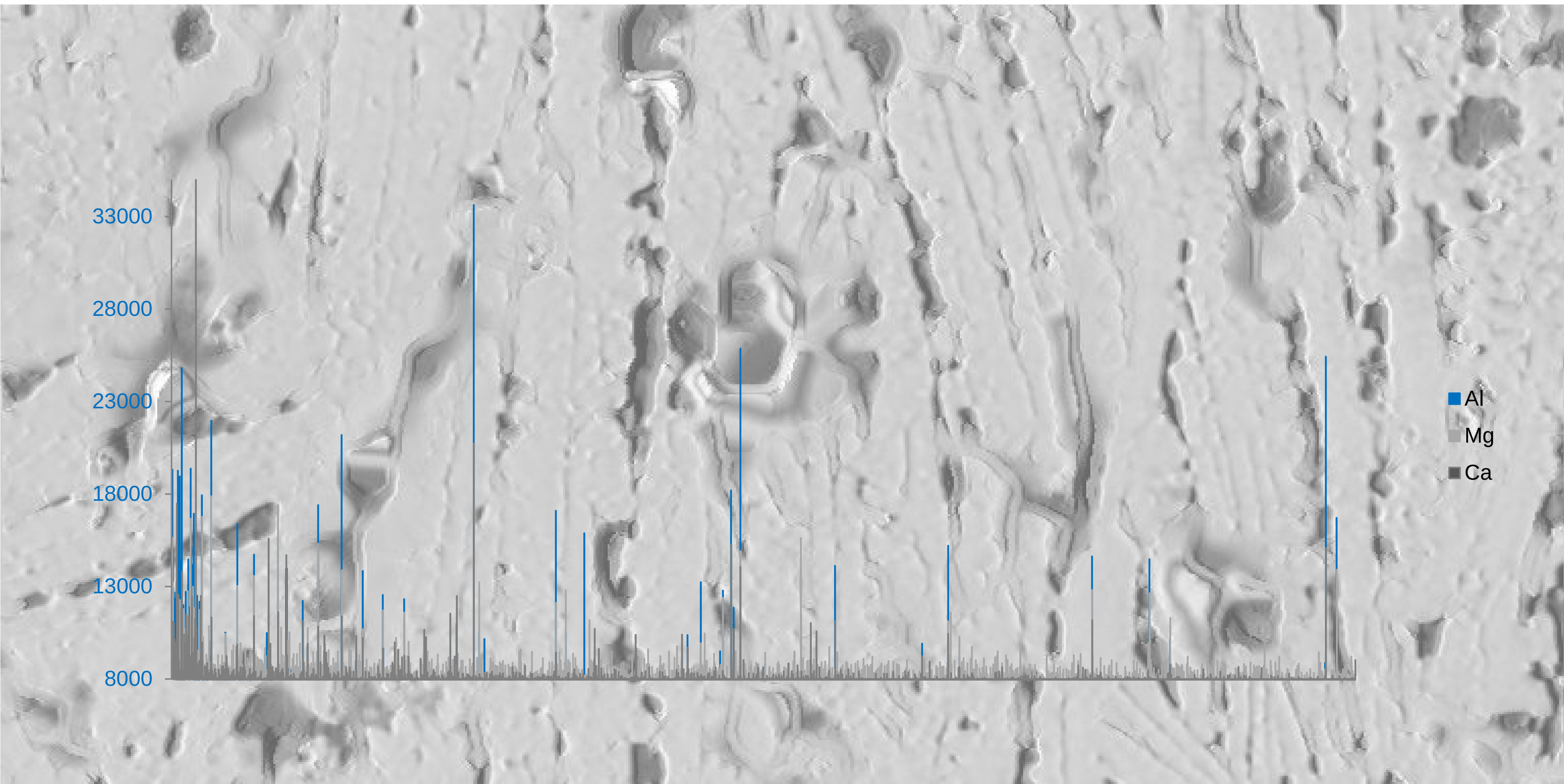
Inclusion Analysis / Steel Cleanliness Determination by Spark OES

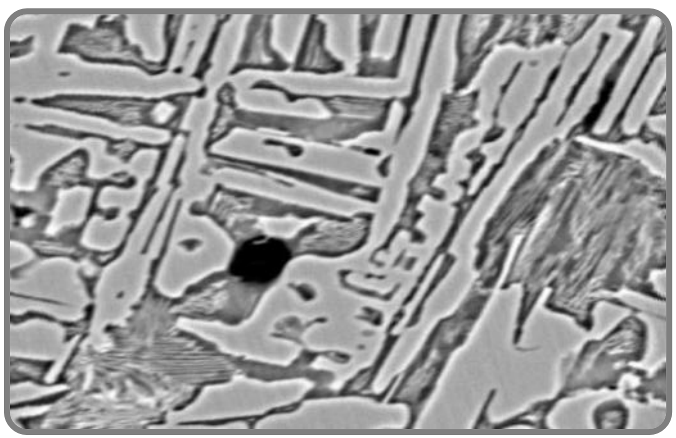
Characterization of inclusions in steel by OES Pulse Discrimination Analysis (OES-PDA)



Inclusion Analysis / Steel Cleanliness Determination by Spark OES

Topics

- Demand & Goals
- Reference Methods for Inclusion Analysis
- Rapid Method for Inclusions & Oxygen
- Hardware - Instrument Features
- Single Spark Evaluation
- Determination of Inclusions
- Principles of the PDA/MCI Method
- Comparison of Methods
- Results of PDA/MCI
- Conclusion



Speakers

Georg Schick
VP, Industrial Sales & Marketing
Bruker Elemental
Billerica. MA. USA

Martin Tilleman
Product Manager, Stationary OES
Bruker Elemental
Germany

Demands & Goals

Increasing demand for higher cleanliness in steels by customers in the metal processing industries.

- Production of high purity steels
  - Process monitoring of production steps
  - Final inspection of outgoing material
- Optimization of the production process
  - Improving Slagging Practices
  - Fast determination of oxide and sulfide inclusions during the process
  - Calculation of oxygen content
- Processing of steel for high demands
  - Fast cleanliness check of incoming billets and outgoing components

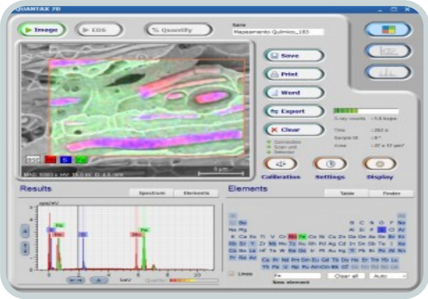
→ **Determination of parameters that define Steel Cleanliness**

Reference Method for Inclusion Analysis: SEM/EDS with Bruker Quantax 400 EDS

BRUKER

Scanning electron microscope with energy dispersive X-ray spectroscopy

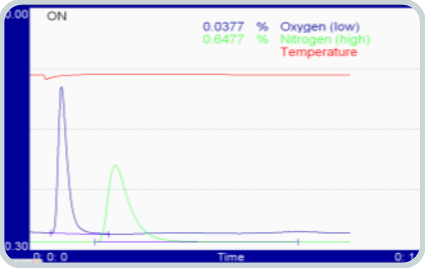
- Universal method: differentiation of carbides. oxides, nitrides, sulfides
- Large observation area
- Imaging method
- Highest accuracy
- Surface method, low penetration depth (~1 µm)
- Costly, long measurement time (~3-10 h)
- Highly educated operating staff



Reference Method for Oxygen Analysis: melt extraction with G8 GALILEO

Melt extraction with carrier gas method for the determination of oxygen

- Accurate analysis of total oxygen
- Fast measurement (~80 s)
- High analysed sample mass (~1000 mg)
- Demanding sample preparation
- Limited to oxygen only



Rapid Method for Inclusions & Oxygen: OES-PDA = MCI = Metal Cleanliness Inspection

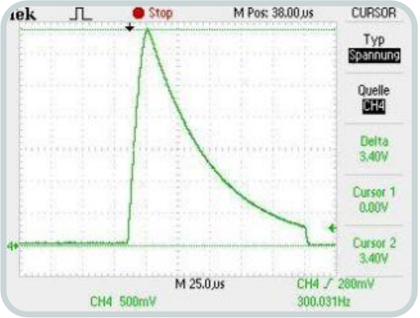
Inclusion characterization & oxygen determination by Optical Emission Spectrometry with Pulse Discrimination Analysis

- Complete elemental analysis
- Determination of various oxide and sulfide inclusions
- Calculation of total oxygen
- Simple sample preparation (grinding w/ SiC paper or milling)
- Fast measurement (~5 s/burn. multiple burns recommended. e.g. 5x)
- User-friendly software for “normal“ OES operator
- Feasibility study advisable

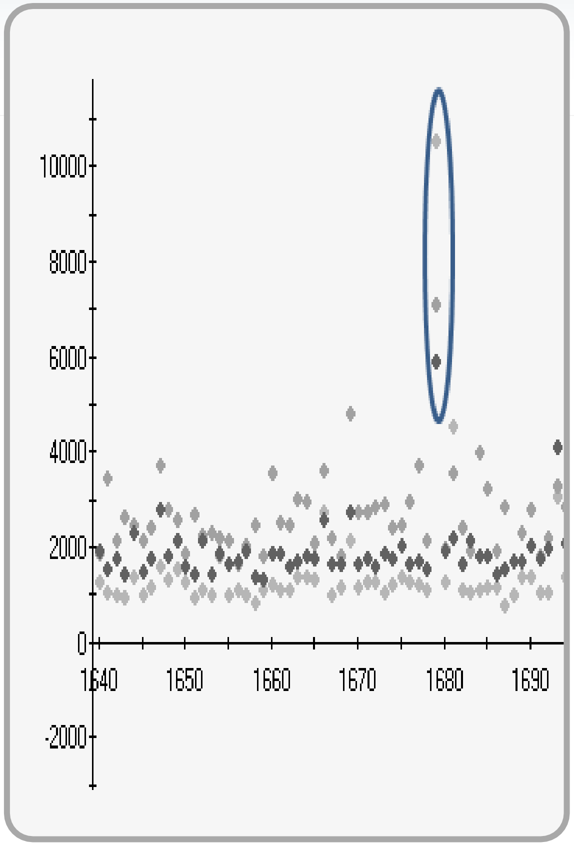
Hardware
Instrument Features

Q8 MAGELLAN Spectrometer

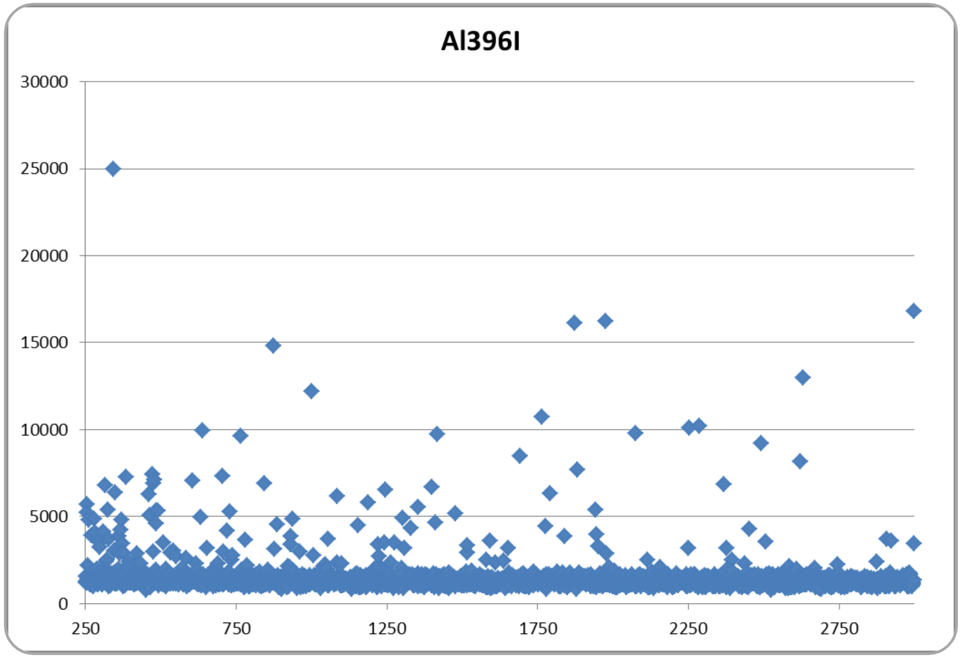
- Single Spark Detection (SSD) with visualization
- Unlimited parameters
  - any frequency up to 1 kHZ
  - all channels available for SSD
  - free source curve design
- Highest spectral sensitivity. lowest detection limits
- Lowest dark current, Channel-Photo-Multipliers (CPM)



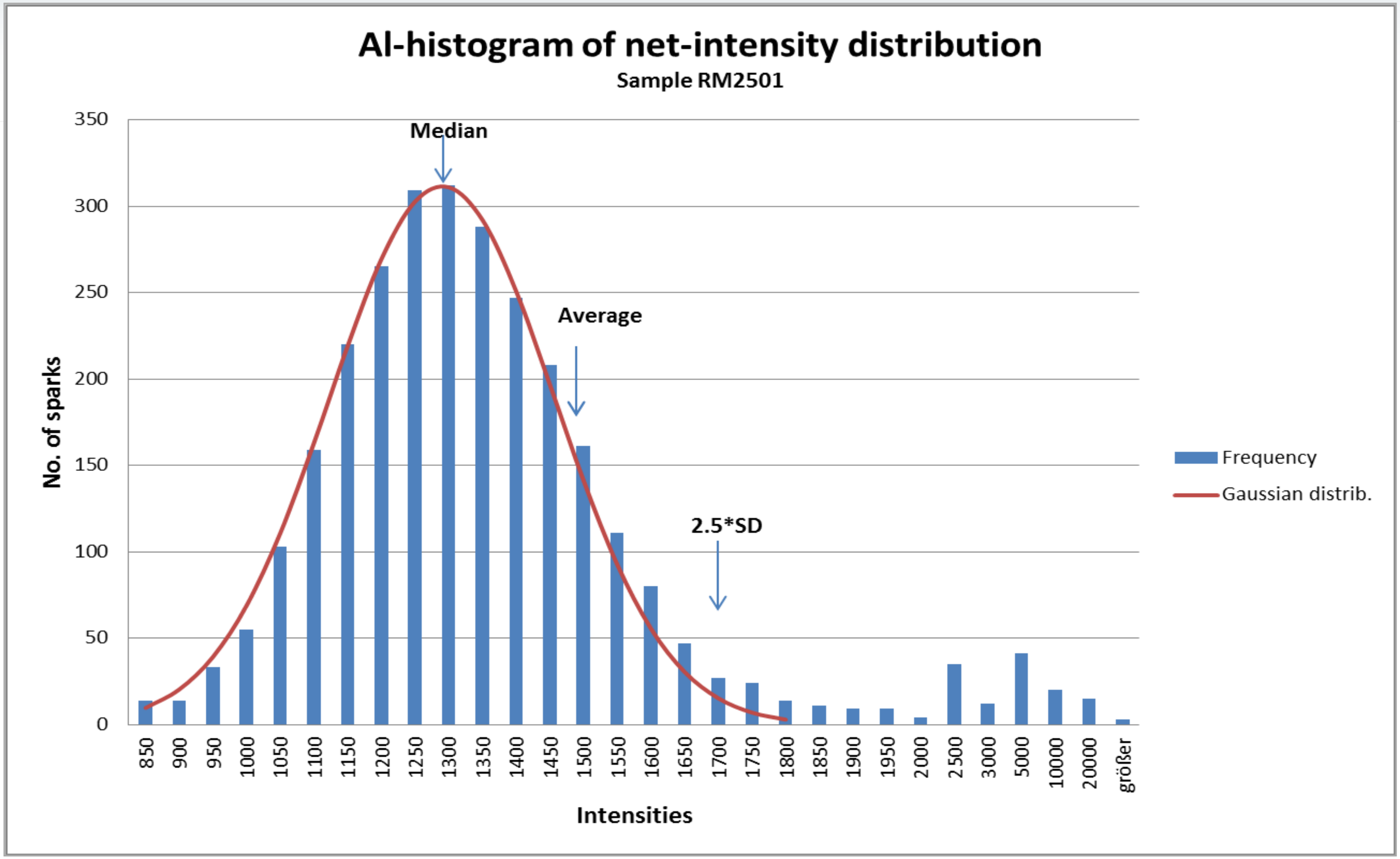
Single Spark Evaluation
Identification of Coincidences



Example for single spark signals with the Q8 MAGELLAN



Determination of Inclusions



Principles of PDA/MCI Method

Application field:

- Low and high alloyed steels with aluminium desoxidation

Workflow calibration & measurement

- Development of inclusion method with definition of spark parameters
- Calibration of method
- Sparking samples / generating data files (CSV format)
- Import data into MCI software
- Execute MCI software
- Print out (PDF) and export (CSV) and transfer results to other databases

Principles of PDA/MCI Method

Calculation steps

- Exclusion of sparks in the instable starting phase
- Detect and remove unenergetic sparks
- SD-Calculation of normalized Gaussian distribution
- Selection of oxide outliers (e.g. >3 SD)
- Calculation of element concentrations
- Identification of Ca as CaS. removing from oxide examination
- Identification of different oxides (correlation of outliers, e.g. Al to Ca)
- Stoichiometric calculation of oxide and sulfide compounds
- Calculation of total oxygen
- Classification of inclusions and grain size distribution

Comparison of Methods

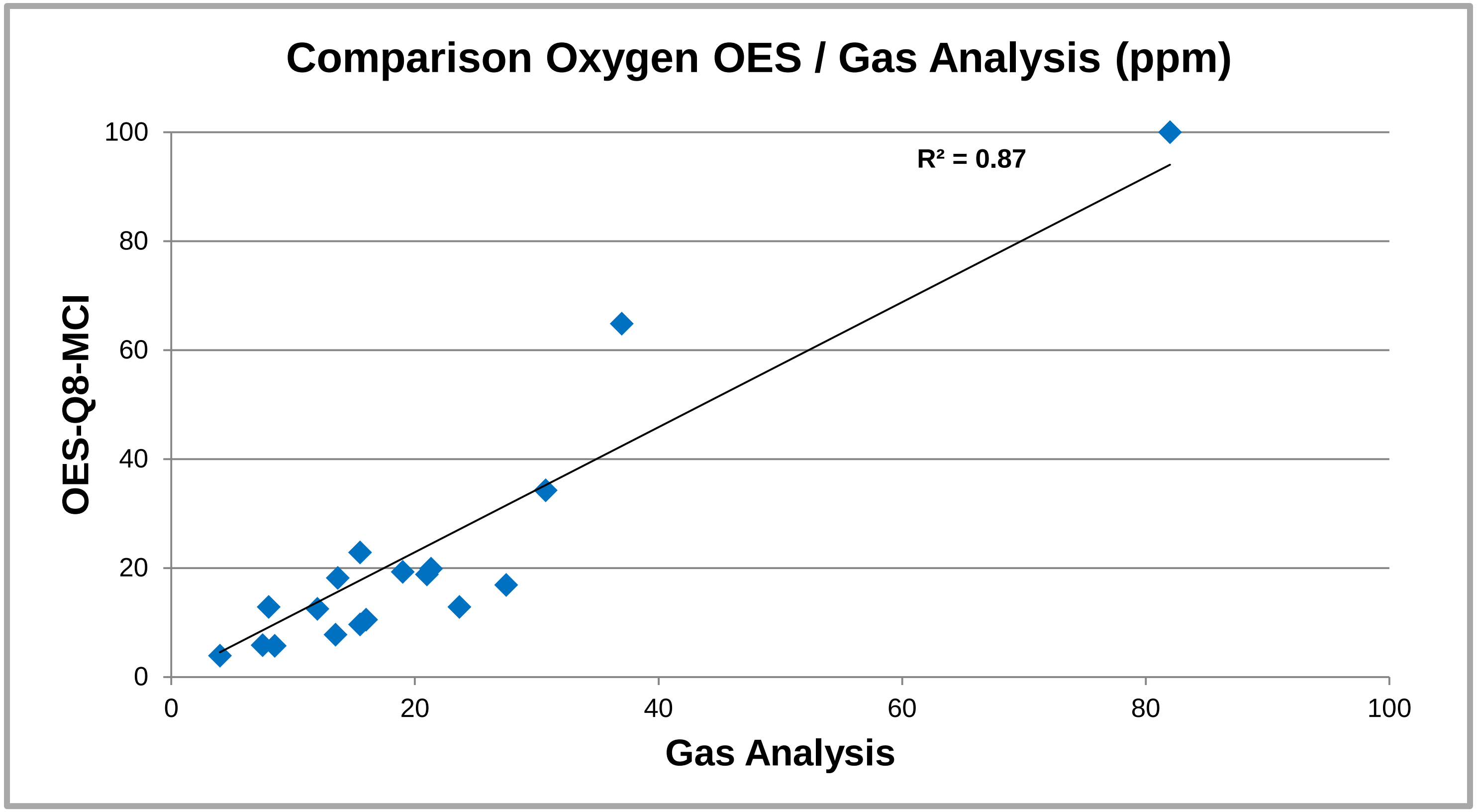
	SEM/EDS	ON/(H)	OES-MCI
Capital investment (approx. k€)	550	60	80
Operating costs	High	Medium	Low
Reference method / norm compliance	Yes	Partly	No
Penetration depth (of sample), approx.	1-3 µm	Complete	10 µm
Tested area (of sample), approx.	200 mm^2	Complete	7 mm^2)*
PDA/MCI-Measurement time, approx.	10 h	80 s)*	5 s)*
Ease-of-use (instrument)	Complex	Medium	Easy
Sample preparation	Medium	Complex	Easy
Analytical performance / value	High	Limited	Medium

)* values given for single reading, multiple measurements necessary

Comparison of Methods

Validation of MCI-method by comparison with Gas Analysis

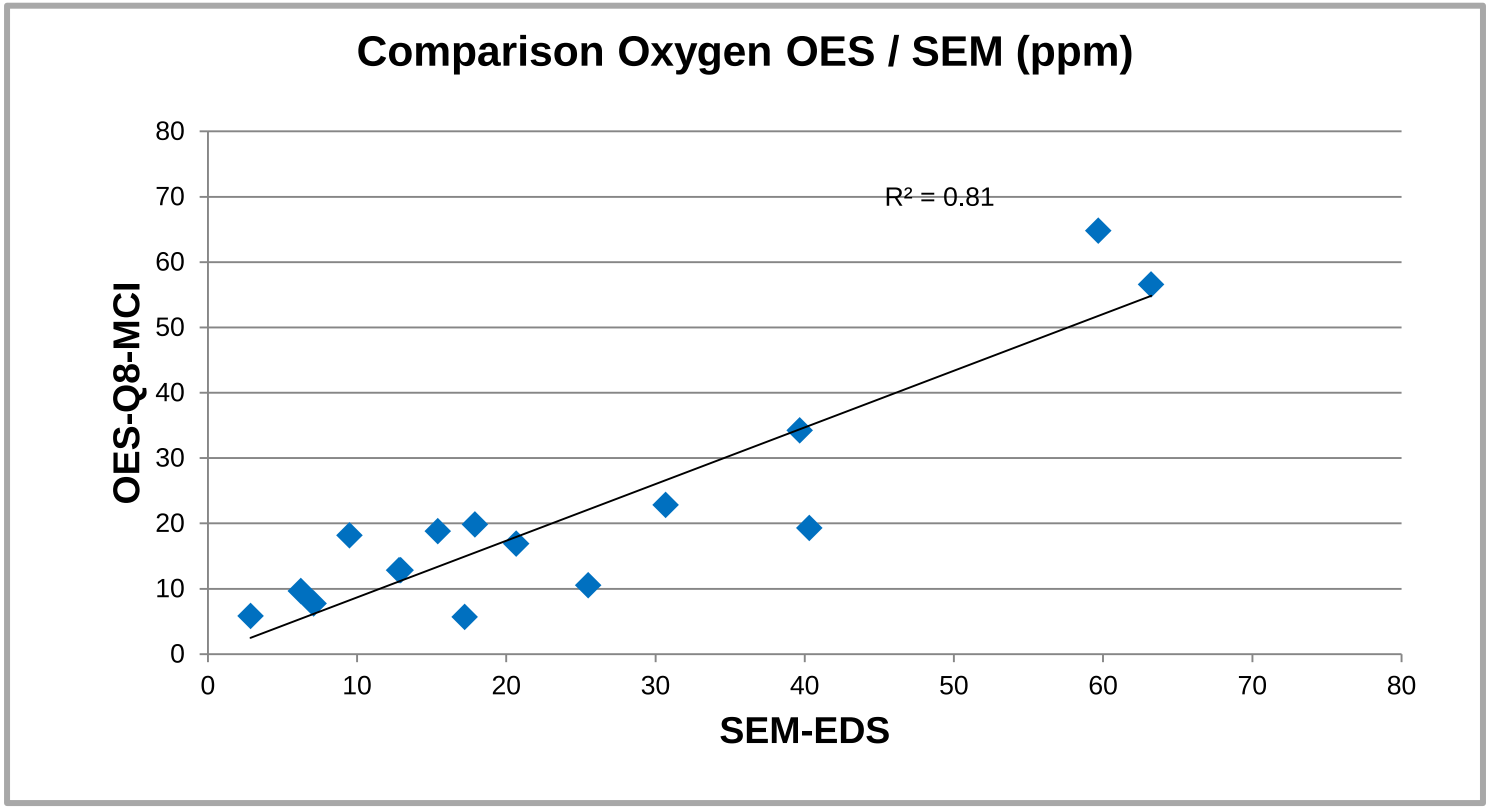
Application: low and mild alloyed steel



Comparison of Methods

Validation of MCI-method by comparison with SEM/EDS

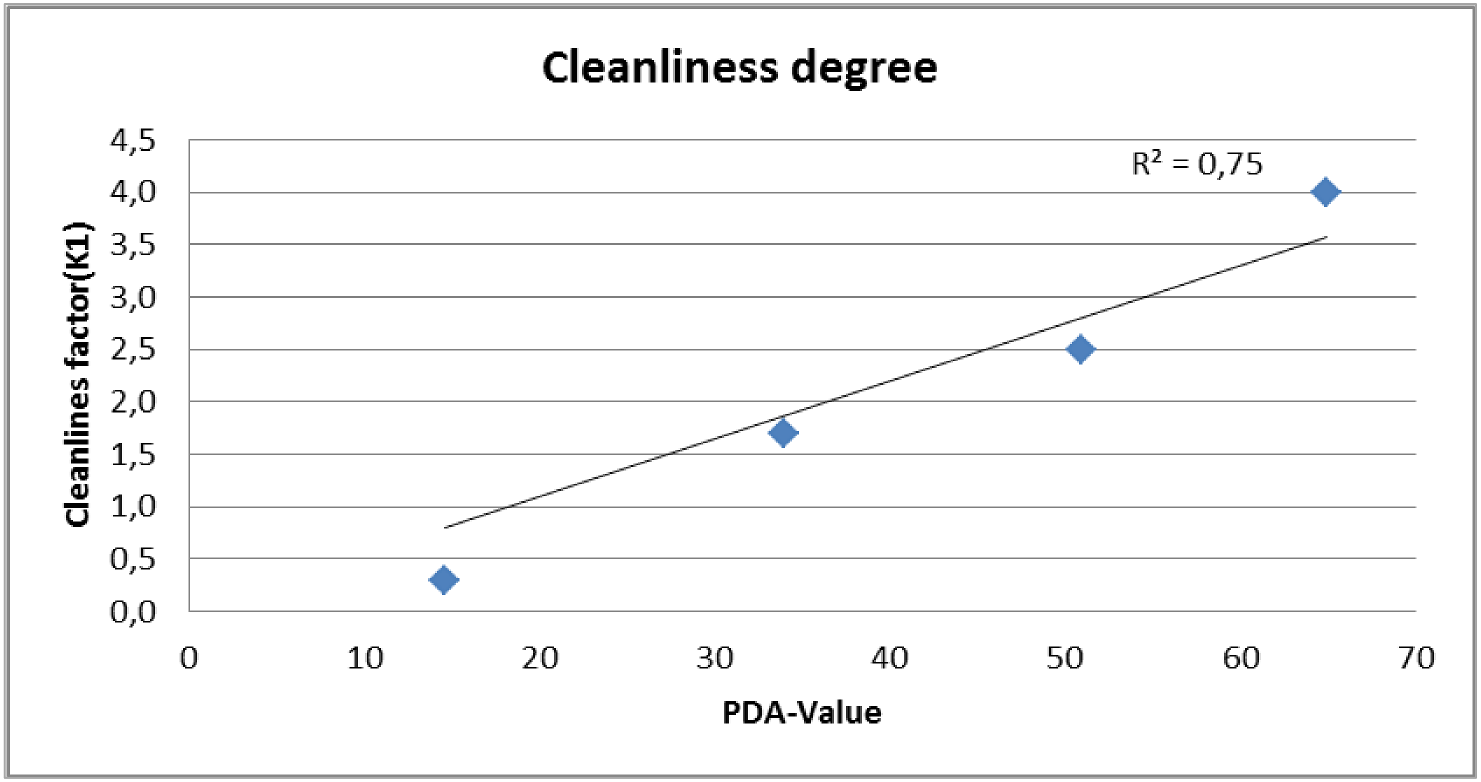
Application: low and mild alloyed steel



Comparison of Methods

Assessment of oxide cleanliness degree

Example of calibration based on customer samples (acc. EN 10247-KOX)



Results of PDA Method

Reproducibility - Example ball bearing steel

Sample	Analyse No	O-total (ppm)	Al-total (ppm)	Al-insoluble (ppm)	app. K1 value (EN 10247)	PDA-value
72 - BBS O_{REF} = 7 ppm	1	5.9	88	5.5	1.9	35100
	2	6.4	88	6.1	2.0	37527
	3	5.4	86	5.1	1.7	31491
	4	6.3	87	6.1	1.9	36122
	5	7.9	89	7.6	2.5	45566
	Mean	**6.4**	**88**	**6.1**	**2.0**	**37161**
	SD or RSD (PDA)	**0.9**	**1.1**	**0.9**	**0.3**	**14**
N14 - BBS O_{REF} = 12 ppm	1	13.1	54	13.3	4.2	77534
	2	13.7	53	13.8	4.2	78343
	3	10.3	50	10.2	3.2	60312
	4	12.9	55	12.8	4.0	73774
	5	13.5	57	13.4	4.2	77671
	Mean	**12.7**	**54**	**12.7**	**4.0**	**73527**
	SD or RSD (PDA)	**1.4**	**2.6**	**1.4**	**0.4**	**10**

Results of PDA Method

Reproducibility - Example reference material, low alloy steel

Sample	Analyse No	O-total (ppm)	Al-total (ppm)	Al-insoluble (ppm)	app. K1 value (EN 10247)	PDA-value
RM - V1 *) O_{REF} = 19 ppm	1	26.8	312	26.4	8.8	163033
	2	17.7	311	15.3	5.7	106909
	3	44.7	364	44.0	14.7	273353
	4	46.0	349	45.3	15.1	280605
	5	15.1	310	14.6	4.9	91548
	Mean	**30.1**	**329**	**29.1**	**9.8**	**183090**
	SD or RSD (PDA)	**14.6**	**25.5**	**14.9**	**4.8**	**49**
RM - V3 O_{REF} = 17 ppm	1	19.4	1059	21.1	6.1	113989
	2	17.9	1062	19.6	5.6	103371
	3	19.3	1043	19.3	6.0	111988
	4	19.8	1061	21.3	6.2	114449
	5	17.6	1052	19.1	5.5	102769
	Mean	**18.8**	**1055**	**20.1**	**5.9**	**109313**
	SD or RSD (PDA)	**1.0**	**8.0**	**1.0**	**0.3**	**5**

***)** Inhomogeneous inclusion distribution!

Results of PDA Method
Presentation in the MCI Software

Software Screenshot

-	22,4	115,0	27,0	260,2	13,6	111,1	18,8	-	161,0	-	-	-	-	-	-	-	-
11,9	34,2	18,1	-	862,0	115,4	248,3	21,9	469,1	-	56,6	-	-	-	-	-	-	-
8,9	-	19,3	39,2	502,3	101,8	116,8	28,2	220,6	-	-	-	-	-	-	-	-	-
11,9	3,1	71,6	19,9	461,6	-	268,2	-	506,6	100,3	-	-	-	-	-	-	-	-
18,9	18,0	14,4	-	398,2	135,8	121,4	40,7	229,2	-	-	-	-	-	-	-	-	-
-	-	59,6	-	-	-	31,5	-	-	83,4	-	-	-	-	-	-	-	-
-	9,3	188,4	-	-	-	240,2	31,3	-	-	-	717,5	-	-	-	-	-	-
-	-	25,9	9,2	-	-	-	43,8	-	36,2	-	-	-	-	-	-	-	-
-	-	50,6	0,7	-	-	-	65,7	-	70,8	-	-	-	-	-	-	-	-
-	8,1	33,7	-	-	-	-	12,5	-	47,2	-	-						
5,9	-	95,1	-	-	-	3,4	15,6	-	133,1	-	-						
-	-	26,5	-	-	-	13,5	43,8	-	37,1	-	-						
1,0	1,9	40,3	-	-	54,3	-	21,9	-	56,5	-	-						
-	13,0	101,1	-	-	-	62,1	72,0	-	141,6	-	-						
-	3,7	68,6	-	-	-	-	78,2	-	96,1	-	-						
3,0	-	32,5	-	-	-	-	34,4	-	45,5	-	-						
-	17,4	29,5	17,0	79,2	122,2	6,7	-	-	41,3	-	-						
-	11,2	-	-	-	237,6	-	-	-	-	-	-						
2,0	11,8	89,1	20,6	72,4	74,7	111,1	75,1	-	124,7	-	-	-	-	-	-	-	-
2,0	-	27,7	11,3	303,2	47,5	166,1	-	313,8	38,8	-	-	-	-	-	-	-	-

	Ti	Mg	Ca	Zr	Mn	Si	Al	S	AlO	CaO	MgO	CaAlO	MgAlO	MgCaAlO	ZrO	TiO	SiO	MnO
tics																		
ıge	[illegible]	1112	1637	1705	14629	970	[illegible]	644										
lard deviation	116	276	695	311	2000	177	2756	[illegible]										
n	649	1090	1434	1700	14598	962	4853	641										
n std. d[illegible]iation	101	246	219	305	1971	165	485	128										
r	25	49	404	18	17	34	205	12	173	383	41	21	8	0	18	25	31	14
entrations (ppm)																		
Intensities)	26141	93318	[illegible]	[illegible]	[illegible]	51551	1522084	32674										
r	0,3	0,6	8,7	0,3	6,2	3,5	21,0	1,4	35,9	9,8	0,8	3,9	0,8		0,4	0,5	6,8	6,6
en		0,4	3,5				18,7											
ximate total	18,8	5,8	35,0	11,3	2415,8	206,2	406,4	31[illegible]										
size																		
1										161	21				18	24		
2									162	220	20	2	8			1		
3									10	[illegible]		19					31	14
4									1									
5																		

nored sparks (Fe): 0 PDA code to[illegible]: 132615,7 PDA code >1 µm: 124849,3

ign[illegible]CaS incl. for PDA-O: 19 Clean[illegible]s degree: 5,9 No. of inclusions in spark area > 1µm: 512

Ternary diagram | [DIA]-Export | Export PDF | E

- Statistics
- Grain size distribution
- Total oxygen

Results of PDA Method
Presentation in the MCI Software

PDF print-out
Analysis of a ball bearing steel (Standard quality)

Q8 MAGELLAN 15.04.2013

Sample name	**B140-1**
Method name	Fe Inc
No. of ignored sparks (Fe)	0
No. of ignored CaS incl. for PDA-O	44
PDA code total	42881,3
PDA code >1 µm	38564,6
No. of inclusions in spark area > 1µm	383
No. of inclusion (per 1000mm²)	2272,7
Cleanliness degree	1,2
PDA Oxygen (ppm)	11,9

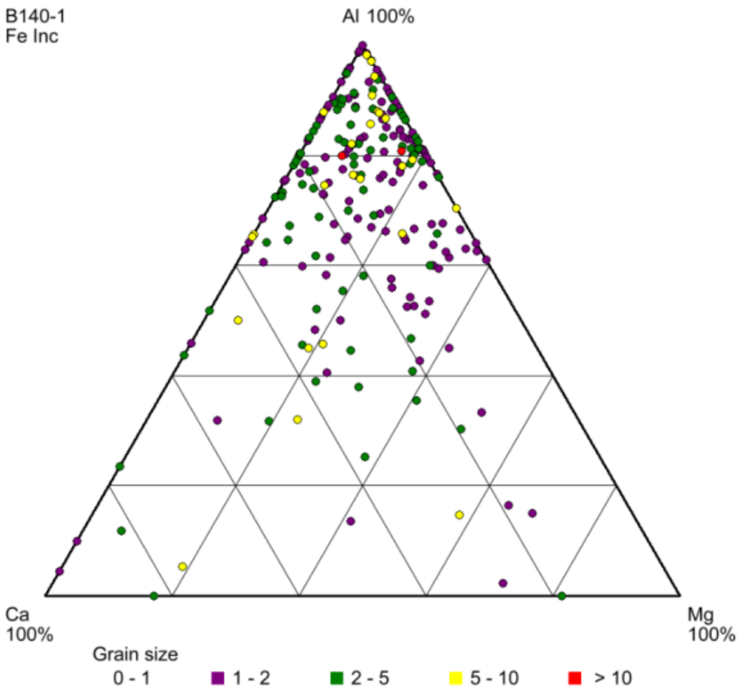
Statistics	**Ti**	**Mg**	**Ca**	**Zr**	**Mn**	**Si**	**Al**	**S**
Average	745	919	926	1719	25751	5785	1185	852
Standard deviation	670	291	225	266	3041	594	722	353
Median	691	885	897	1702	25642	5764	1059	732
Median std. deviation	87	207	136	248	2774	563	116	116
Outlier	168	84	61	35	20	34	261	435

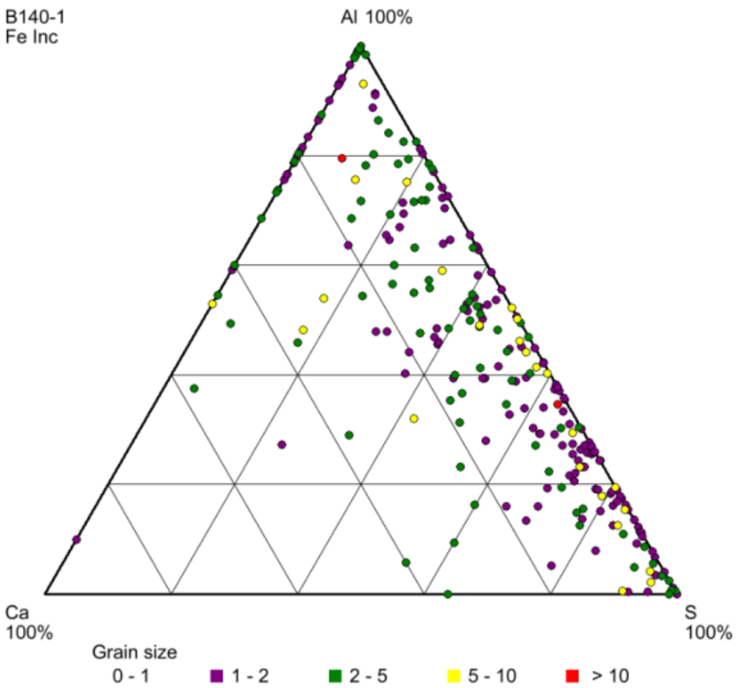
Concentrations (ppm)								
Outlier	4,9	1,2	1,2	0,5	24,8	10,4	12,0	35,5
Oxygen		0,8	0,5				10,6	
Approximate total	26,6	2,5	3,8	12,2	4428,9	2758,3	45,5	94,3

Inclusion analysis	**AlO**	**CaO**	**MgO**	**CaAlO**	**MgAlO**	**MgCaAlO**	**ZrO**	**TiO**	**SiO**	**MnO**	**TiAlO**	**MnSiO**	**CaSAlO**	**CaS**	**MnS**
No.	174	51	51	10	33	0	35	148	32	18	20	2	24	20	14
Outlier	13,1	0,7	1,0	0,4	5,2		0,7	3,7	20,7	15,7	5,9	2,7	6,9	2,5	14,8
Grain size															
Class 1	31	42	33				35	108							
Class 2	124	9	18	9	23			38			14		6	13	
Class 3	19			1	10			2	32	18	5	2	18	7	14
Class 4											1				
Class 5															

Results of PDA Method
Presentation in the MCI Software

Classification & Presentation in Ternary Diagrams





Conclusion

- OES-MCI reduces effort and costs, exceptionally in comparison to other methods
- Quick complementary method for material inspection
- Allows process control
- Final product assessment for steelworks and processing plants
- Easy extension of typical OES element/bulk analysis

New Application Report

Steel Cleanliness Analysis by OES-PDA

Application Report

Steel Cleanliness Analysis by OES-PDA

Rapid Quality Assurance Check with
Q8 Magellan and MCI-Software

Abstract

The present production of steels shows demanding requirements for quality, especially the needs for homogeneity and cleanliness are increasing. The key for premium steel products are best cleanliness degrees and lowest levels of Oxygen.

The classic way to determine non-metallic inclusions is usually done with metallographic microscope and scanning electron microscope with EDX analysis (SEM-EDX). These methods are slow and cost-intensive applications. Using optical emission spectroscopy with pulse discrimination analysis (OES-PDA) means fast and efficient determination of the cleanliness degree and Oxygen level. During the melting process these quality indicators are available and allow the process control in real-time. In addition a fast quality check of the final product is one of the main advantages of this method.
For this purpose Bruker Elemental has developed the software package MCI, "Metal-Cleanliness-Inspection". The MCI-Software evaluates on statistical basis the sparks, which are caused by nonmetallic inclusions.

The main features are the determination of:

- **Total oxygen content,**
- **Oxide and sulfide inclusions,**
- **Grain size distribution of different oxide types** (e.g. alumina, Ca-aluminate, Mg-spinel)
- **Cleanliness degree in correlation to reference methods** (e.g. EN 10247, ASTM E45)

Bruker Elemental - Billerica Demo Lab

k

O
E
S

&

G
A
S

Visit us at AISTech 2013, booth #2035
Pittsburgh, PA, USA on May 6-9

Q & A

Any questions?

Please type any questions you may have for our speakers in the Q&A panel and click Send.

Georg Schick
VP, Industrial Sales & Marketing
Bruker Elemental
Billerica. MA. USA

How did we do?

When you exit the webinar, please fill out our evaluation survey to let us know. We appreciate your feedback.

Martin Tilleman
Product Manager, Stationary OES
Bruker Elemental
Germany

Thank you!

OES and CS/ONH Webinars
Live and On-Demand

Industries Products Service News Events About us

Globally interacting with experts

Related links

- News
- Events

Contact

Home • Service • Education & Training • Webinars • OES and CS/ONH

OES and CS/ONH Analysis Webinars

Live and On-Demand

Available at www.bruker.com/service/education-training/webinars/oes-and-csonh.html

Webinar	Content
Apr 23, 2013 **Inclusion Analysis / Steel Cleanliness Determination by Spark OES**	Join Bruker Elemental panelists Georg Schick and Martin Tilleman for this one-hour webinar in which they highlight Spark OES as a simple and easy elemental and inclusion analysis method, even for non-metallurgists. They will provide an overview of steel cleanliness inspection software that gives full details of your steel sample, including graphic presentation of data in ternary diagrams.

Like what you learned in this webinar?

Subscribe to Bruker's ***FIRST Newsletter*** to get webinar announcements, fascinating articles, and elemental analysis news delivered right to your inbox!

Subscribe at:

https://www.bruker.com/about-us/register.html

BRUKER

Innovation with Integrity