\partial c}{\partial x}$$

Stationary Interface Appr.

$$\frac{dR}{dt} = -\frac{k \cdot D}{R} - k \cdot \sqrt{\frac{D}{\pi \cdot t}}$$

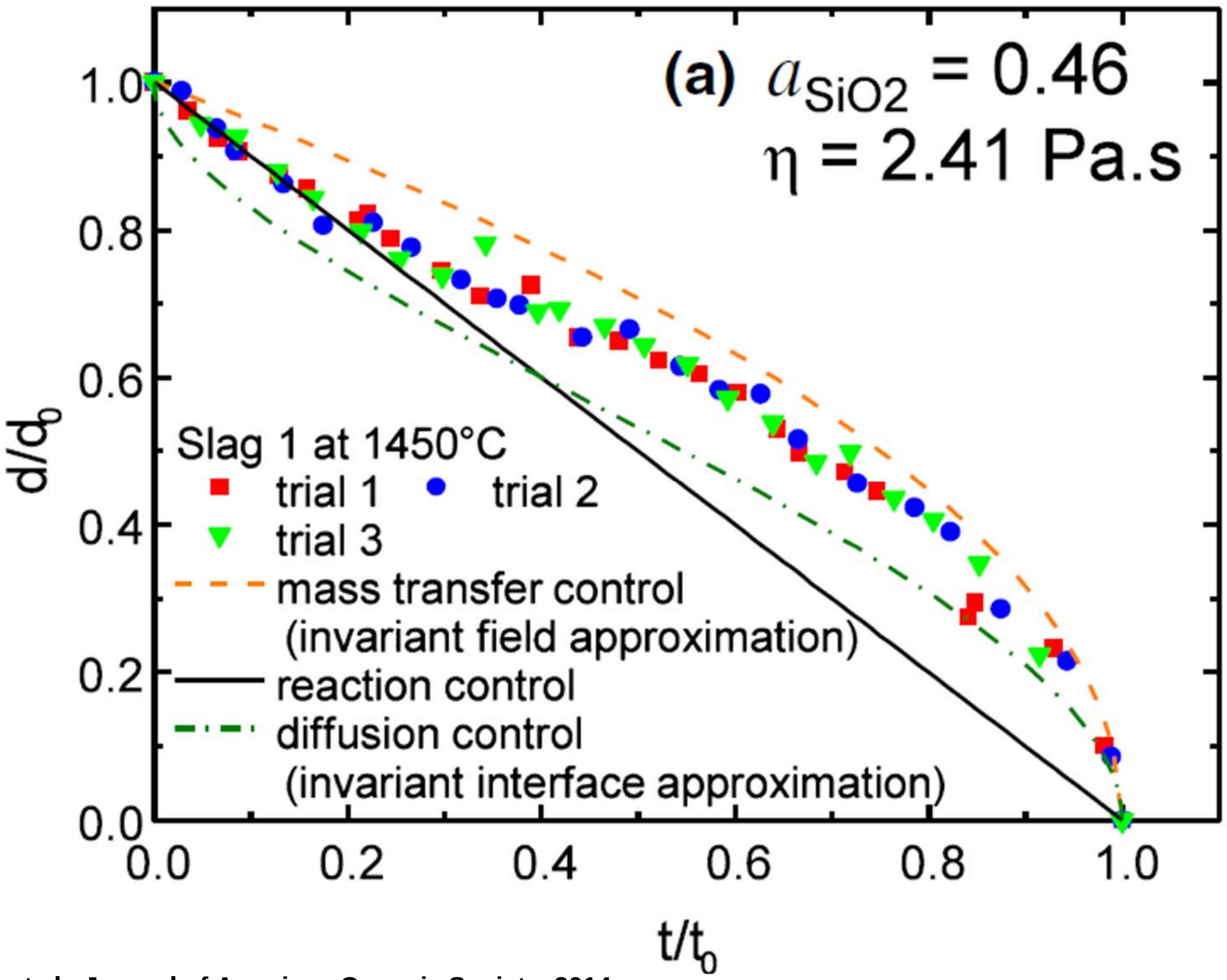
Invariant Field Approximation

$$\frac{dR}{dt} = -\frac{k \cdot D}{R}$$

$$k = \frac{c_{sat} - c_0}{c_{incl} - c_{sat}}$$

Lattice Boltzmann Modeling

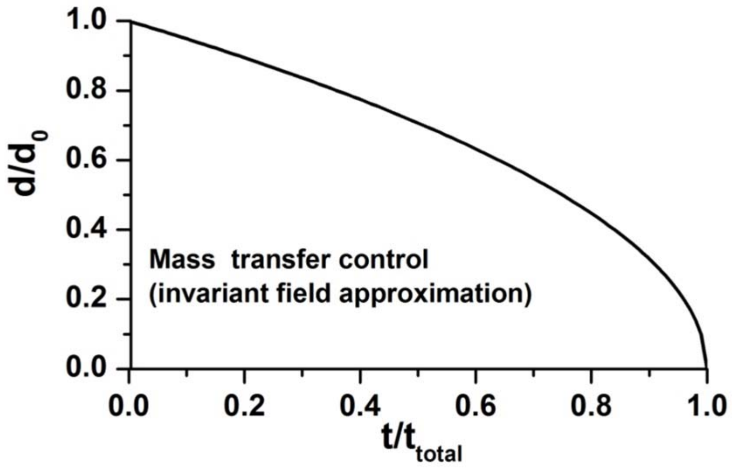
Interpretation: SiO_2 dissolution in CaO-Al_2O_3-SiO_2 slags



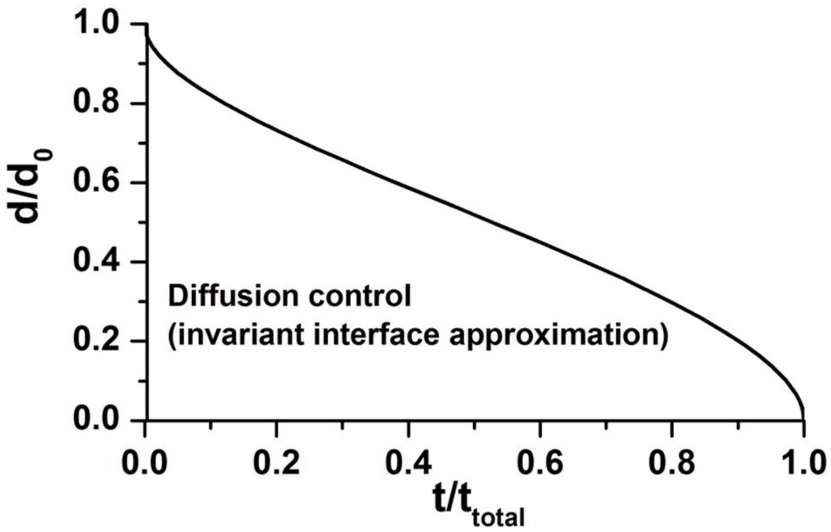
Source: Feichtinger et al.: Journal of American Ceramic Society, 2014.

Interpretation: Modified approach by Feichtinger et al.

$$\frac{\mathrm{d}R}{\mathrm{d}t} = -\frac{k \cdot D}{R} - f \cdot k \cdot \sqrt{\frac{D}{\pi \cdot t}}$$



low slag viscosity – low f-factor

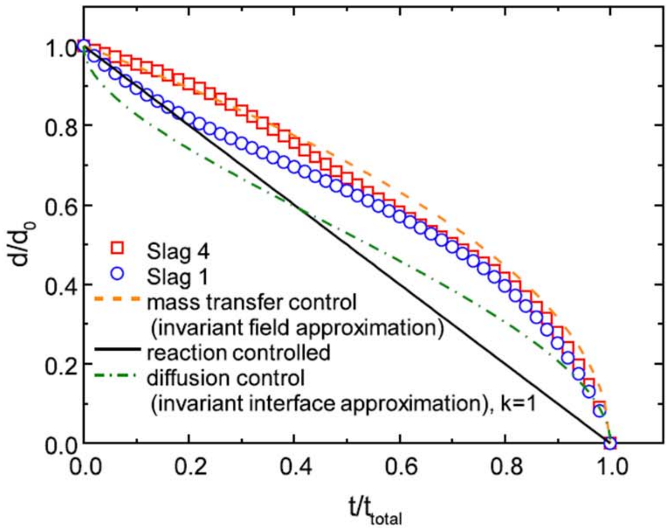


high slag viscosity – high f-factor

Source: Feichtinger et al.: Journal of American Ceramic Society, 2014.

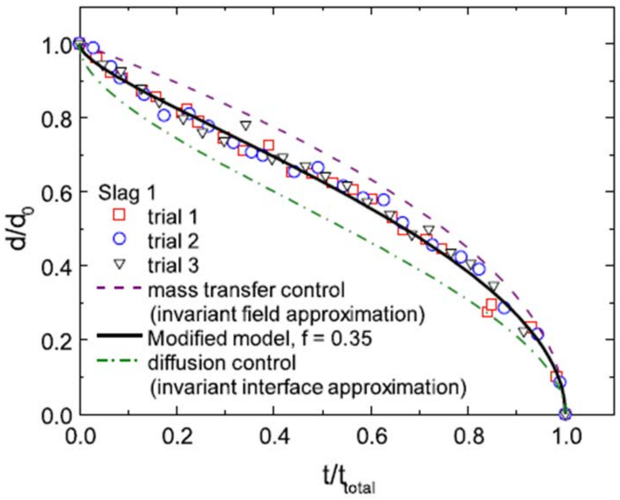
Interpretation: Modified approach by Feichtinger et al.

Common approach

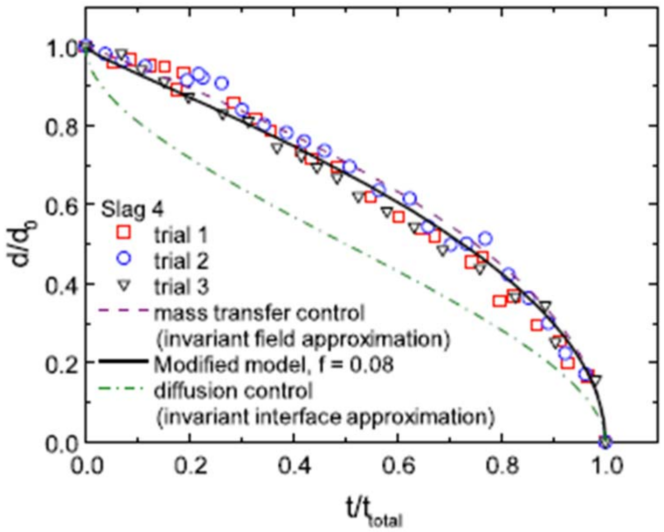


Modified approach

High slag viscosity



Low slag viscosity



Investigated systems up to now

- SiO_2 dissolution in CaO-Al_2O_3-SiO_2 slags
- Al_2O_3 dissolution in CaO-Al_2O_3-SiO_2-MgO slags
- Al_2O_3 and $MgOAl_2O_3$ dissolution in CaO-Al_2O_3-SiO_2-MgO slags

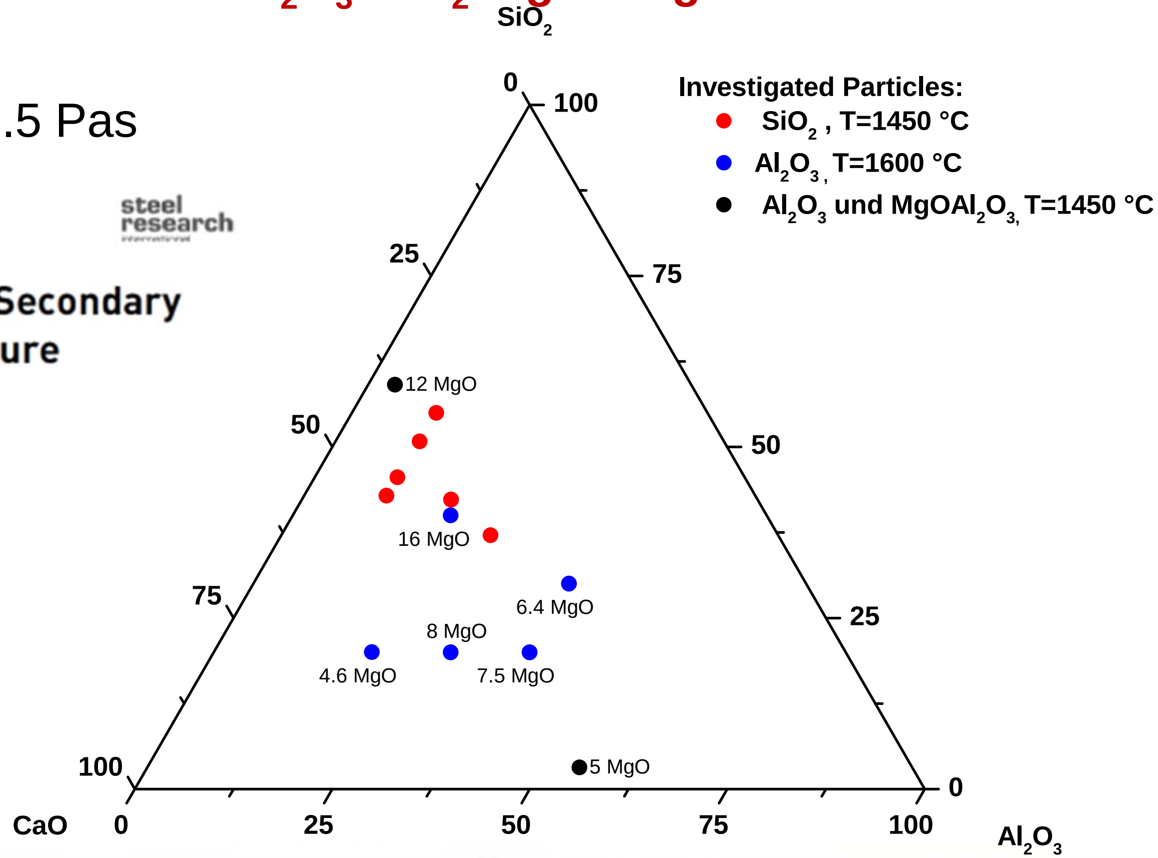
Slag viscosities between 0.2 and 2.5 Pas

www.steel-research.de

steel research

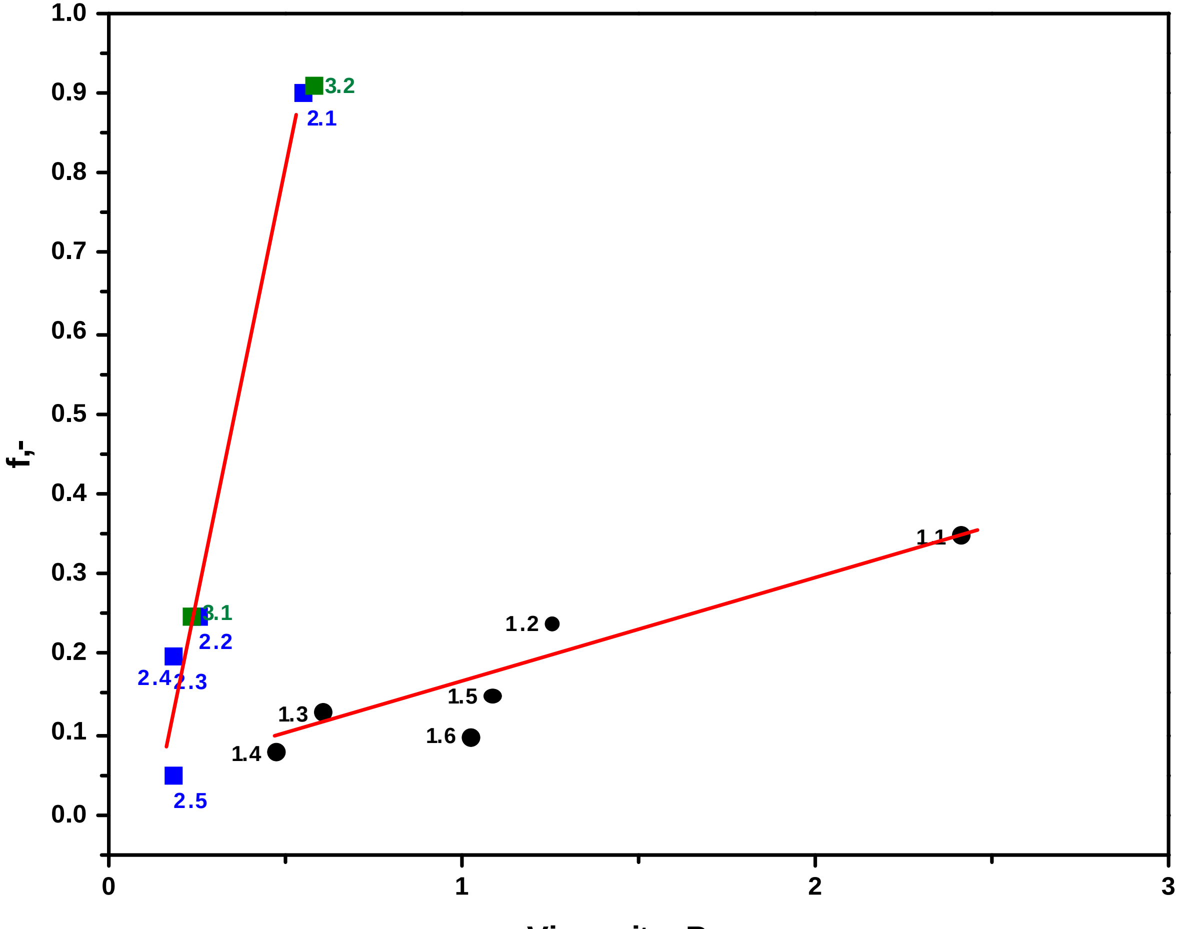
Study on Oxide Inclusion Dissolution in Secondary Steelmaking Slags using High Temperature Confocal Scanning Laser Microscopy

Susanne Michelic,* Jürgen Goriupp, Stefan Feichtinger, Youn-Bae Kang, Christian Bernhard, and Johannes Schenk



Relationship between all investigations

Linear dependence between f and slag viscosity



Source: S.Michelic et al.: steel research international 2015

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Summary and Conclusion

- **Laser Scanning Confocal Microscopy** enables the **in-situ observation** of different reactions of non-metallic inclusions between steel and slag

- **Inclusion dissolution in a slag:**

  - Al_2O_3 proved to dissolve faster than MgO Al_2O_3 in all investigated slags. SiO_2 dissolves the fastest.

  - Regarding, CaO-Al_2O_3-MgO-SiO_2 slags, slag viscosity is proved to be an essential influencing factor for the dissolution mechanism.

  - Slags with higher viscosity tend to show a slightly S-shaped normalized dissolution curve, whereas slags with lower viscosity are characterized by a parabolic pattern.

  - For all as far investigated systems a linear dependence between f and slag viscosity was observed.

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