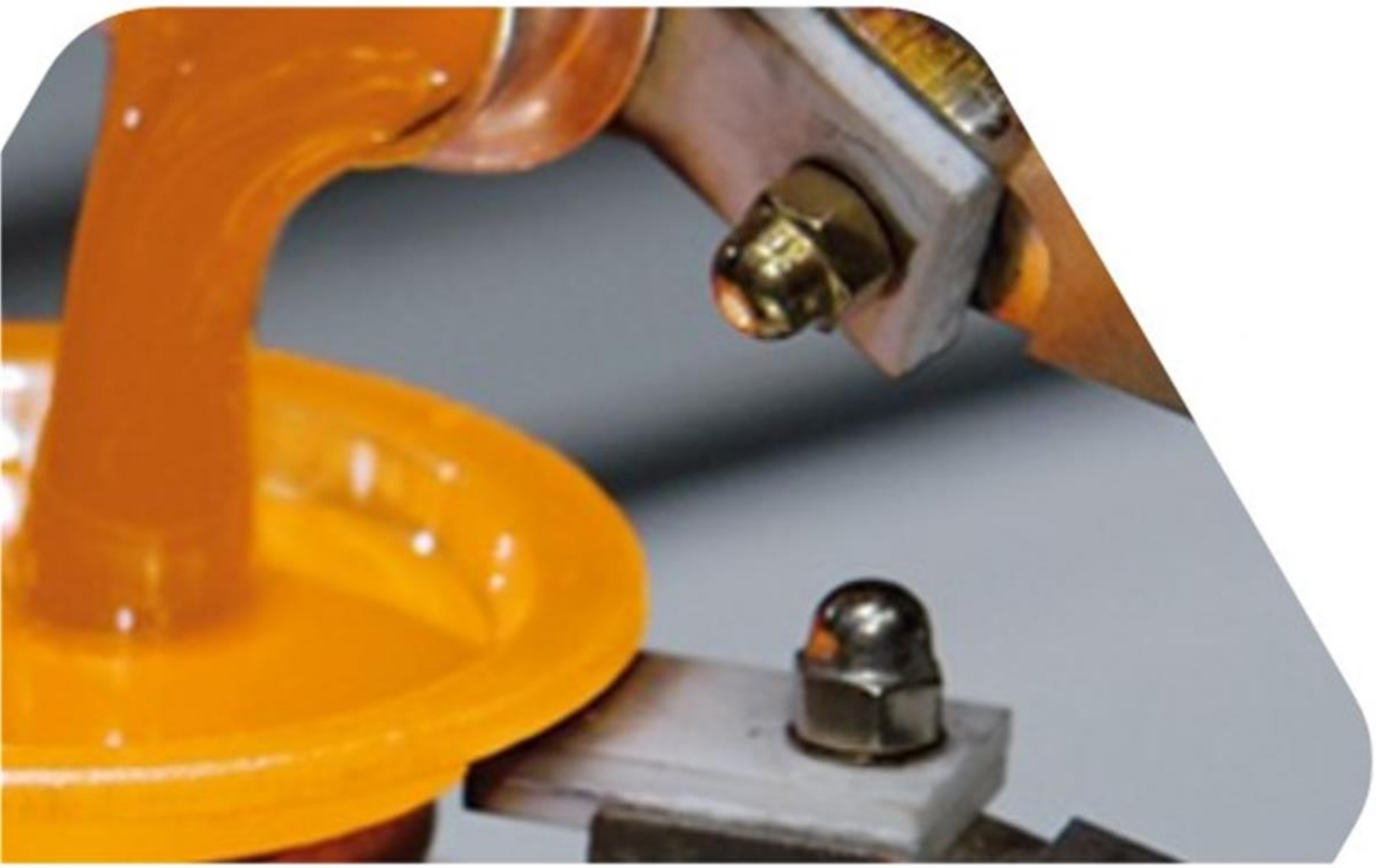
Wed 24th June 2020

HERZOG & BRUKER

WEBINAR

The Importance of Good Sample Preparation

In partnership with DSL Datech Scientific Limited



HERZOG

MARTIN LISCHKA

Research & Development

BRUKER

COLIN SLATER

Research & Development

Datech Scientific Limited

SUPPORTING GOOD SAMPLE PREPARATION SINCE 1989

XRF, XRD & OES SAMPLE PREPARATION EQUIPMENT

MANUAL BENCHTOP SEMI–AUTOMATED FULL AUTOMATION

Adam.housley@datech-scientific.co.uk

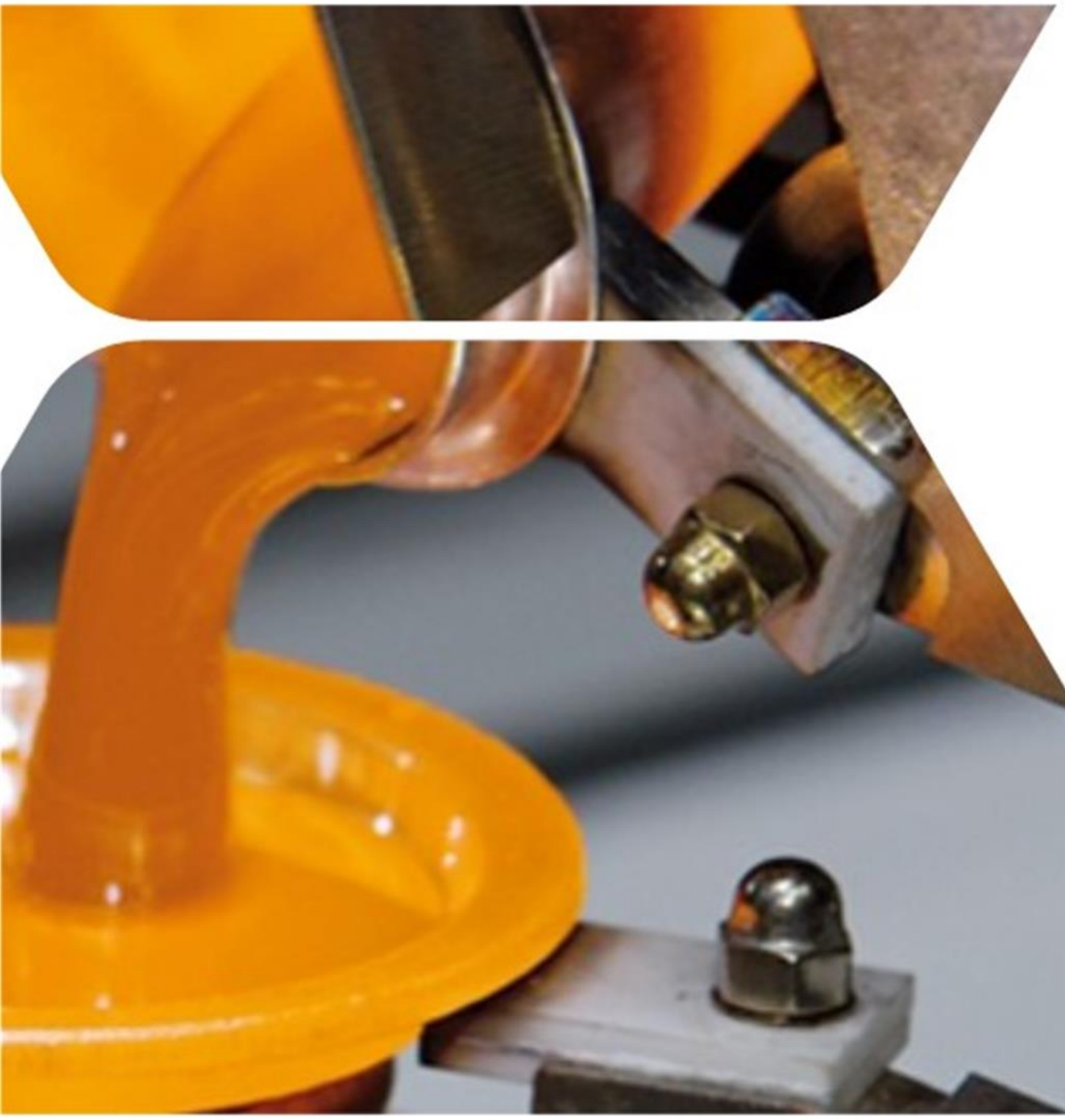
Datech Scientific Limited

SERVICE & SUPPORT

XRF, XRD & OES
CONSUMABLES

GRINDING AID
STEEL RINGS
Al CUPS
BINDER
FILMS
FLUX
CRM

Adam.housley@datech-scientific.co.uk



Datech Scientific Limited

STRATEGIC COLLABORATIONS

HERZOG MASCHINENFABRIK

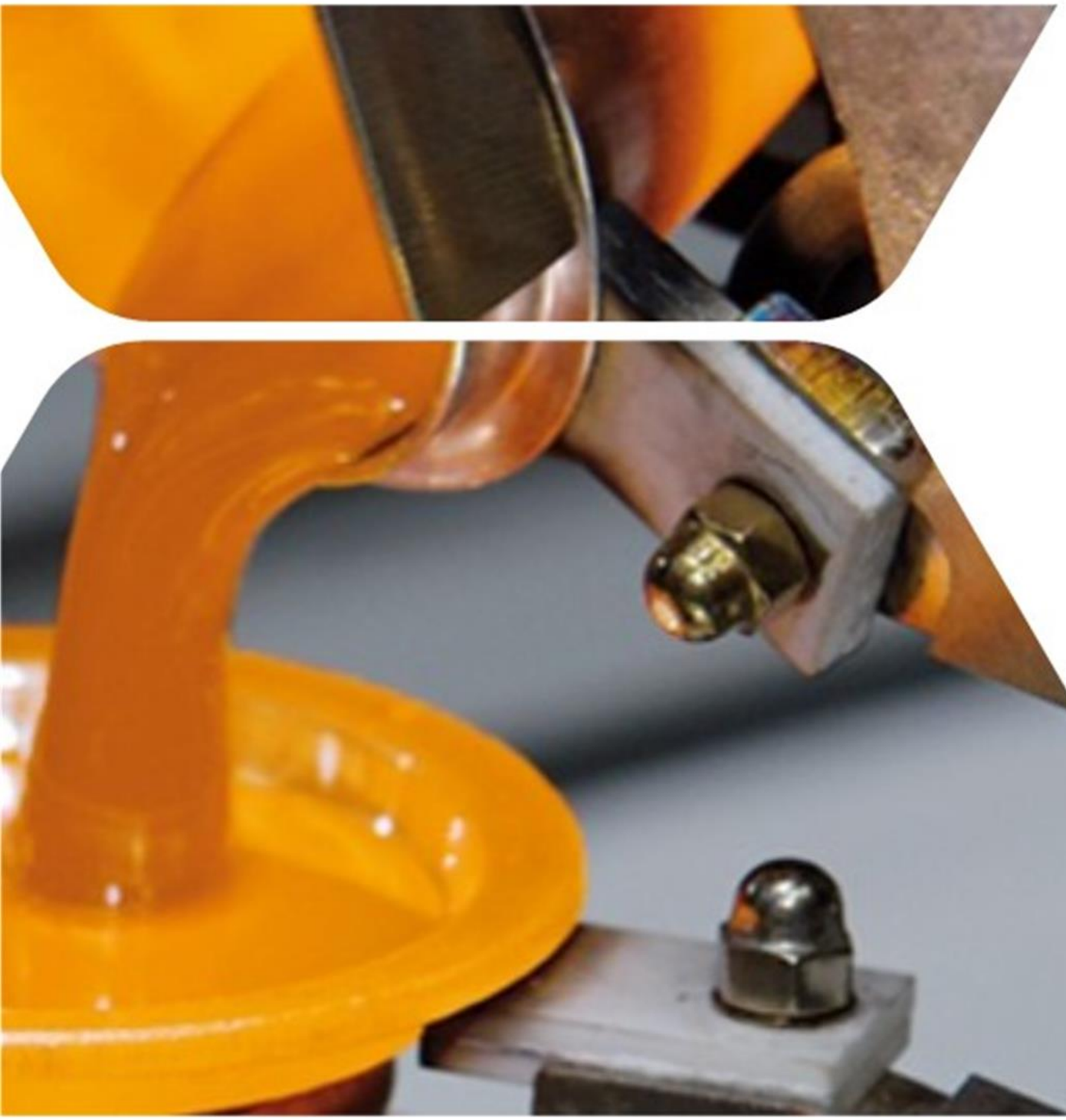
PD INSTRUMENTS

ANALYTICAL OEMS

APPLIED SPECTRA

LIBS & LA-ICP-MS

Adam.housley@datech-scientific.co.uk



HERZOG

MARTIN LISCHKA

Research & Development

HERZOG

Your Partner – For automatic sample preparation

HERZOG

Maschinenfabrik Herzog GmbH& Co.KG
Osnabrück, Germany

Parent

Subs

HERZOG
Automation Corp.
Cleveland, OH, USA

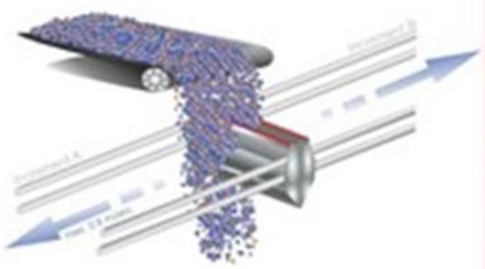
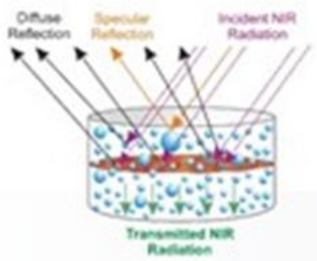
HERZOG
Japan Ltd.
Tokyo, Japan

HERZOG
Herzog Automation
Shanghai, China

Reps

Egypt	Australia	Bolivia	Brazil	Canada	Colombia	Czech Republic	India
Iran	Italy	Korea	New Zealand	Norway	Pakistan	Peru	Philippines
Poland	Portugal	Russua	South Africa	Sweden	Spain	United Kingdom	Venezuela

• Measurement uncertainty

Multistage sampling & preparation process	Reality	Countering methods & measurements	Target
Sampling 	*Example* S_x = 55%	Heterogenity assesment Good sampling approach Proper sampling equipment Replication experiment Variography	*Example* S_x = 5%
Sample preparation 	S_x = 35%	Automation Solid methodology Repeatabilty checks Preparation monitoring Equipment monitoring	S_x = 2%
Analysis 	S_x = 1,5%	Specific Calibration Solid methodology Monitor samples Drift correction Equipment monitoring	S_x = < 1%
	Total error 65 %		*Total error 5,5 %*

- **Pressed pellet or fusion**

Sample taking | Sample transport | Sample preparation | Control system

Fusion

Sampling (FSE < 5% rel.)

Air tube systems

Manual to Semi-automatic solutions

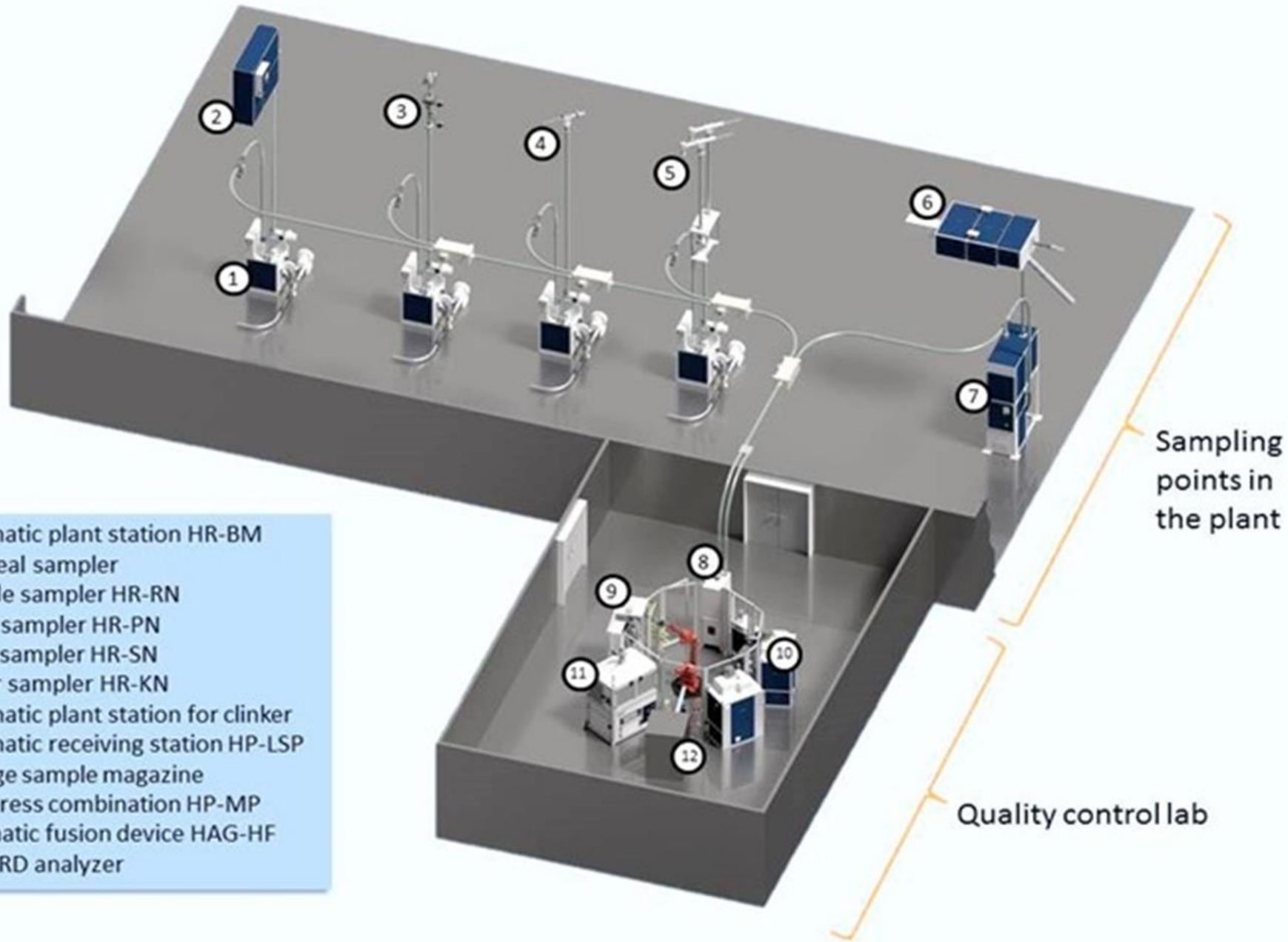
Fully automatic solution

Prep Master Software

Pelletizing

QC fusion

- **Fully automated cement laboratory**



• Factors influencing grinding

Method related:

- Input size of sample
- Selected grinding time
- Selected grinding speed
- Grinding aids

Material related:

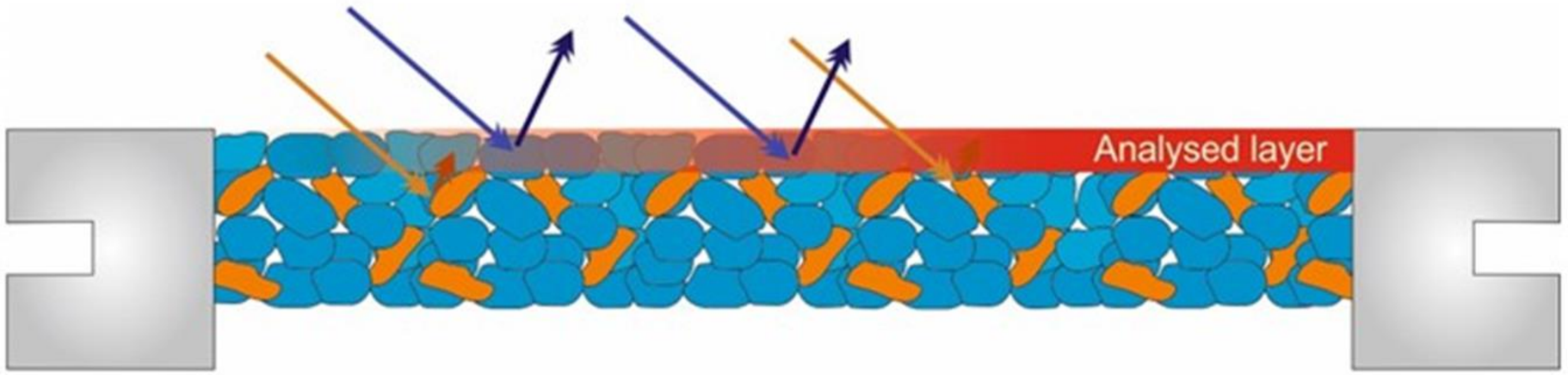
- Material heterogeneity
- Mineral content
- Mohs hardness of constituents
- Particle size distribution

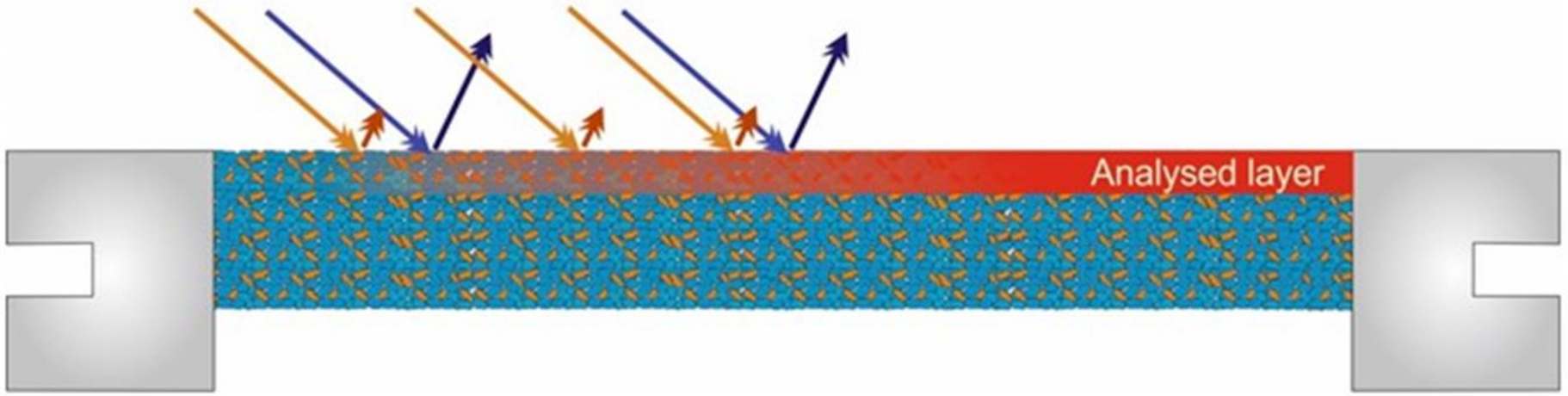
Equipment related:

- Grinding method (*hammer mill, vibrating mill*)
- Material grinding tools
- Constitution of grinding tools
- Operational mode (*manual/automatic*)
- Equipment performance (*material loss*)
- Vessel temperature

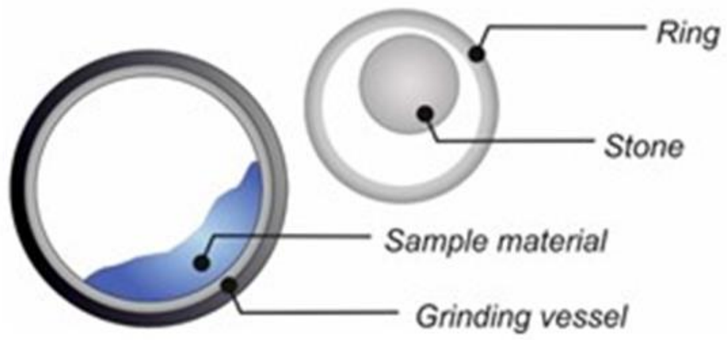
Nevertheless those are not all…

- **Particle size effect**

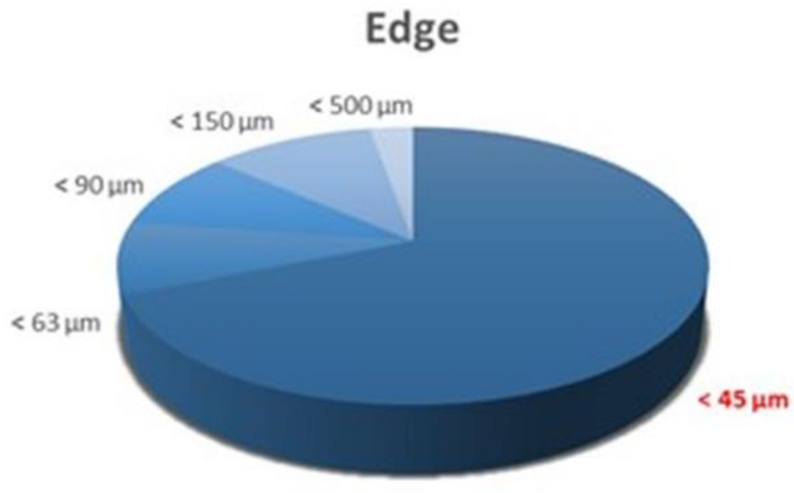


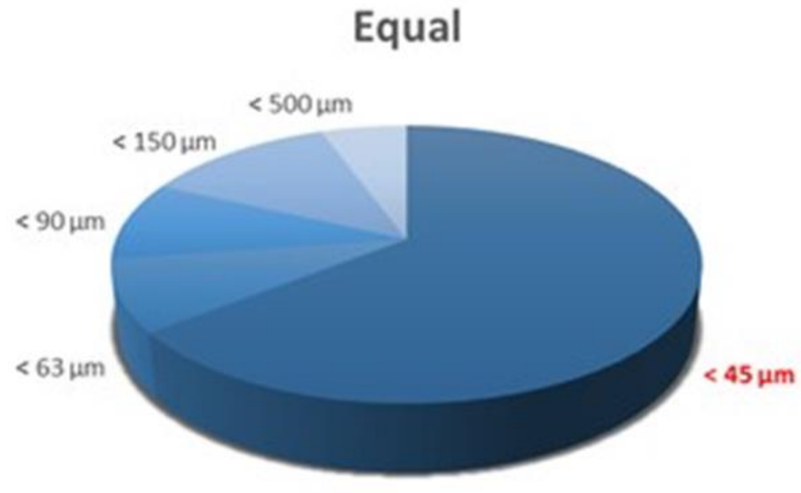


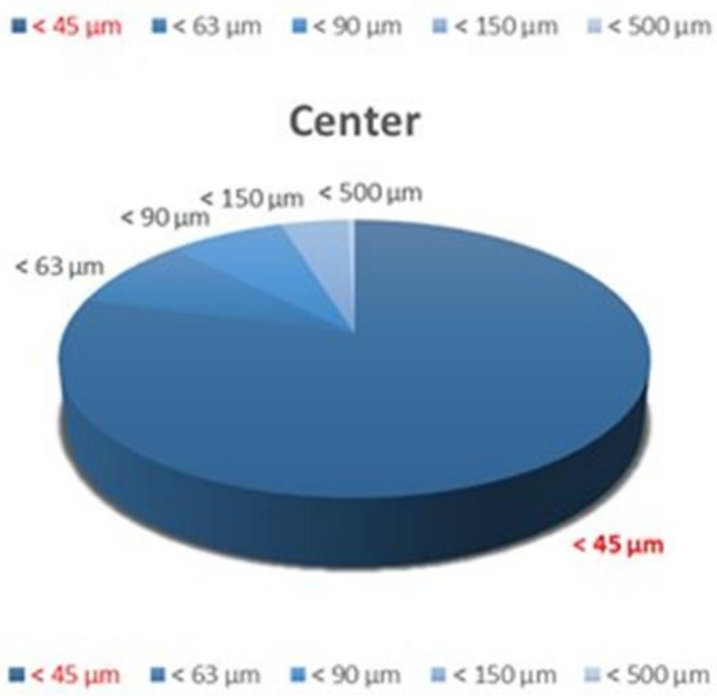
• Influence manual loading HSM 100

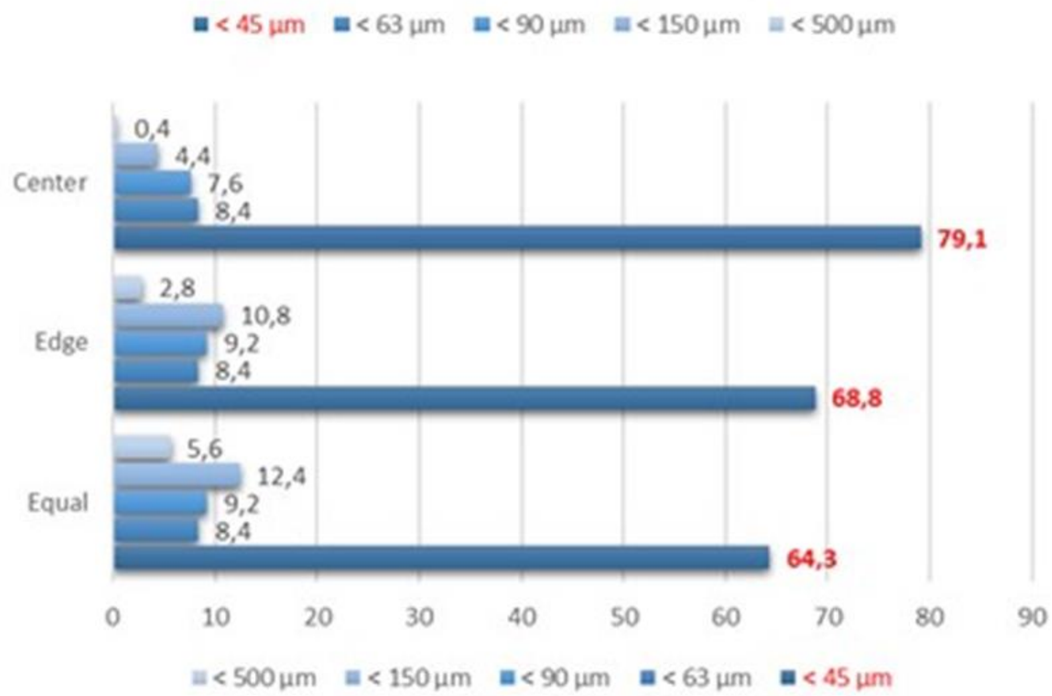


- **Impacting particle size distribution**

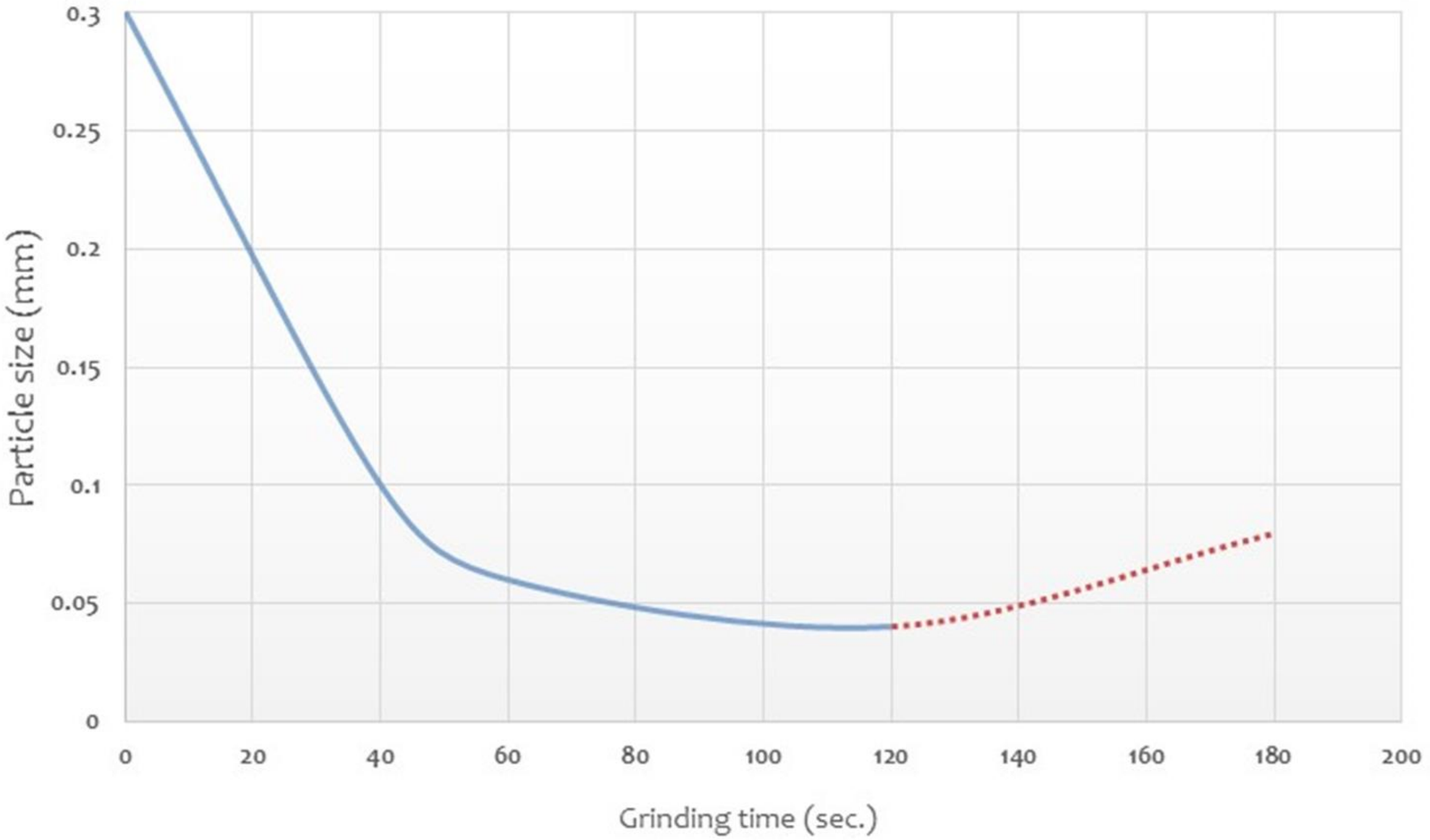








- **Development of particle size during grinding**



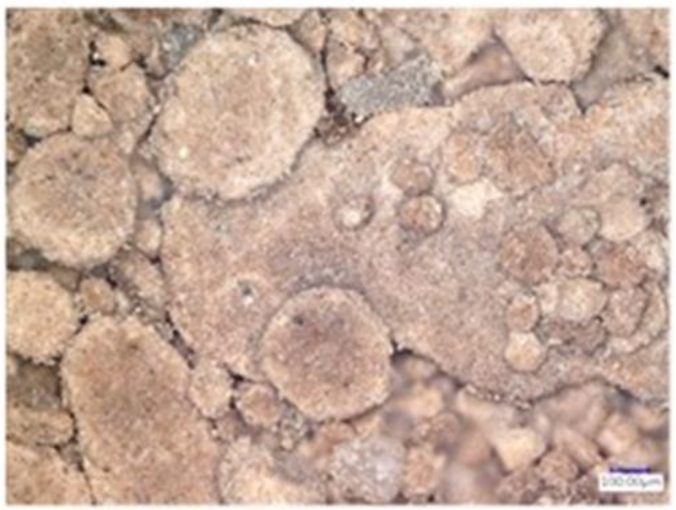
- **Agglomerate formation** (Raw meal)

Speed: 800 rpm
Fraction X ≥ 75 µm

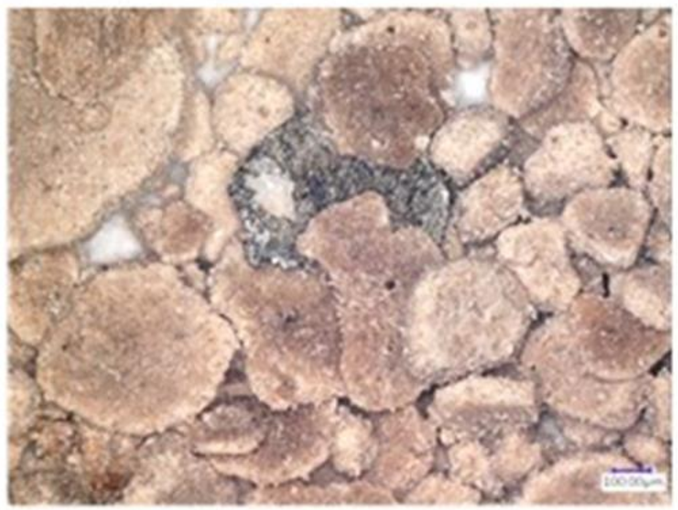
Speed: 900 rpm
Fraction X ≥ 75 µm

Speed: 1000 rpm
Fraction X ≥ 75 µm

Speed: 1100 rpm
Fraction X ≥ 75 µm



Speed: 1200 rpm
Fraction X ≥ 75 µm

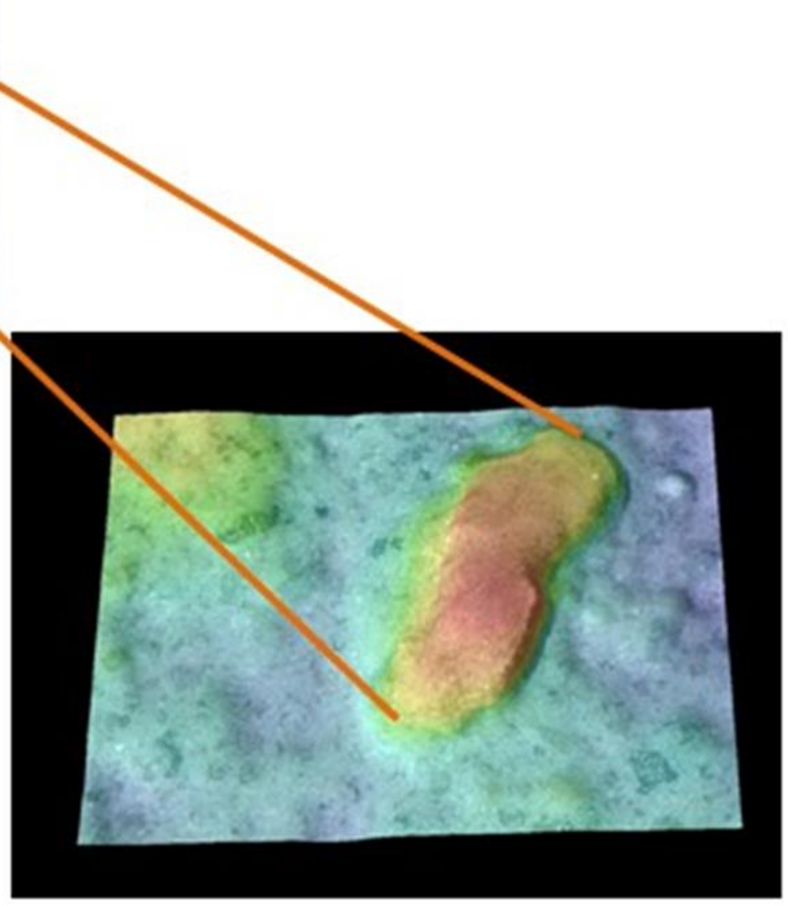


Speed: 1300 rpm
Fraction X ≥ 75 µm

- **Agglomerates**

Grinding

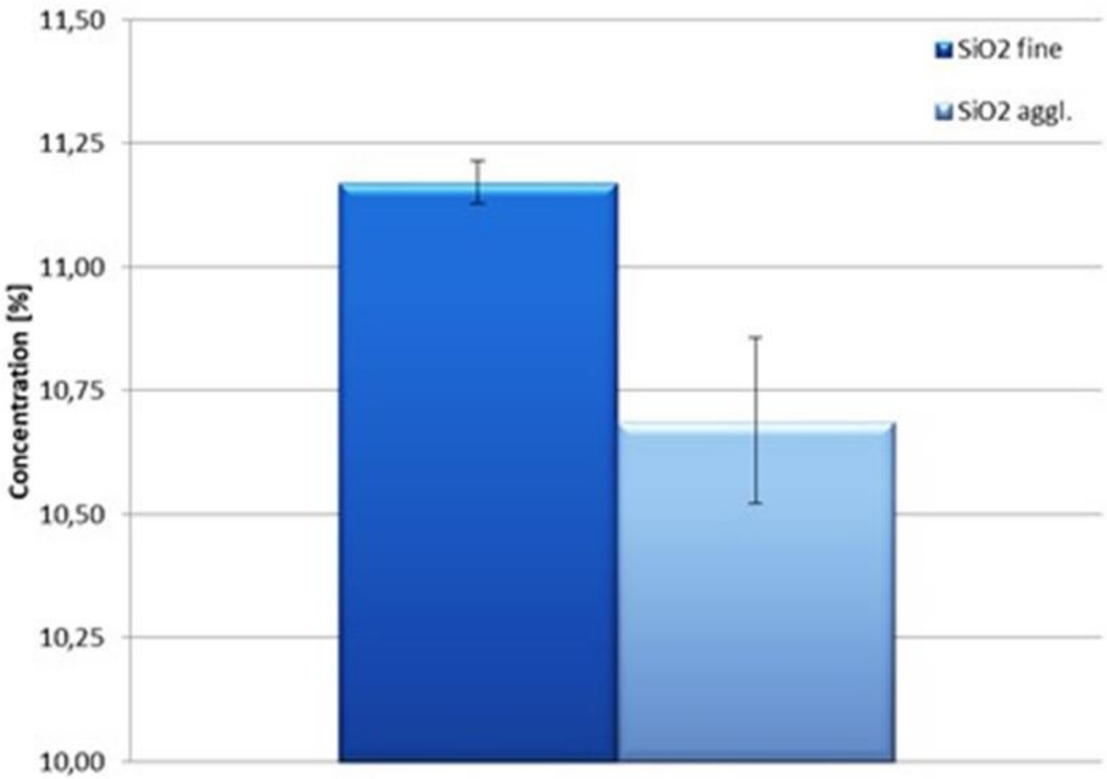
Pressing

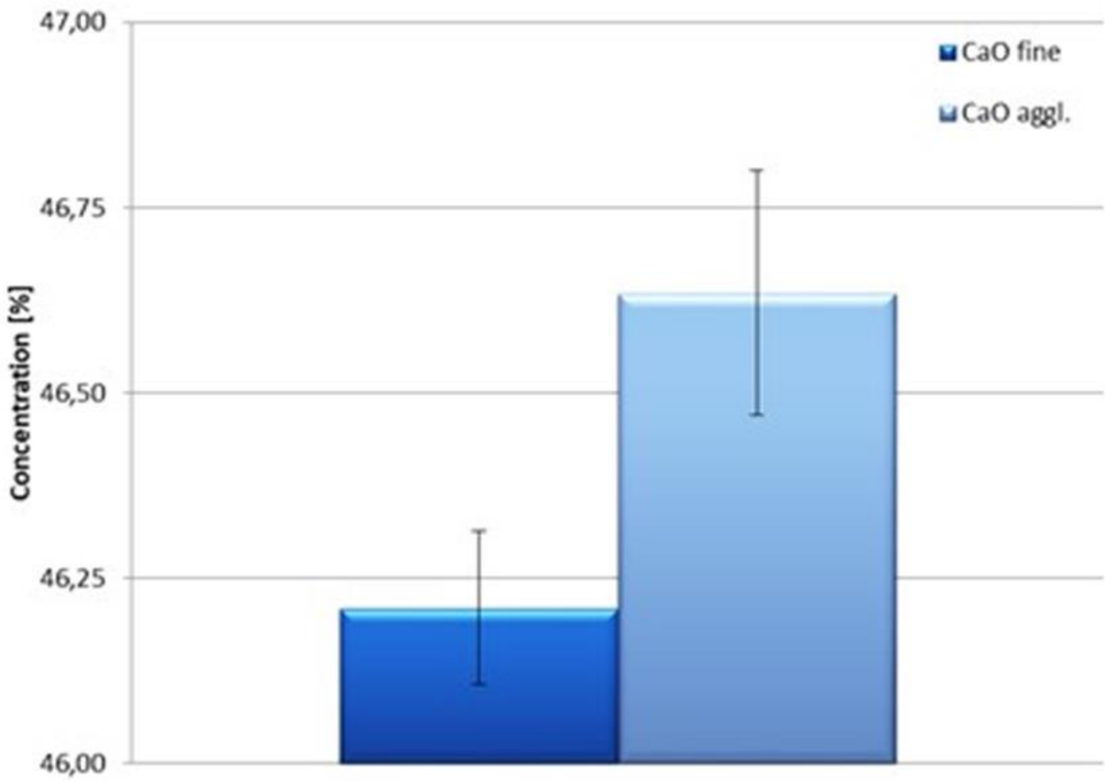


• Agglomerates

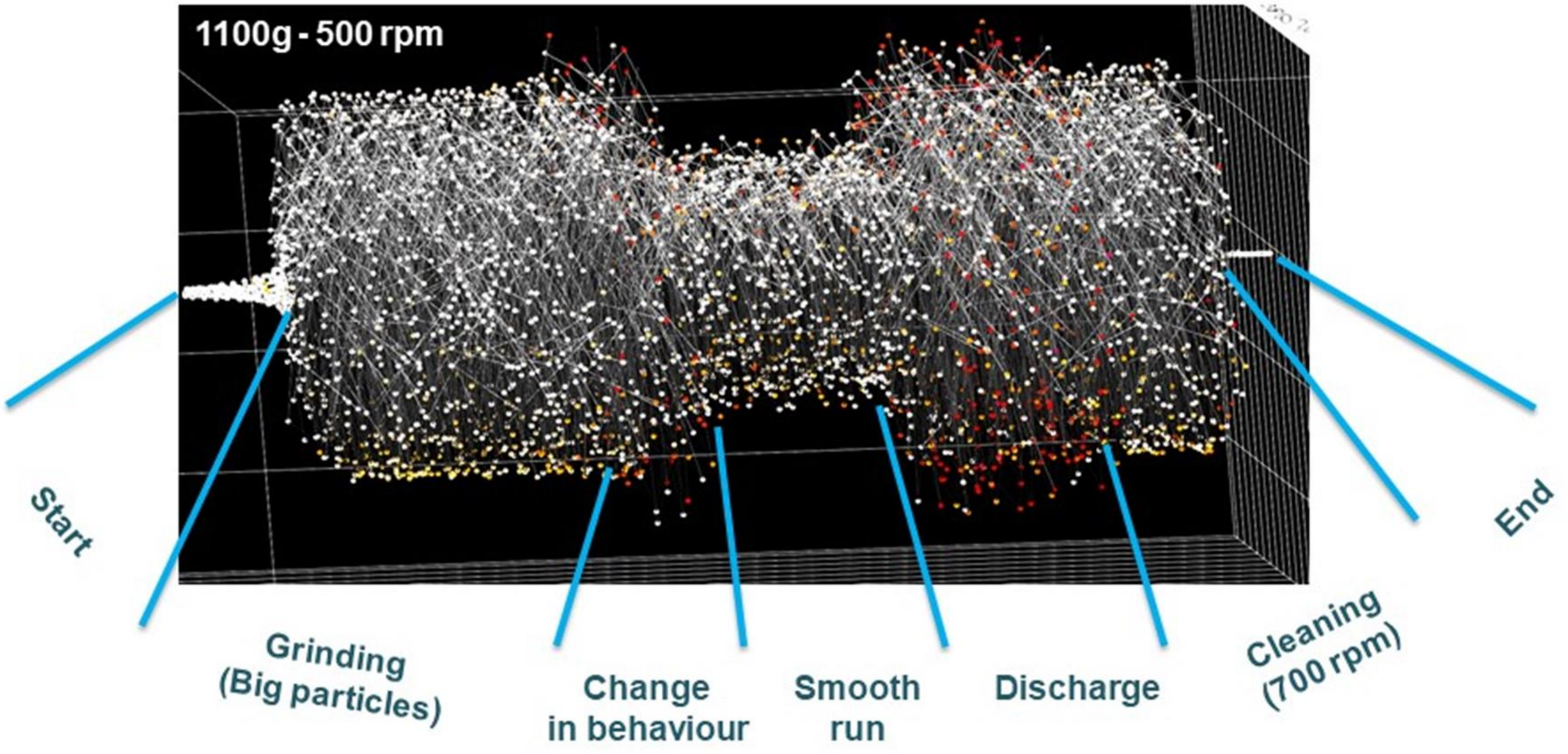
Fine

Aggl.



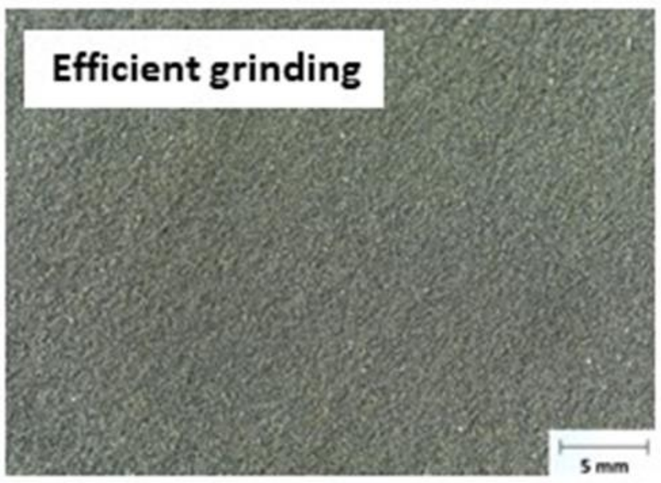


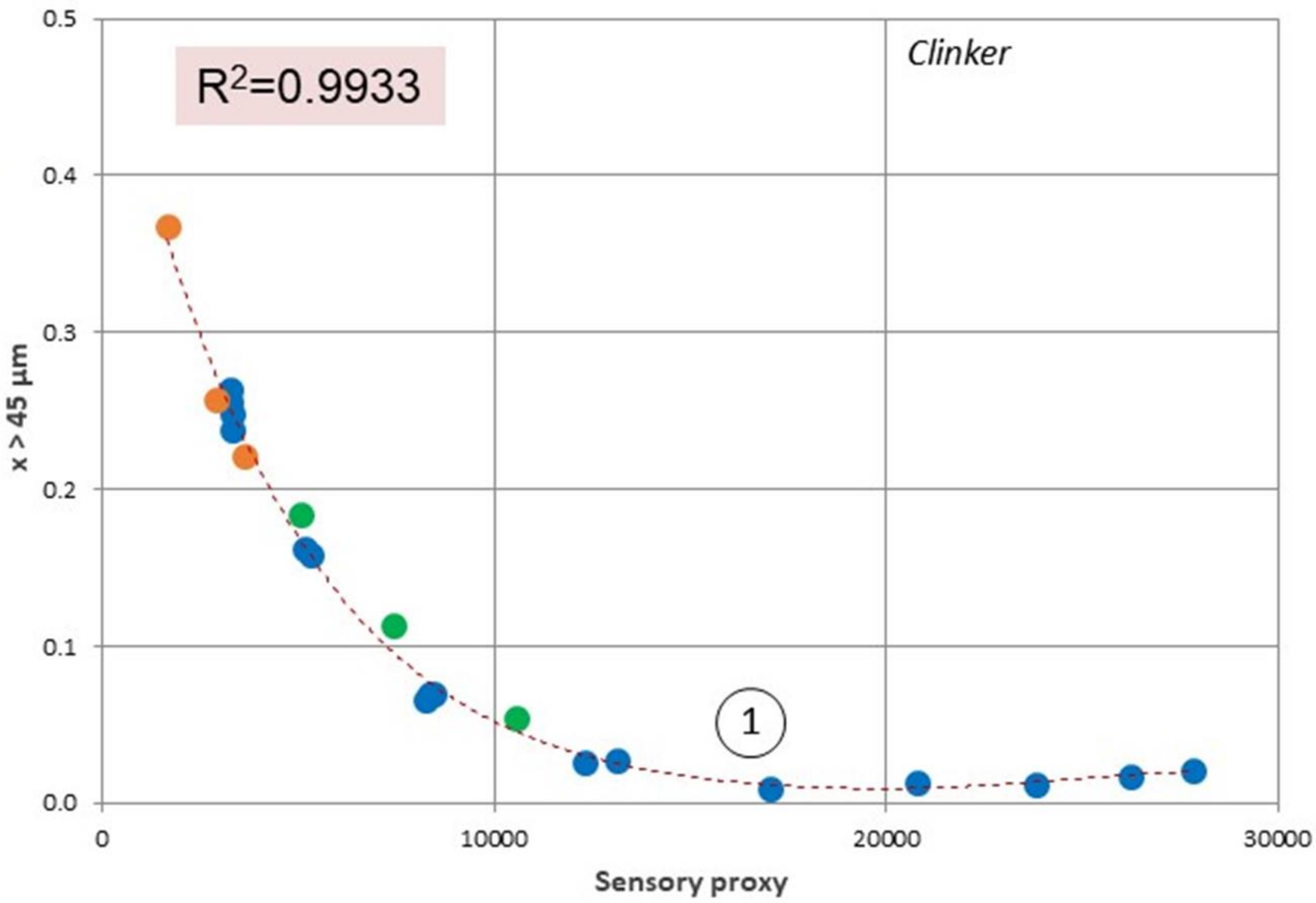
- **Monitoring vessel movement**



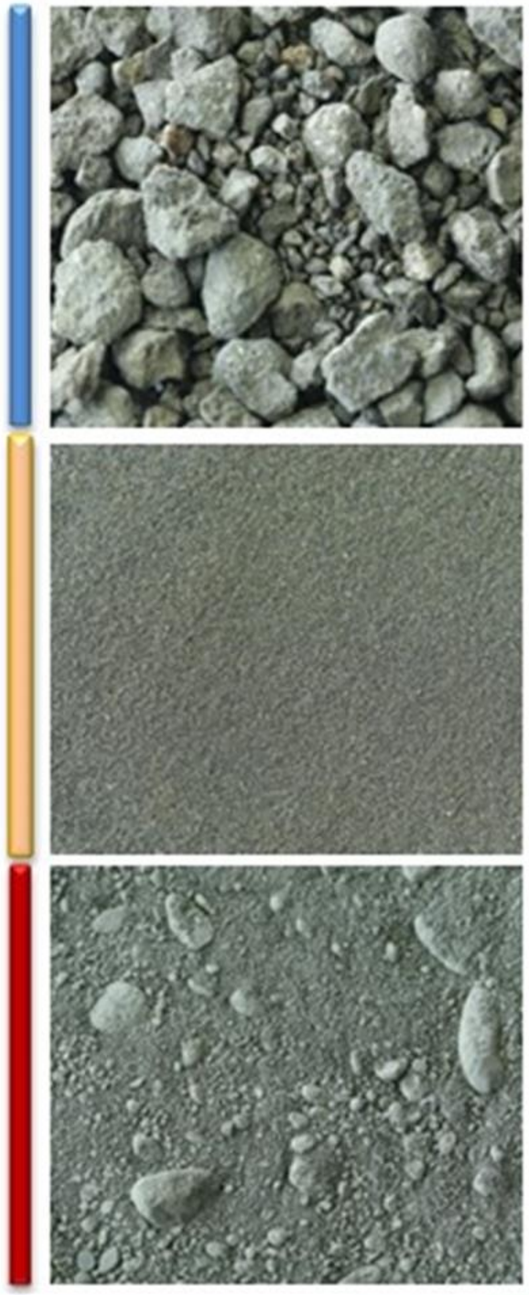
• Sensory data vs. Particle size

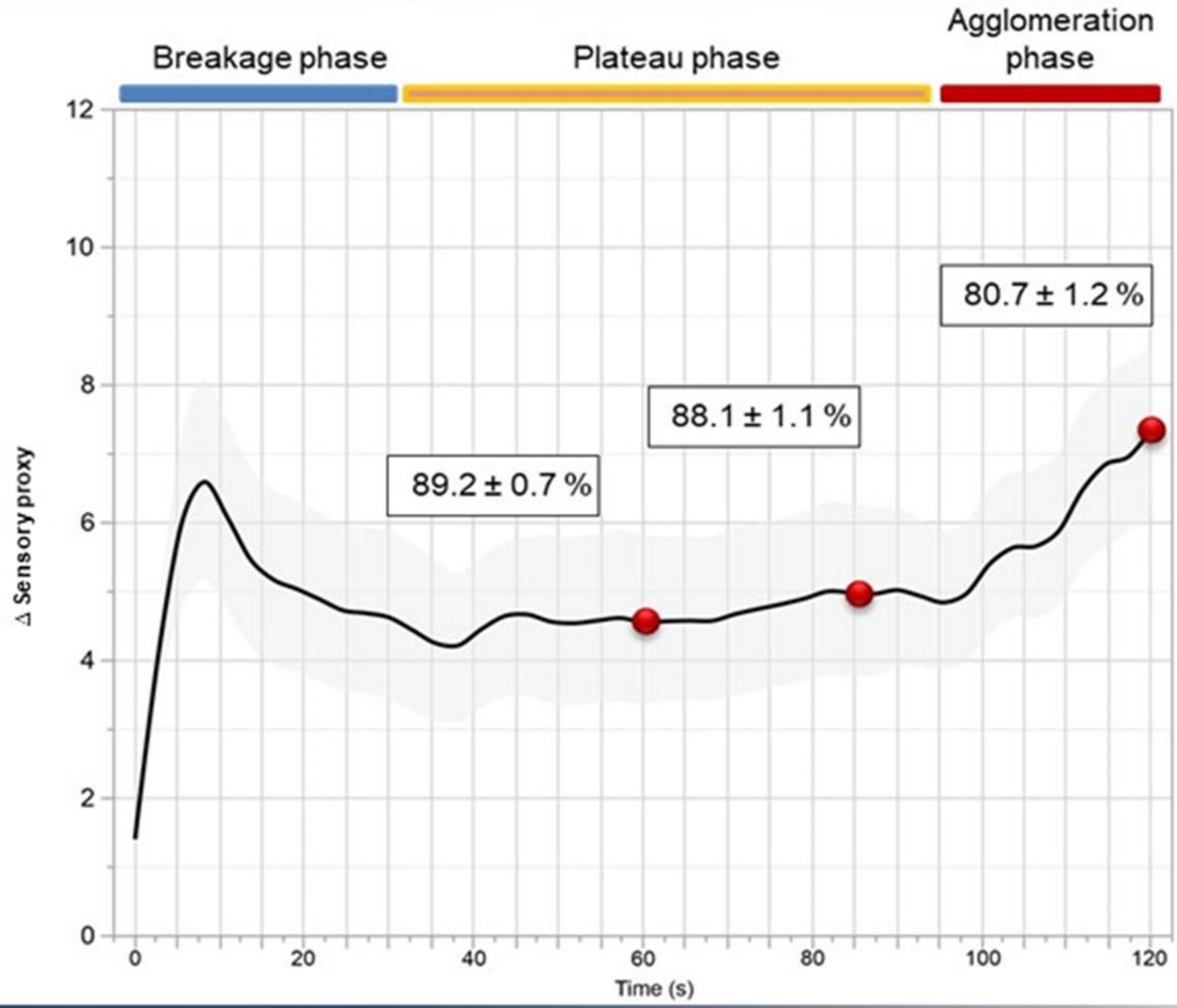
Assessment of grain size distribution by sensory data



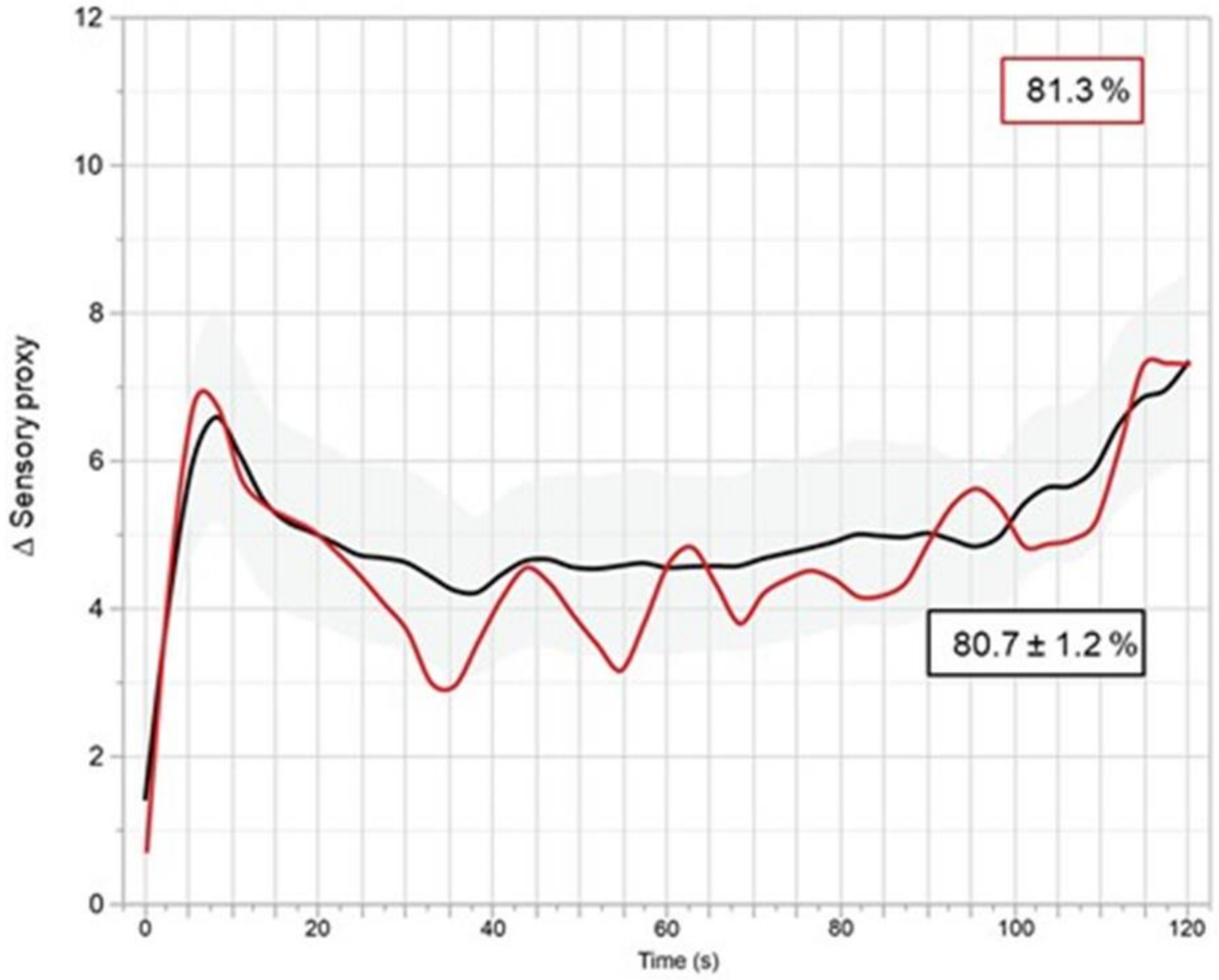


- **Grinding mechanism vs. Particle size**

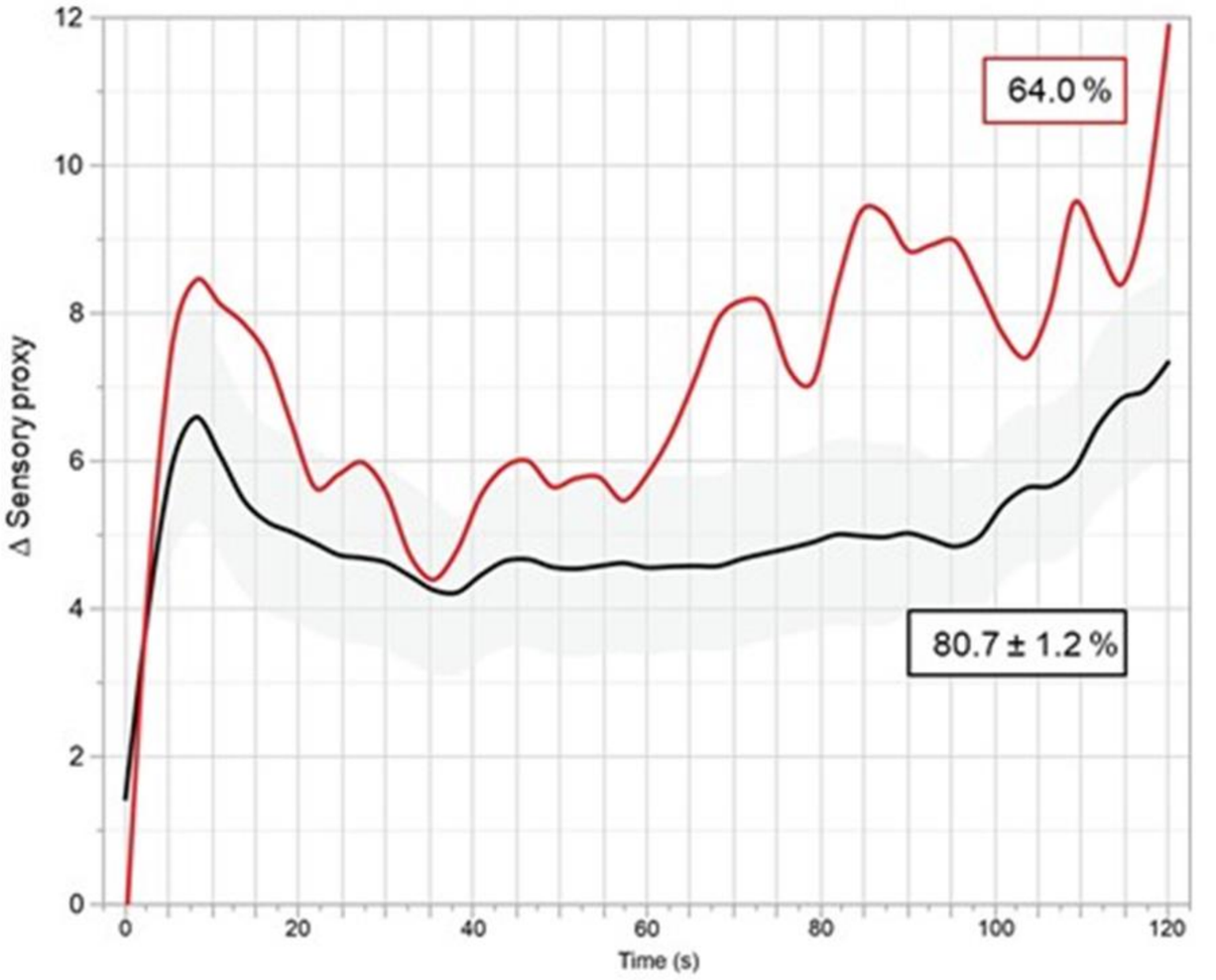




- **Data validation**



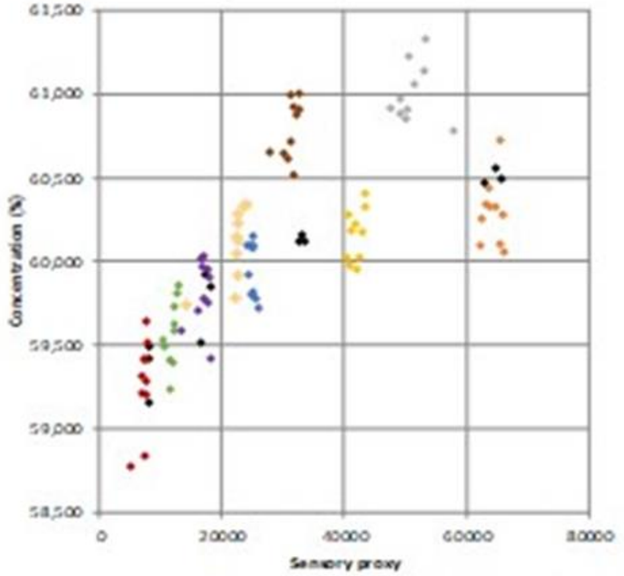
Good sample preparation



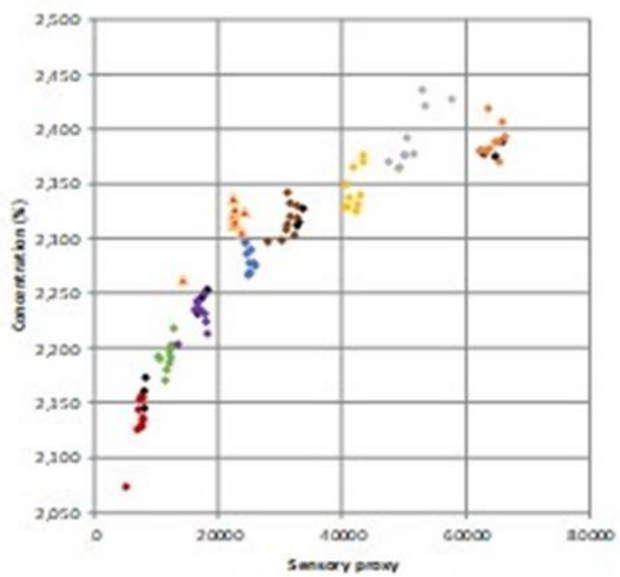
Biased sample preparation

- **Sensory data linked to XRF measurement**

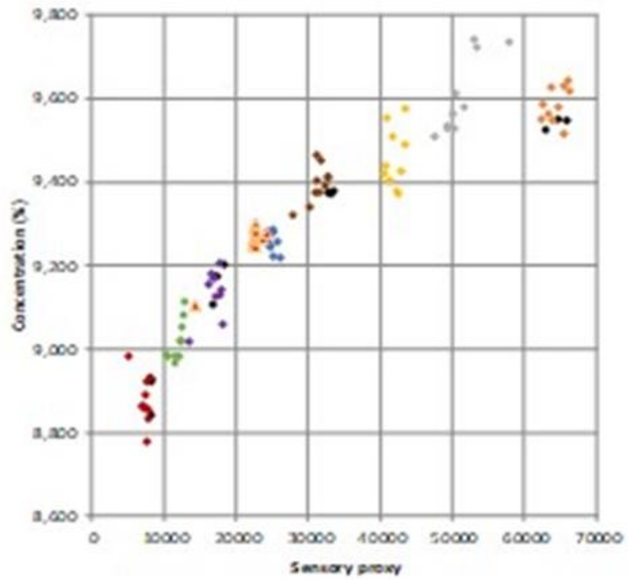
Calcium

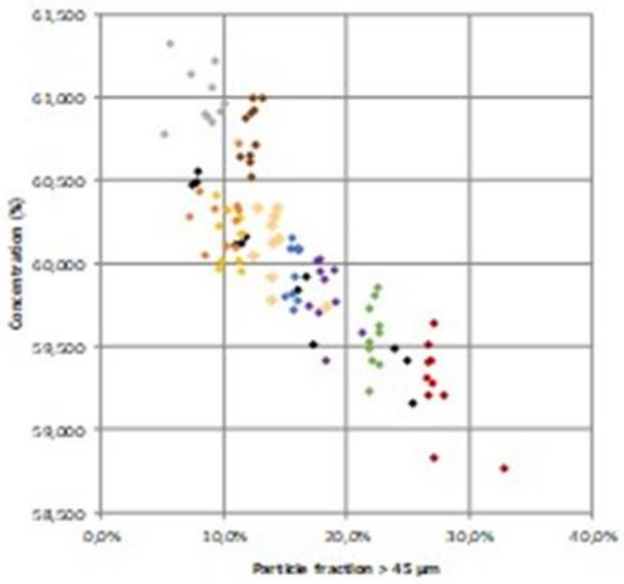


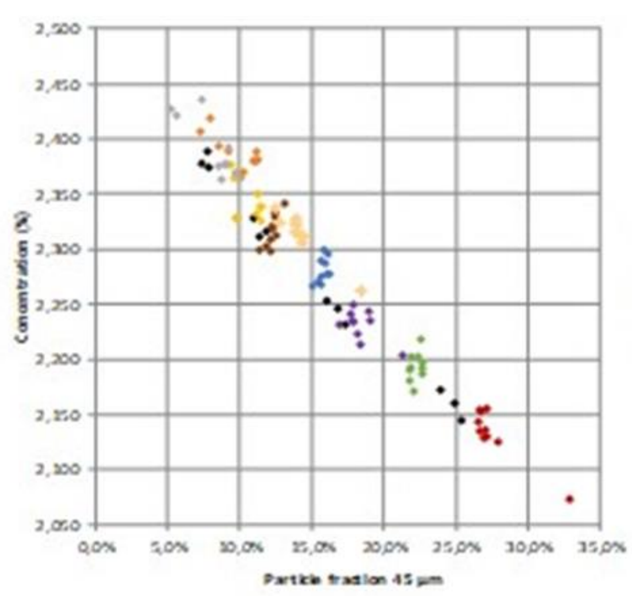
Aluminium

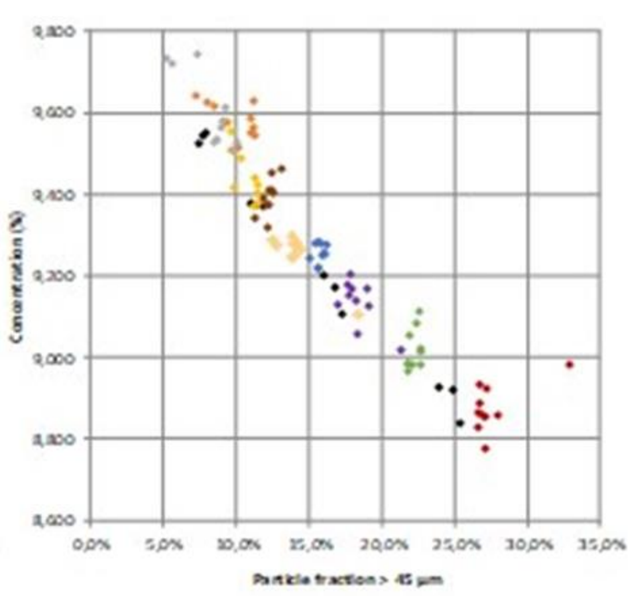


Silica

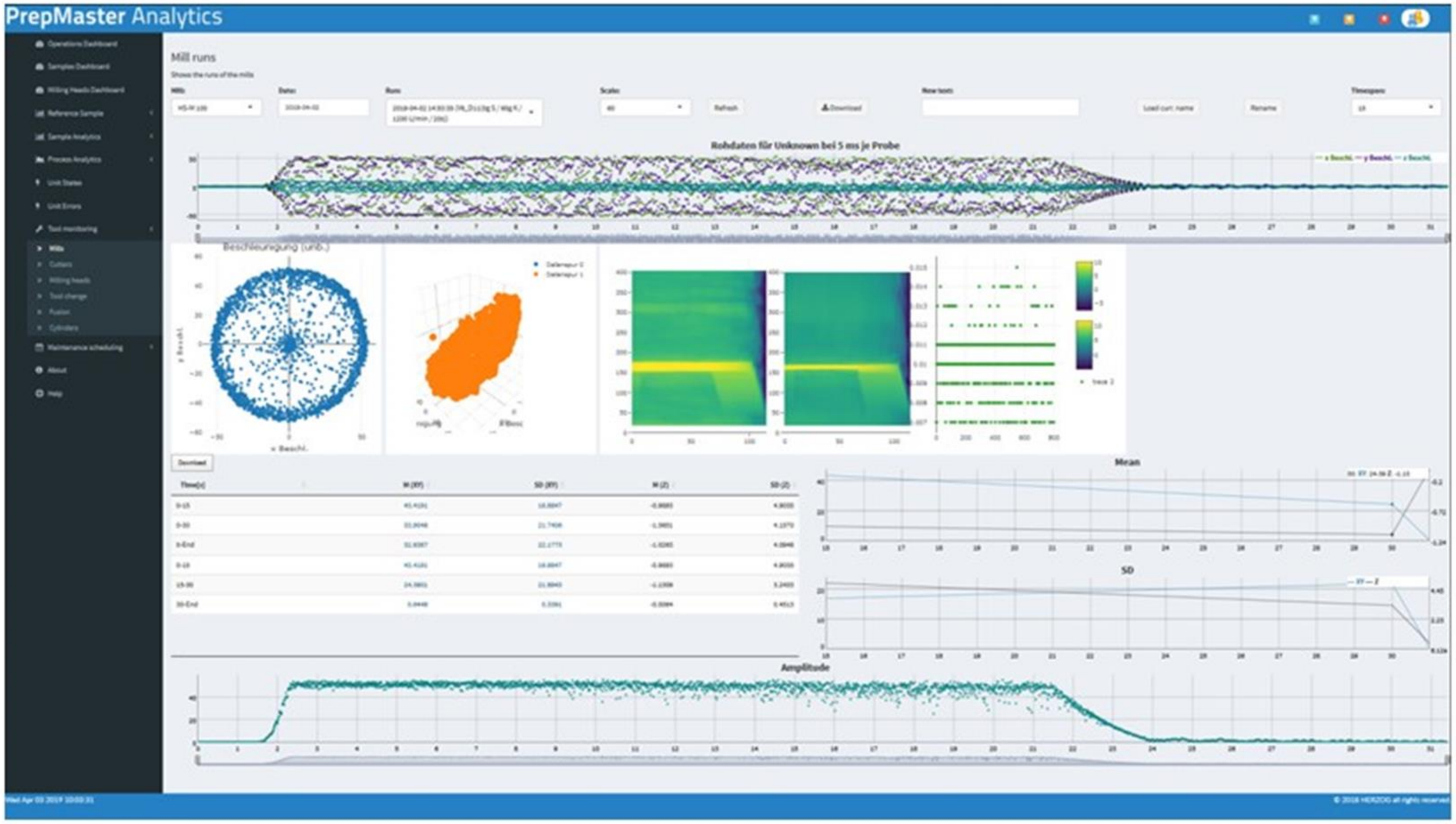




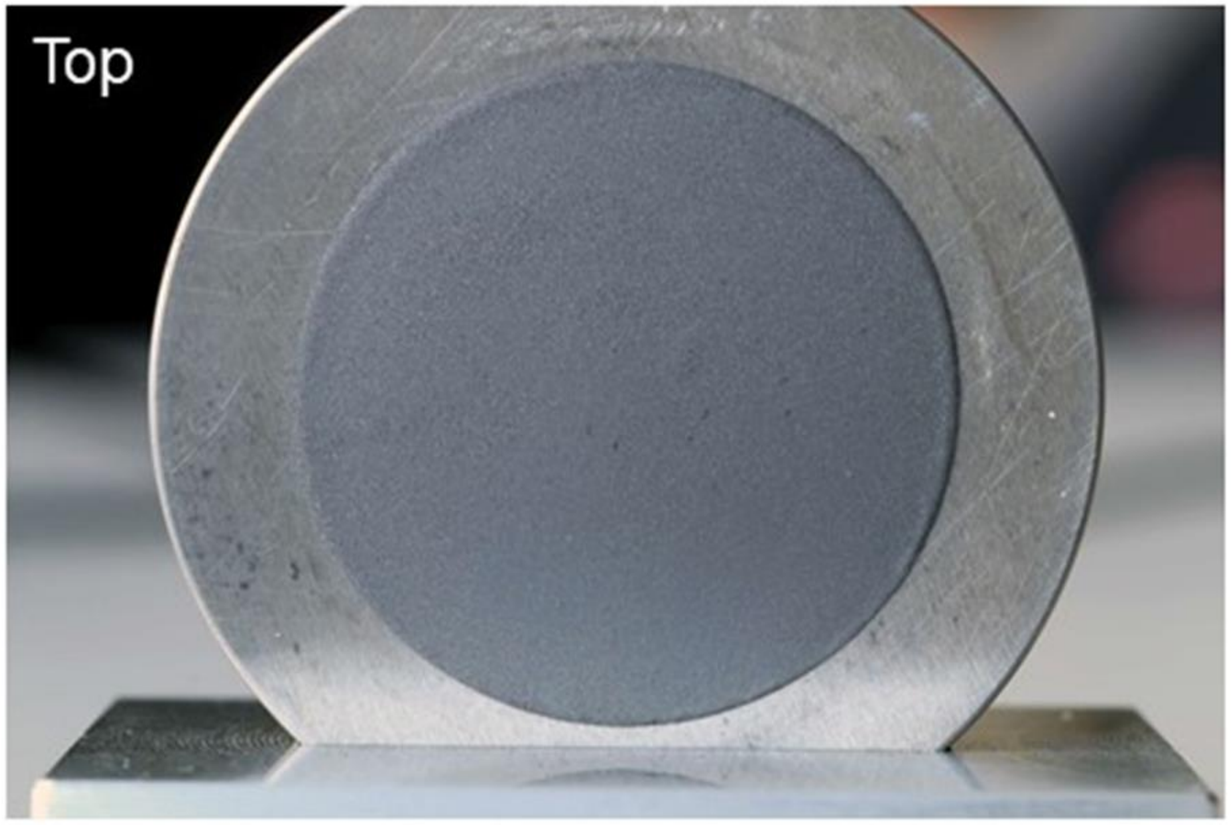


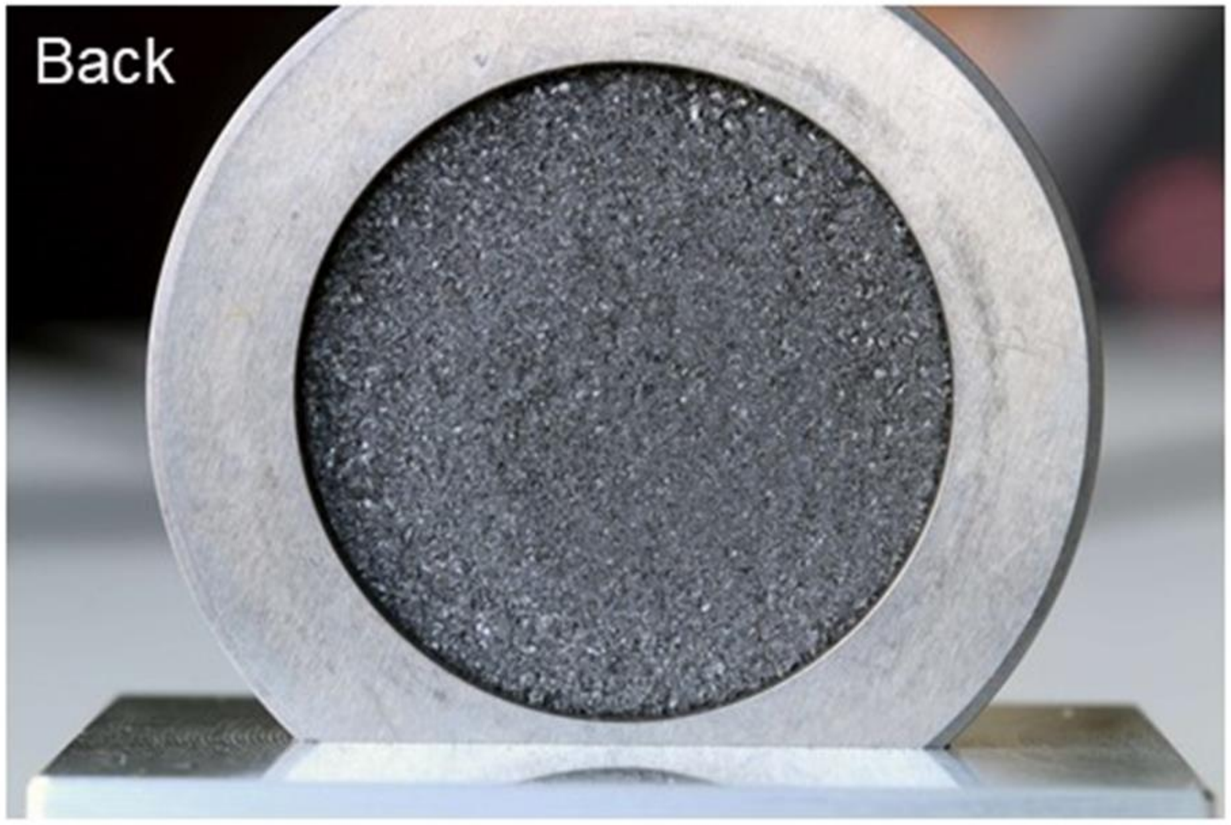


PrepMaster Analytics

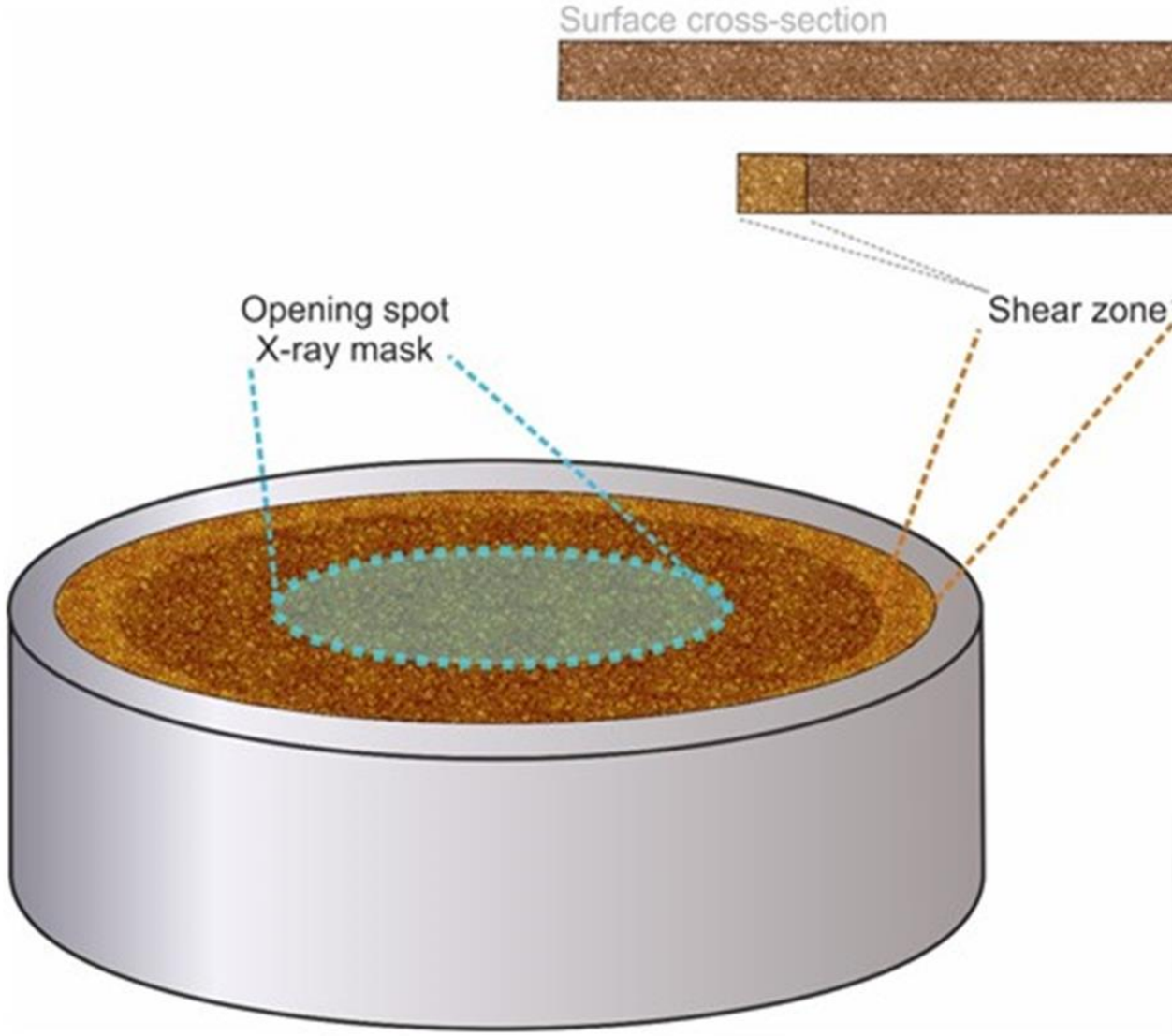


- **Segregation during pressing**

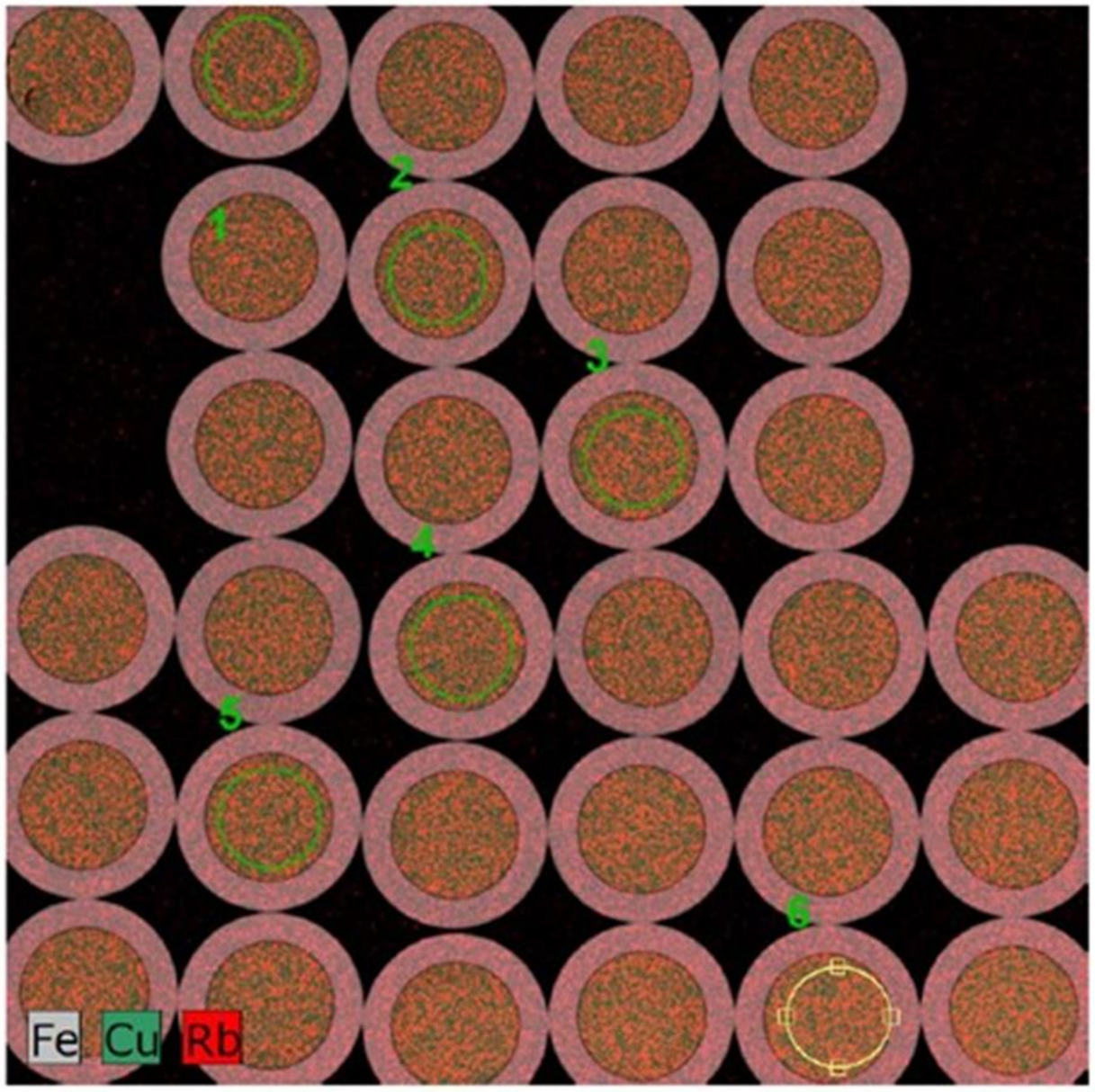


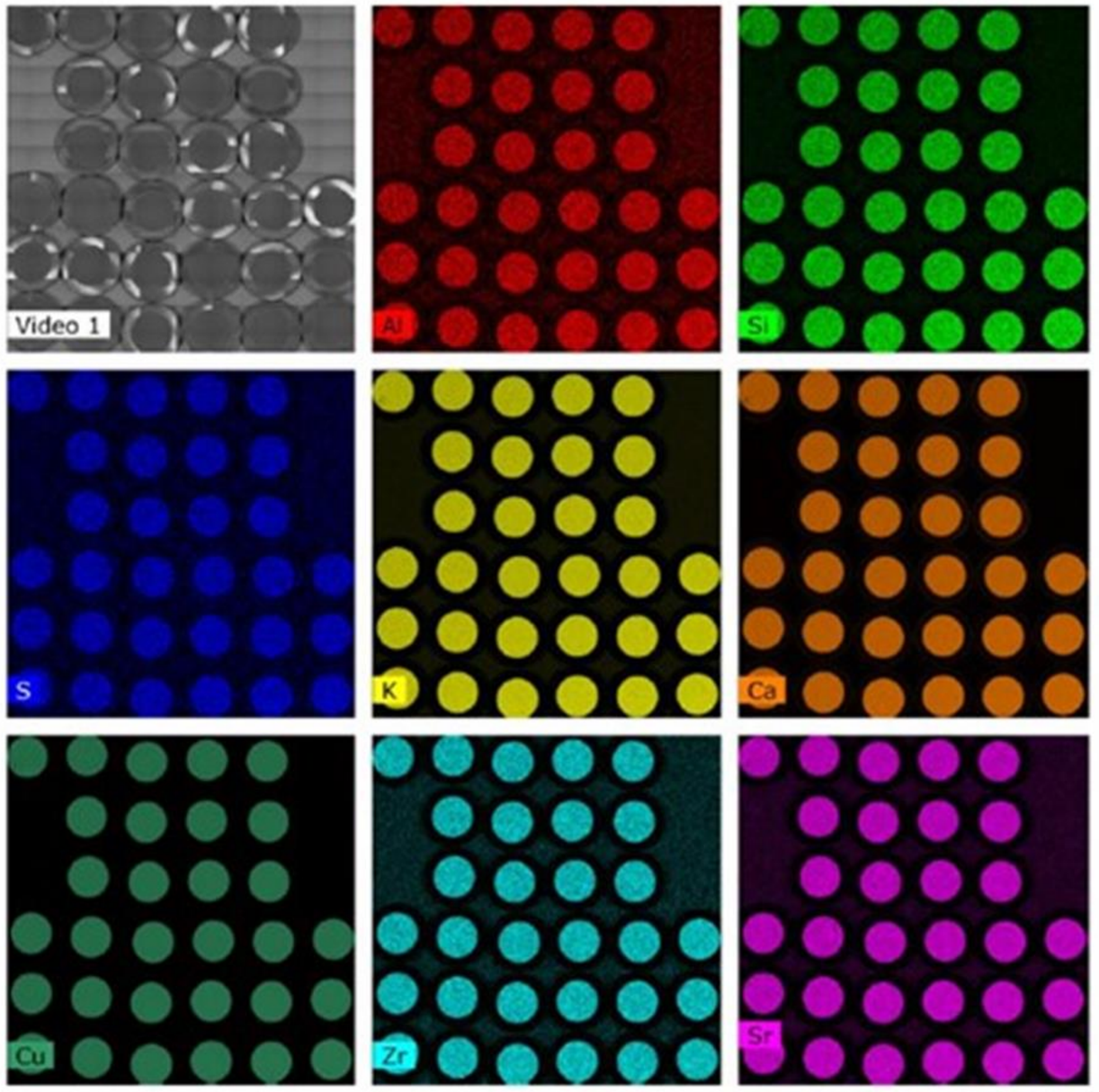


- **Shearing zone at edge of pressed pellet**

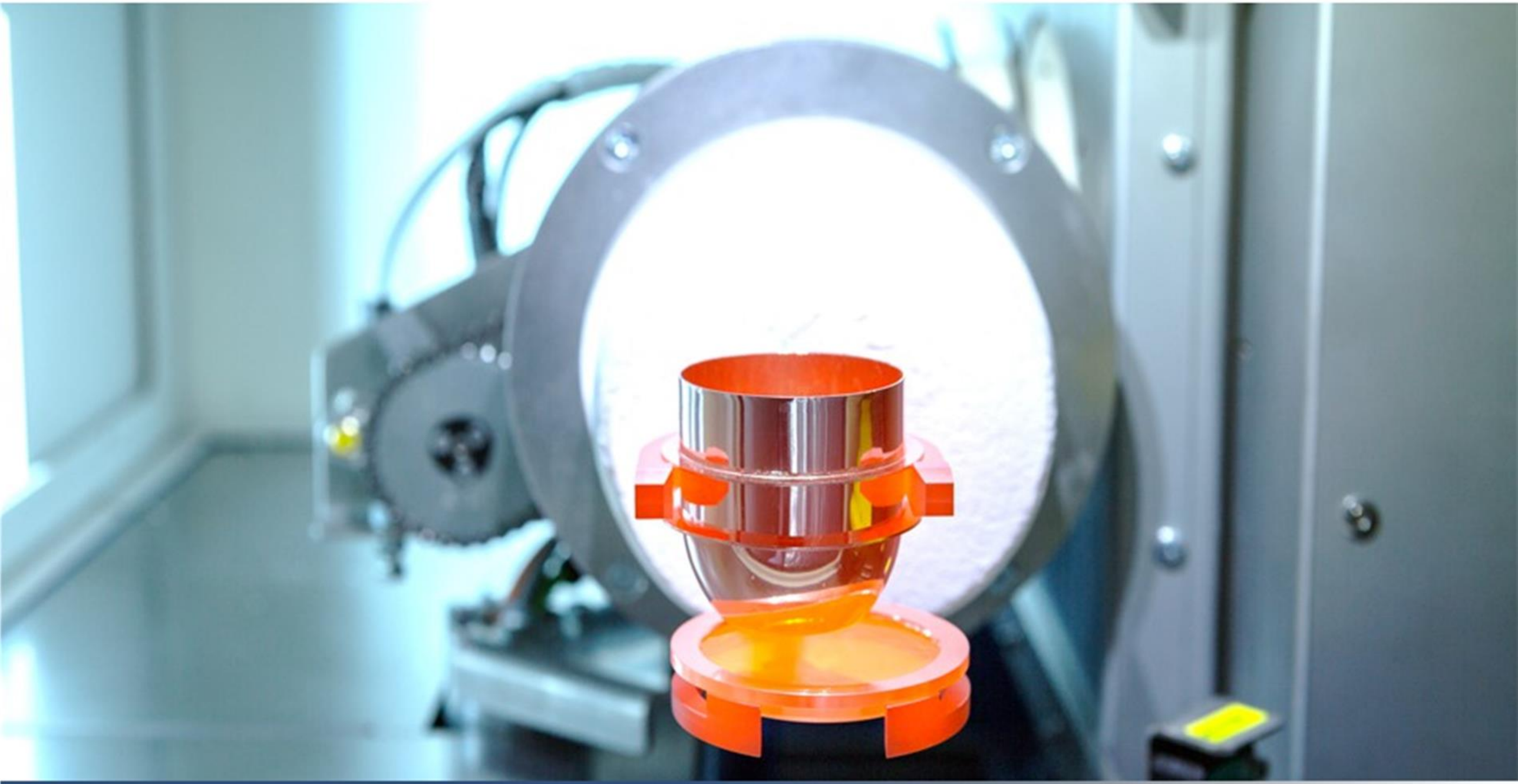


- **Quality measure – M6 Jetstream**

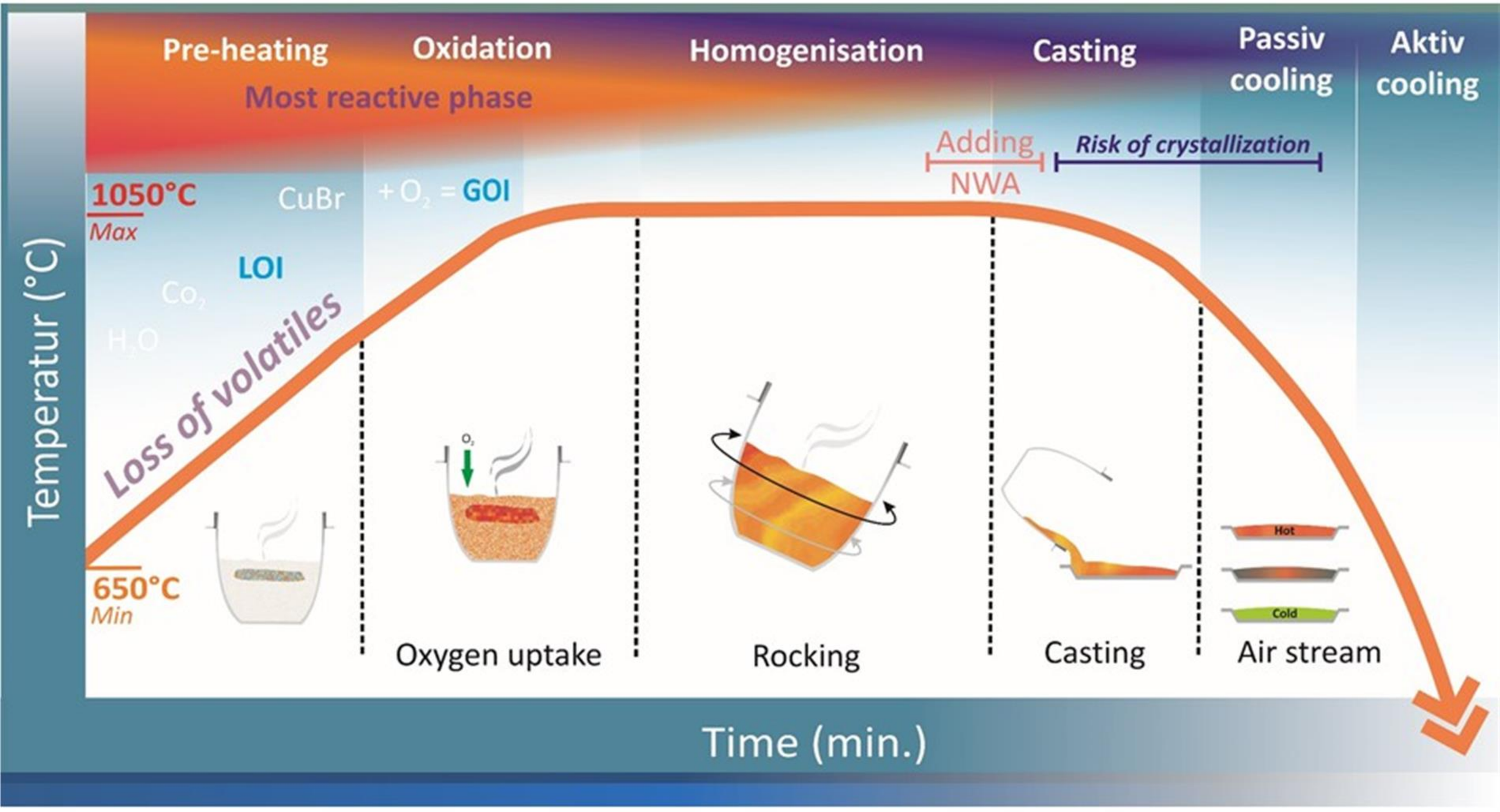




- **Fusion**

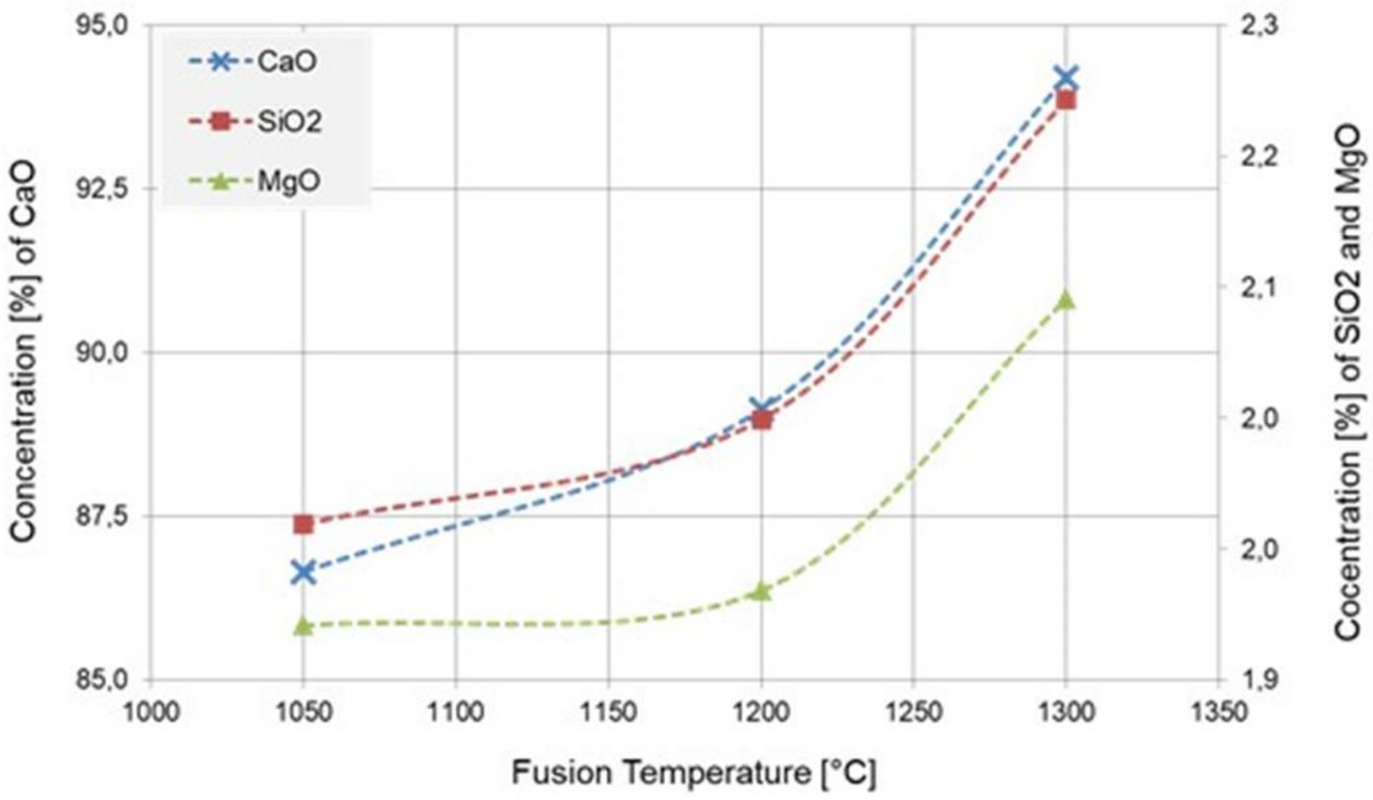


- **The fusion process**

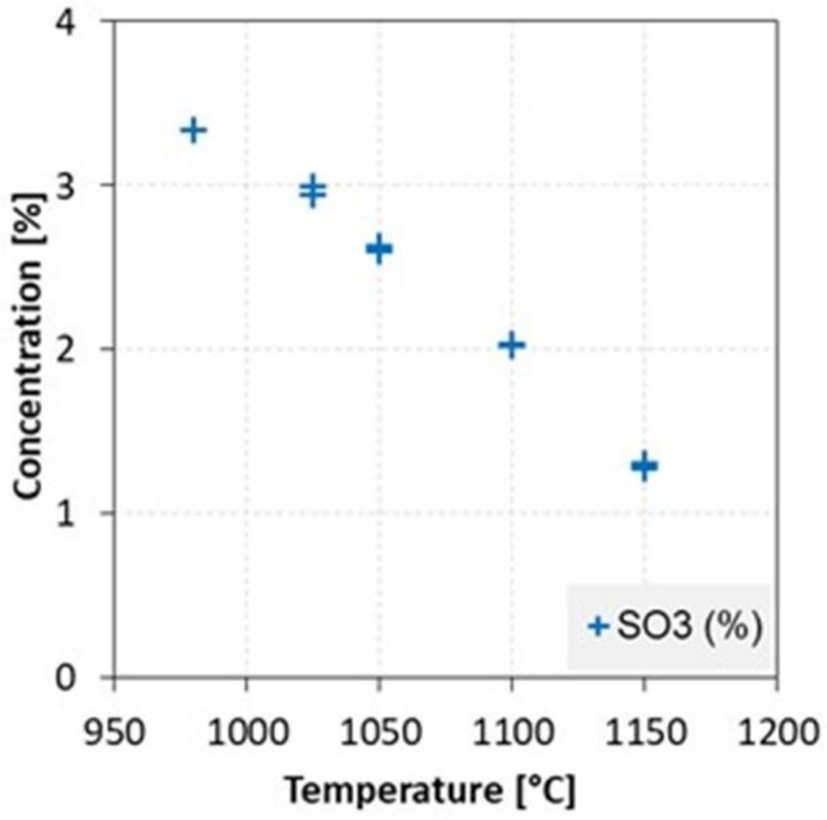


- **Influence off temperature on counting rate**

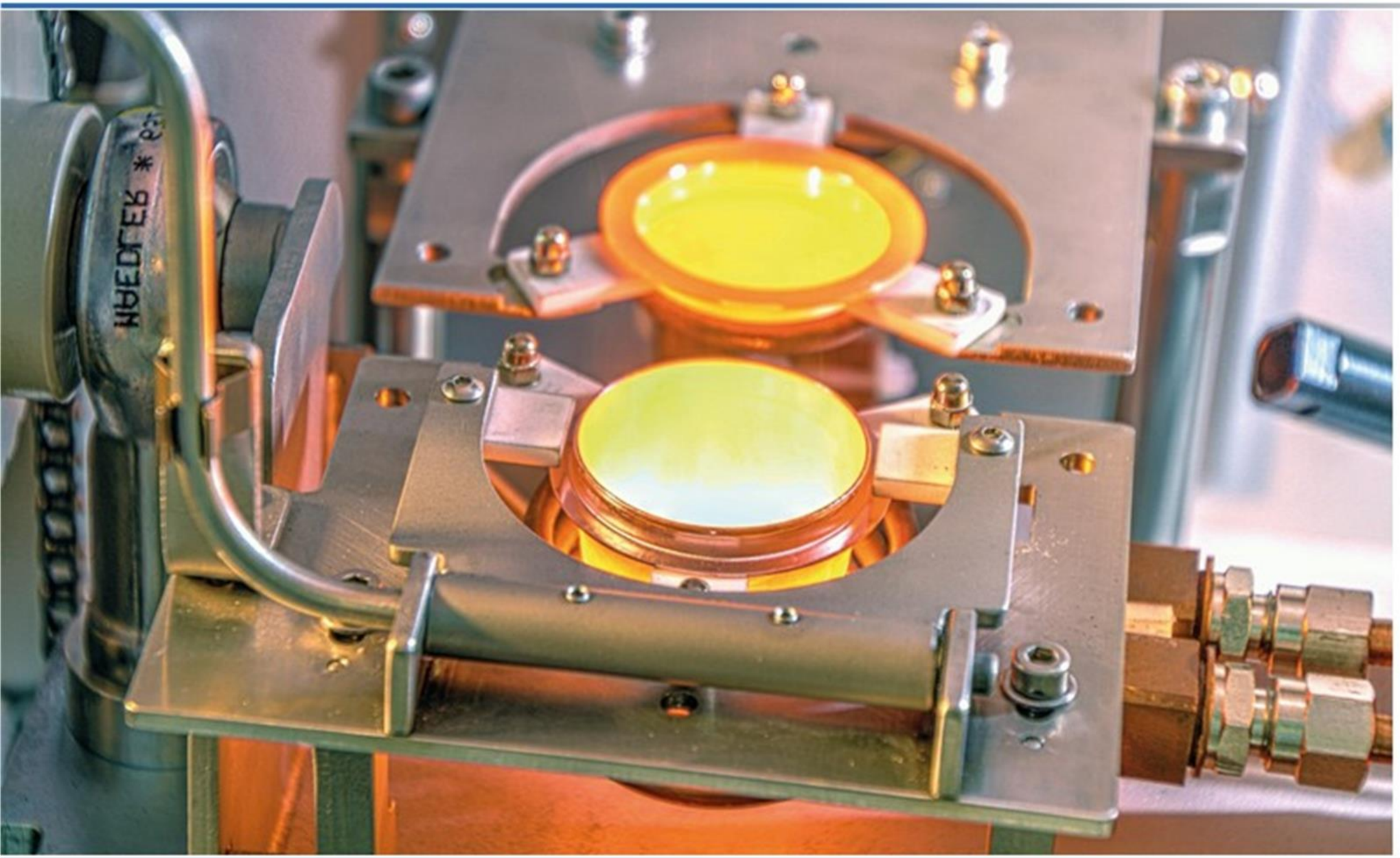
Loss of flux



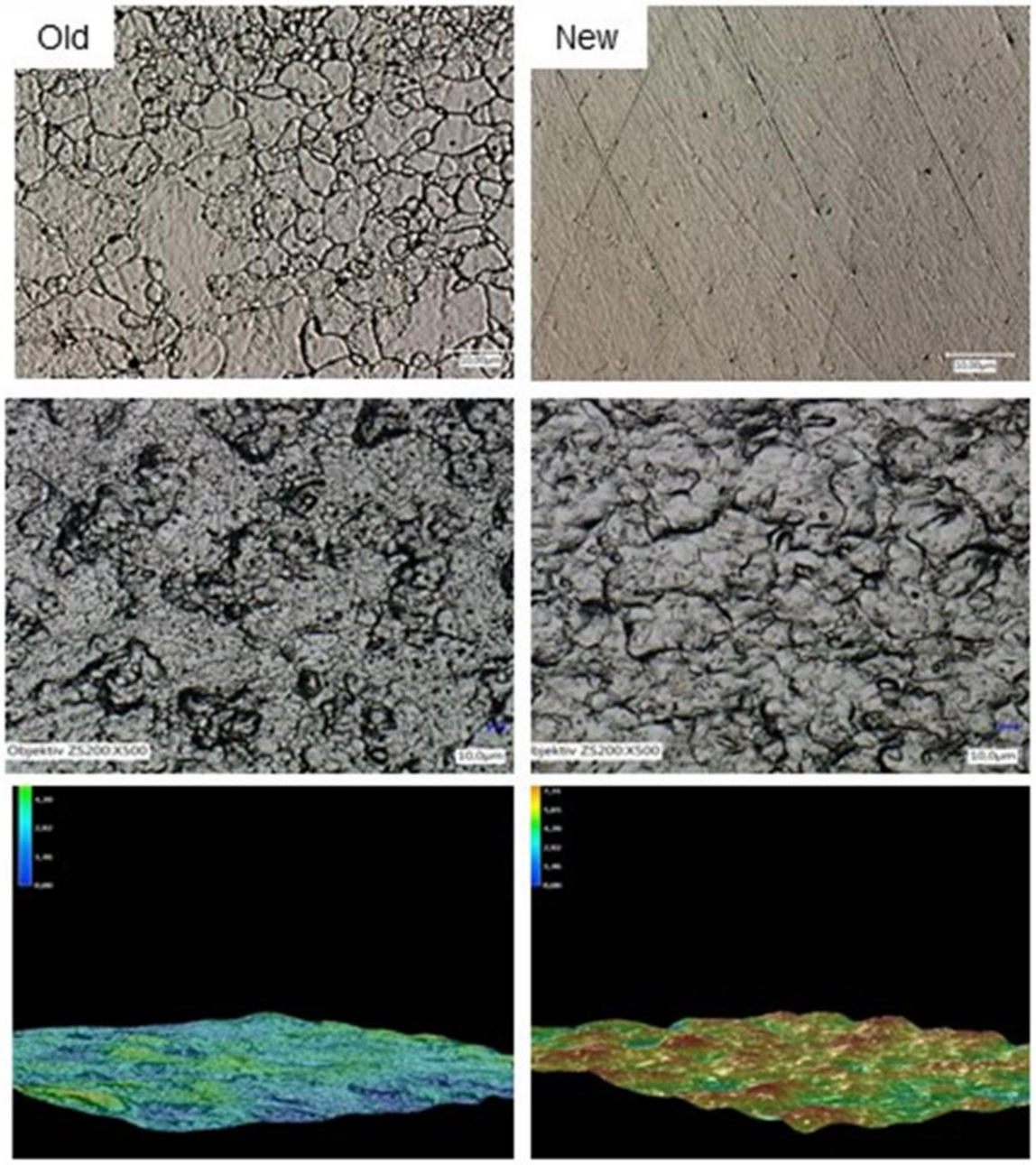
Loss of volatile elements

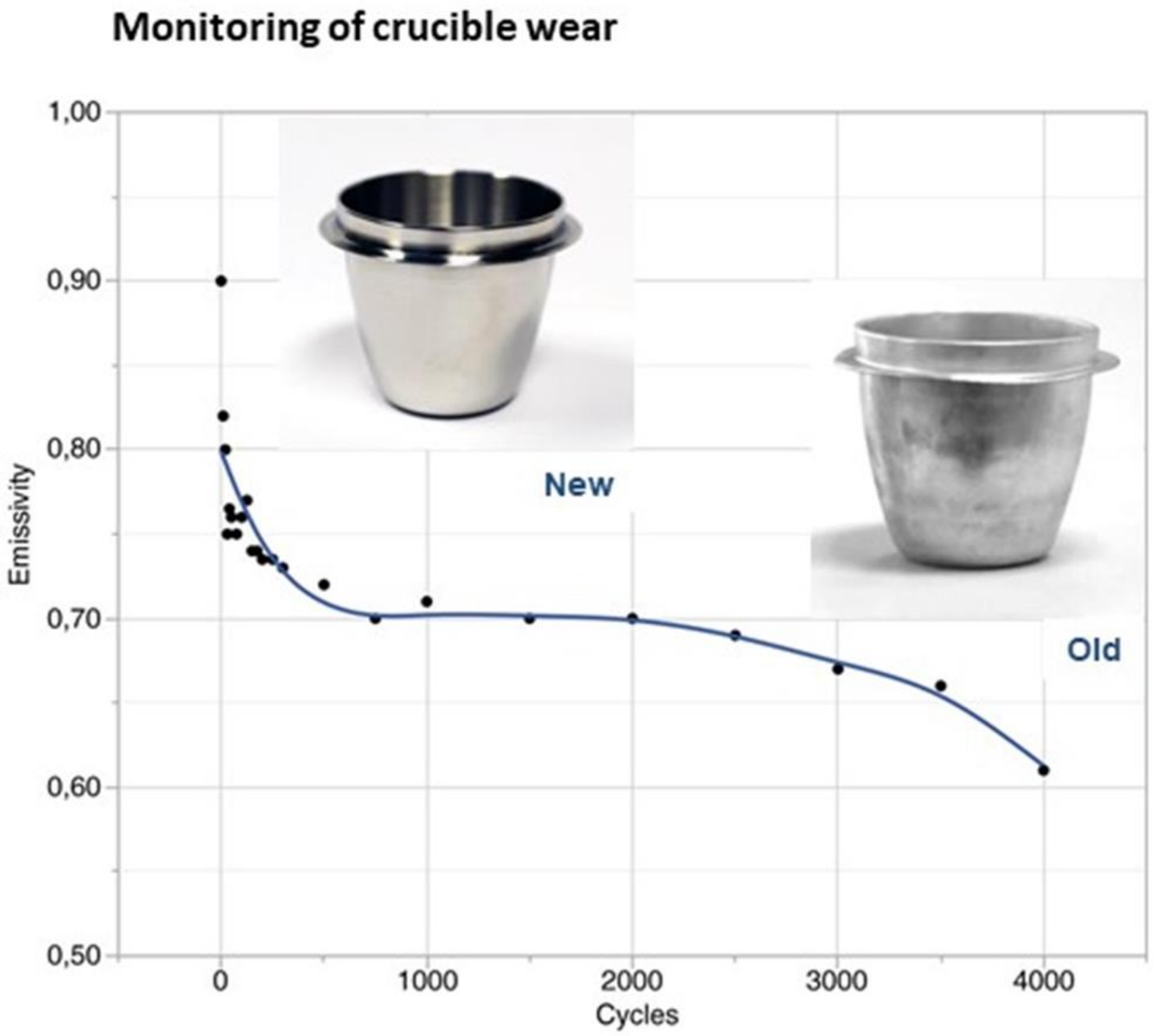


- **Induction fusion systems – IR temperature measurement**

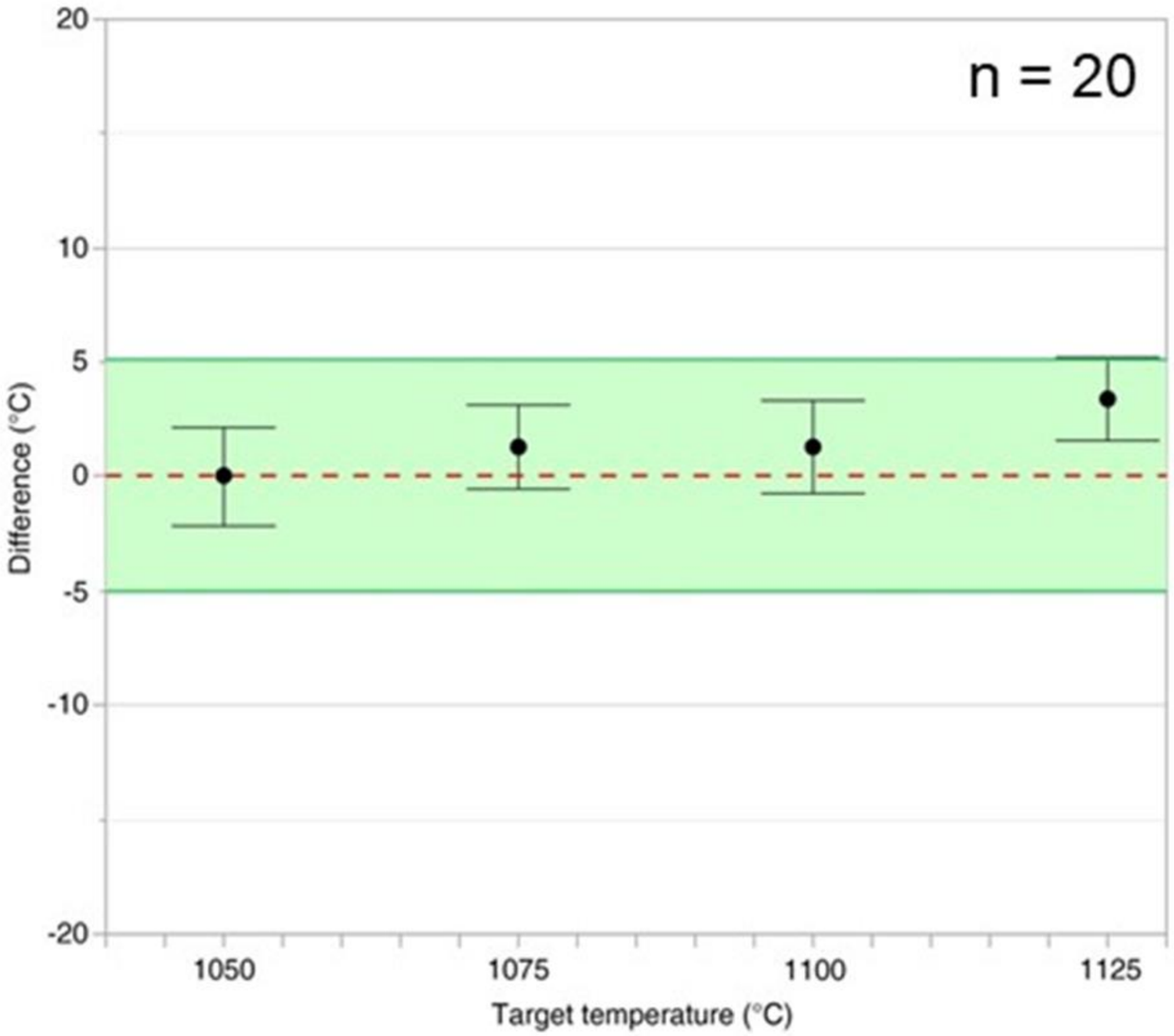


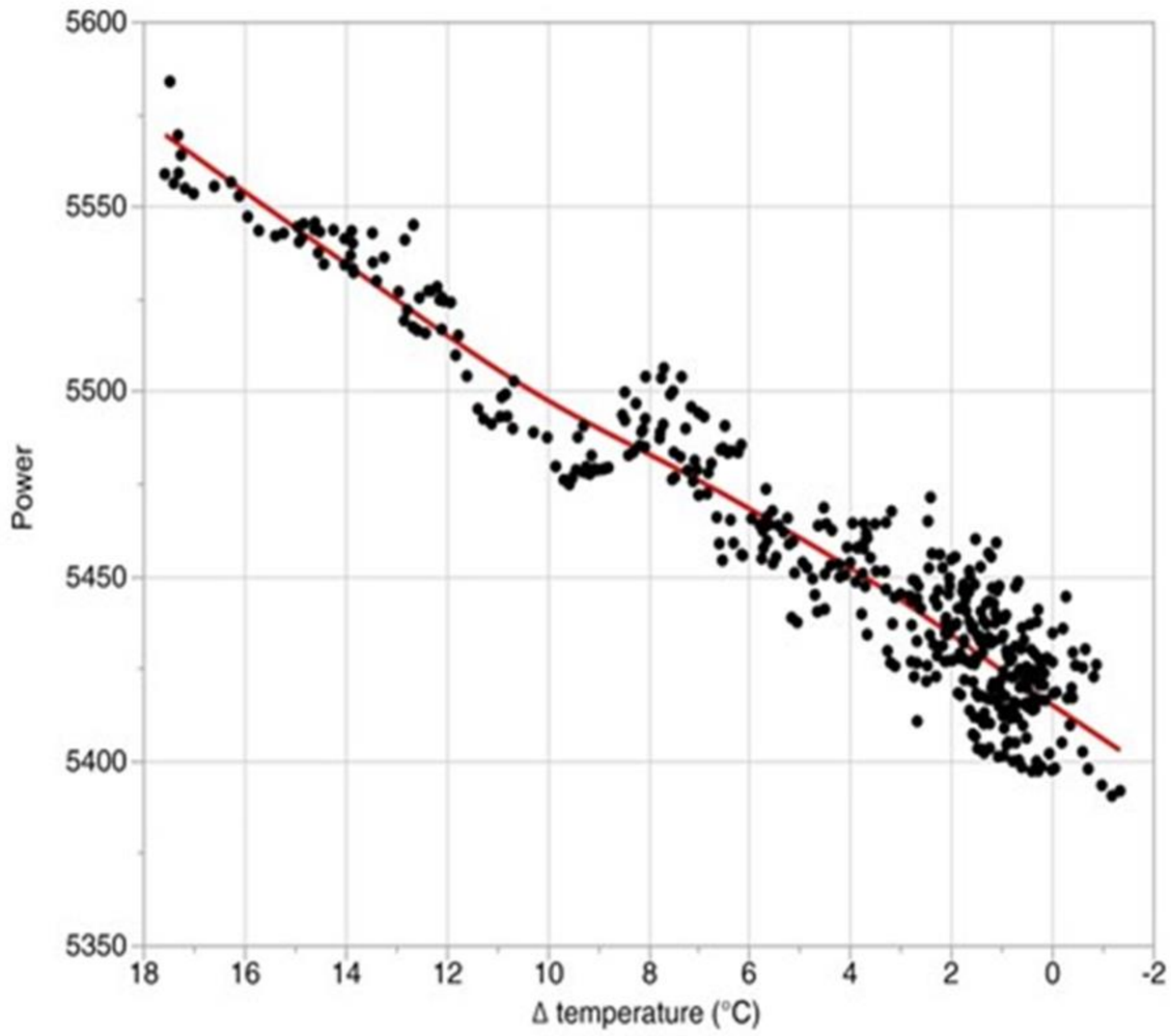
• Pt-Au - Surface changes



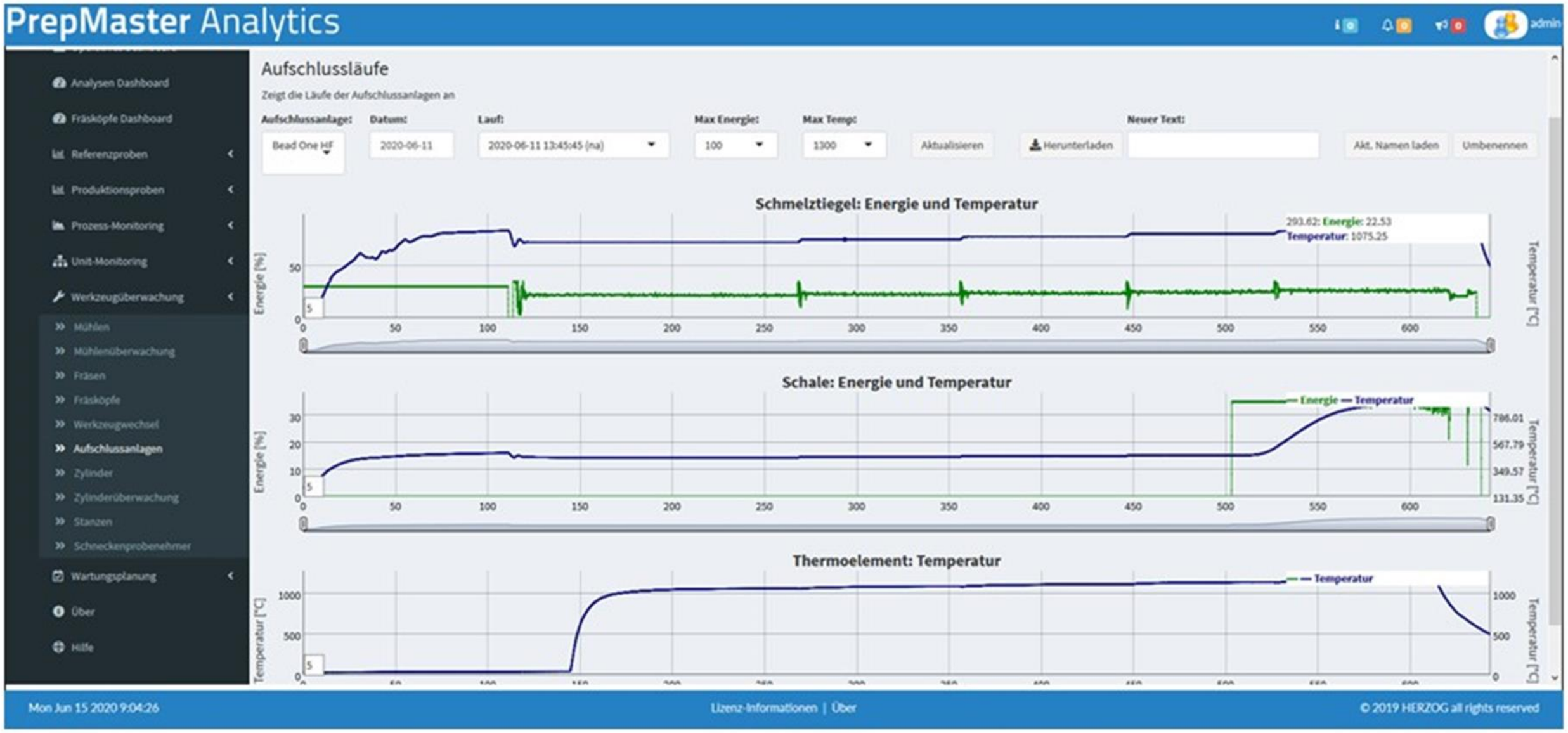


- **Temperature stability and controlling**

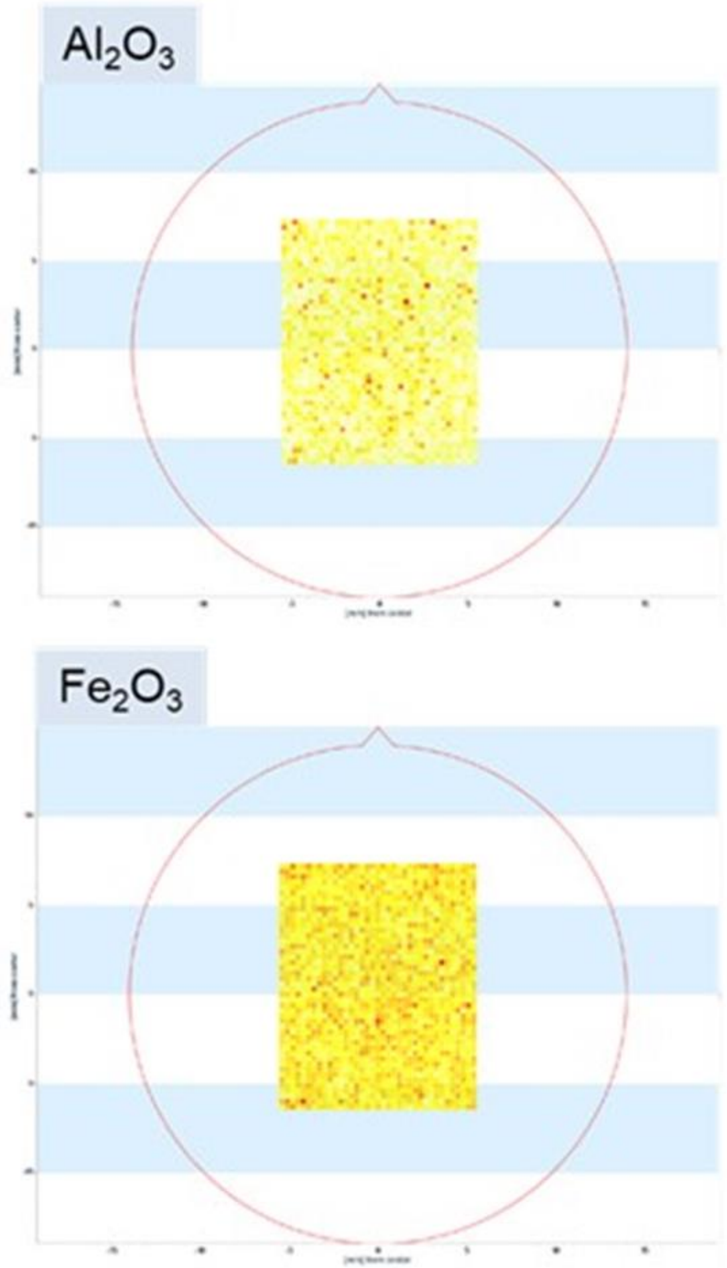


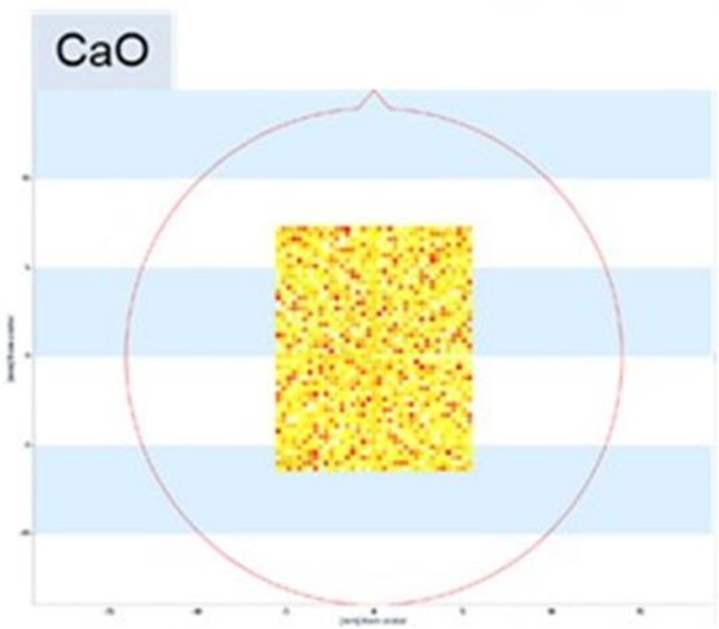


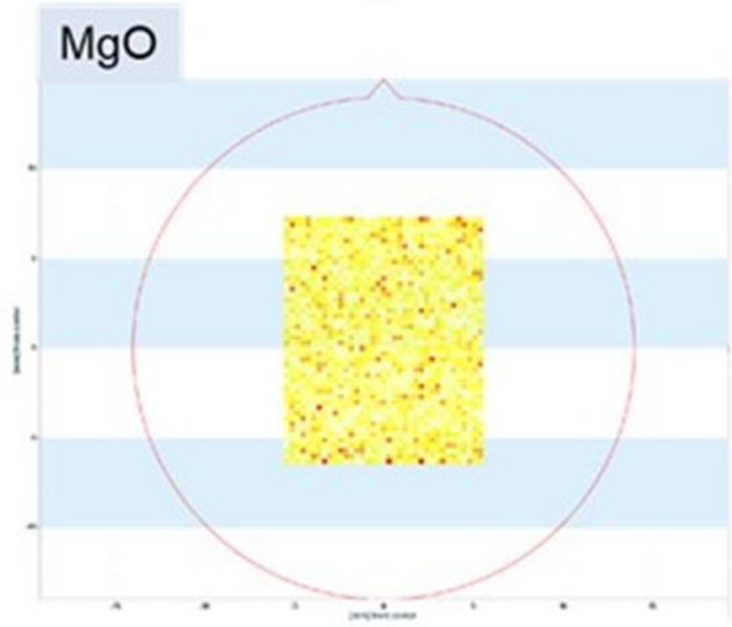
PrepMaster Analytics

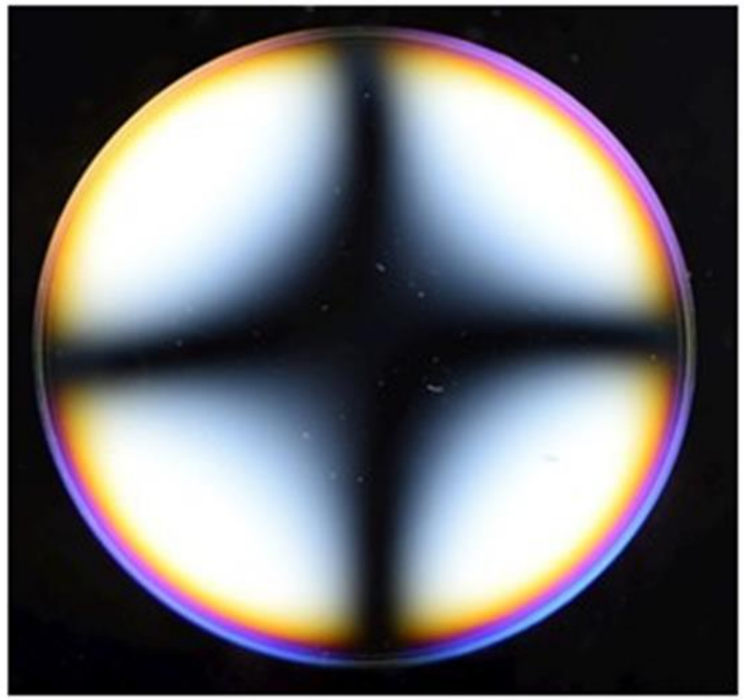


• Quality measure – M4 Tornado









Strain in the bead

BRUKER

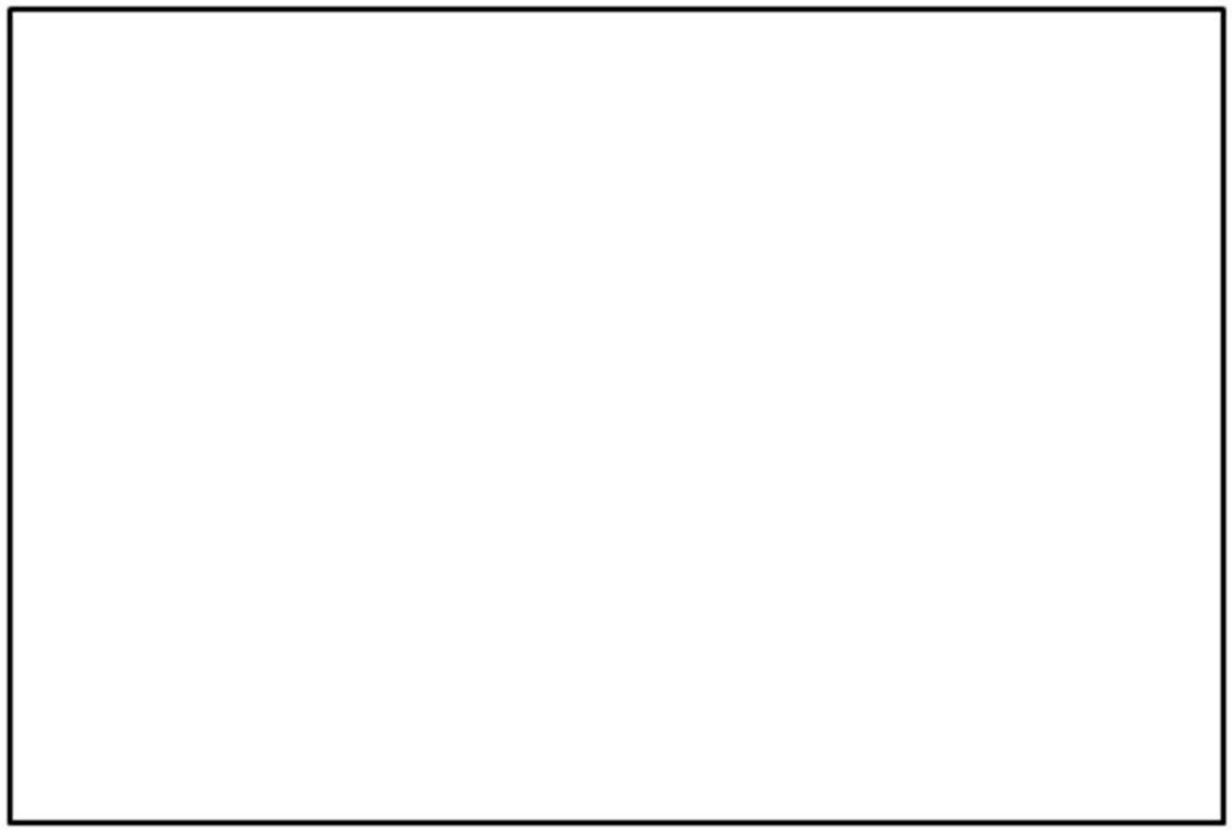
COLIN SLATER

Research & Development

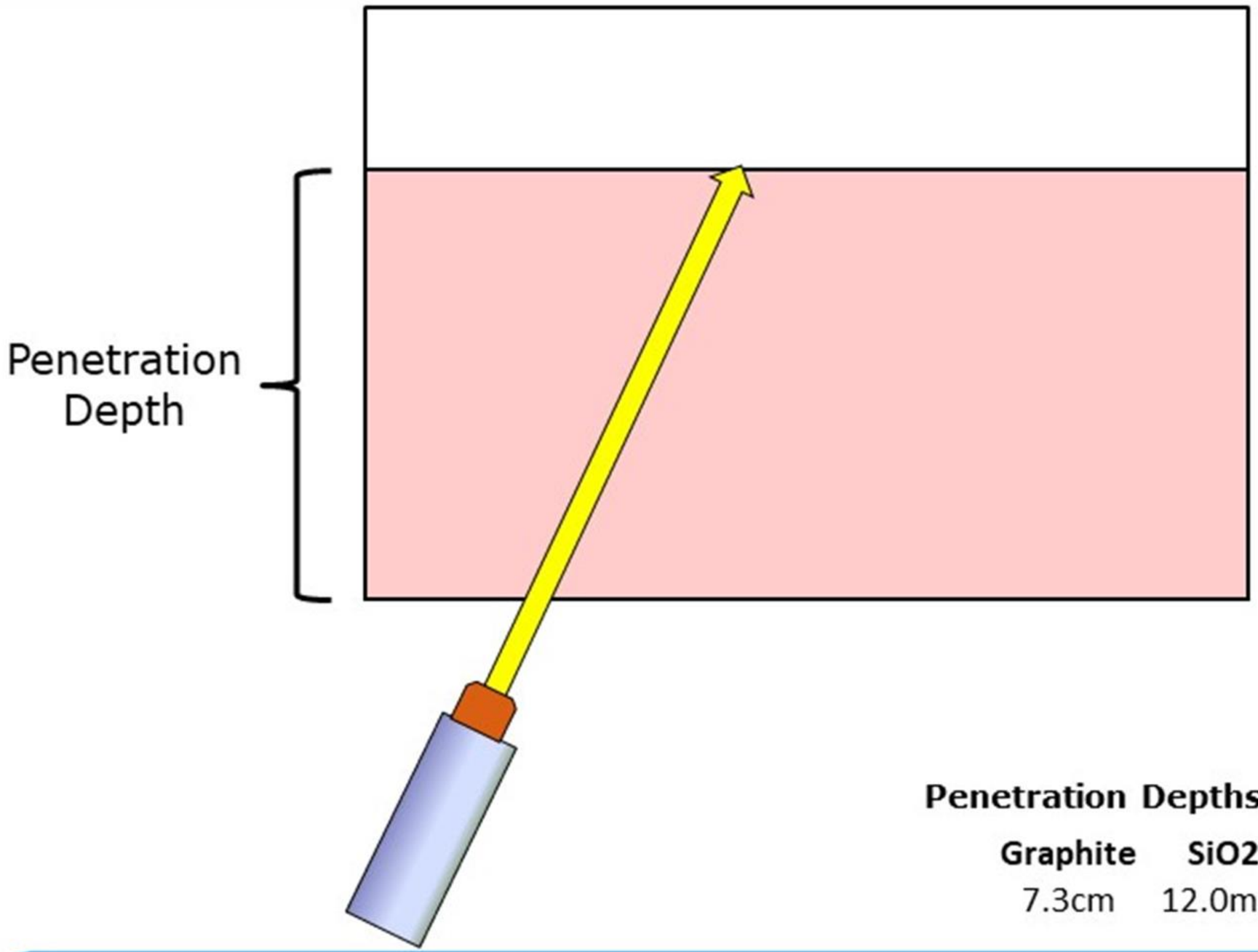

S8 TIGER


S8 TIGER

Sample Preparation Effects



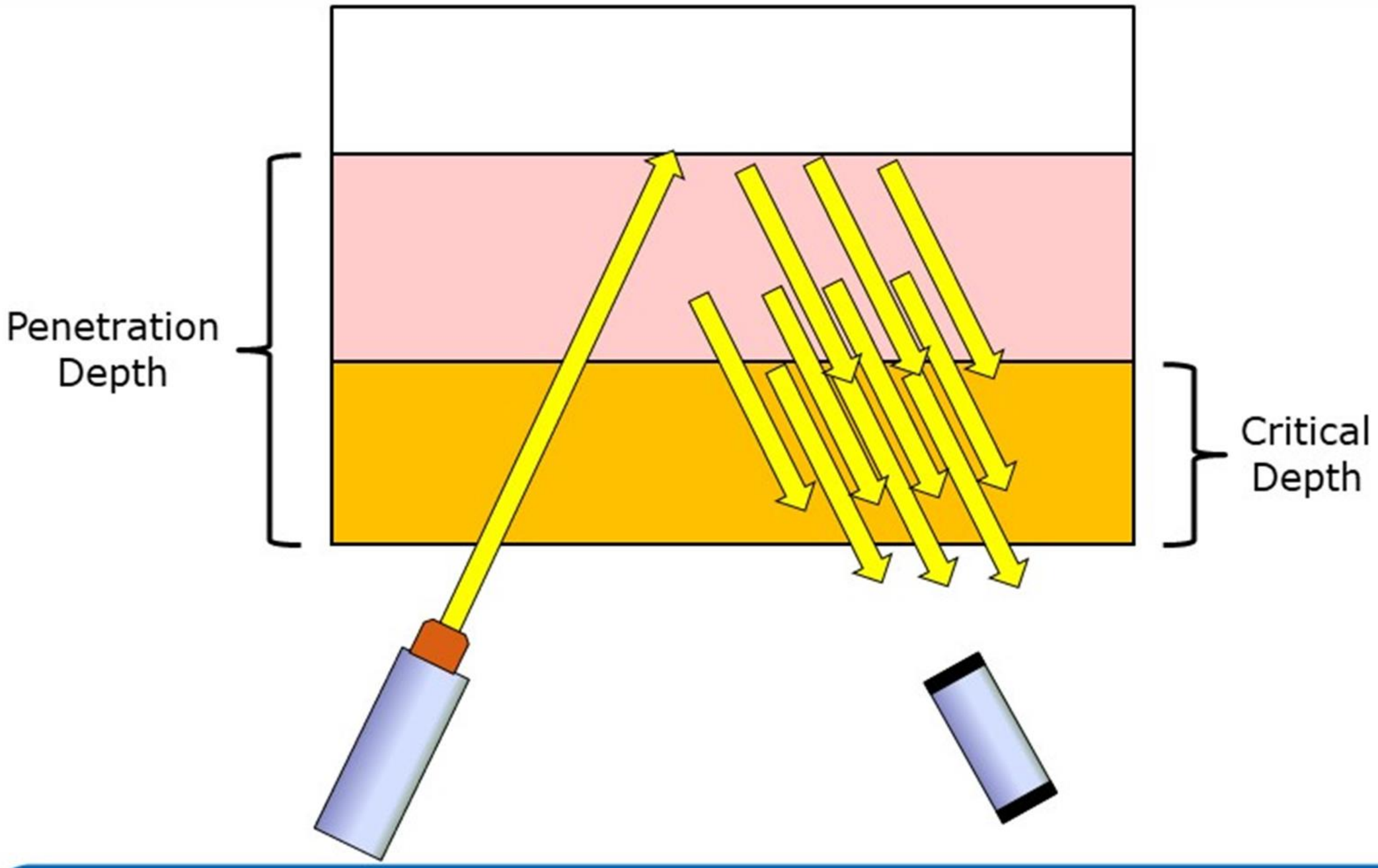
Sample Preparation Effects



Penetration Depths for Rh-KA1 (20.2keV)

Graphite	SiO2	Fe	Pb
7.3cm	12.0mm	355.0µm	70.0µm

Sample Preparation Effects



Sample Preparation Effects

A quick experiment:

- Sample: Quartz Sand
- Preparation Equipment: Herzog HSM Manual Pulverising Mill, with Tungsten Carbide Grinding Vessel
- Instrument: S6 JAGUAR (Benchtop WDX)

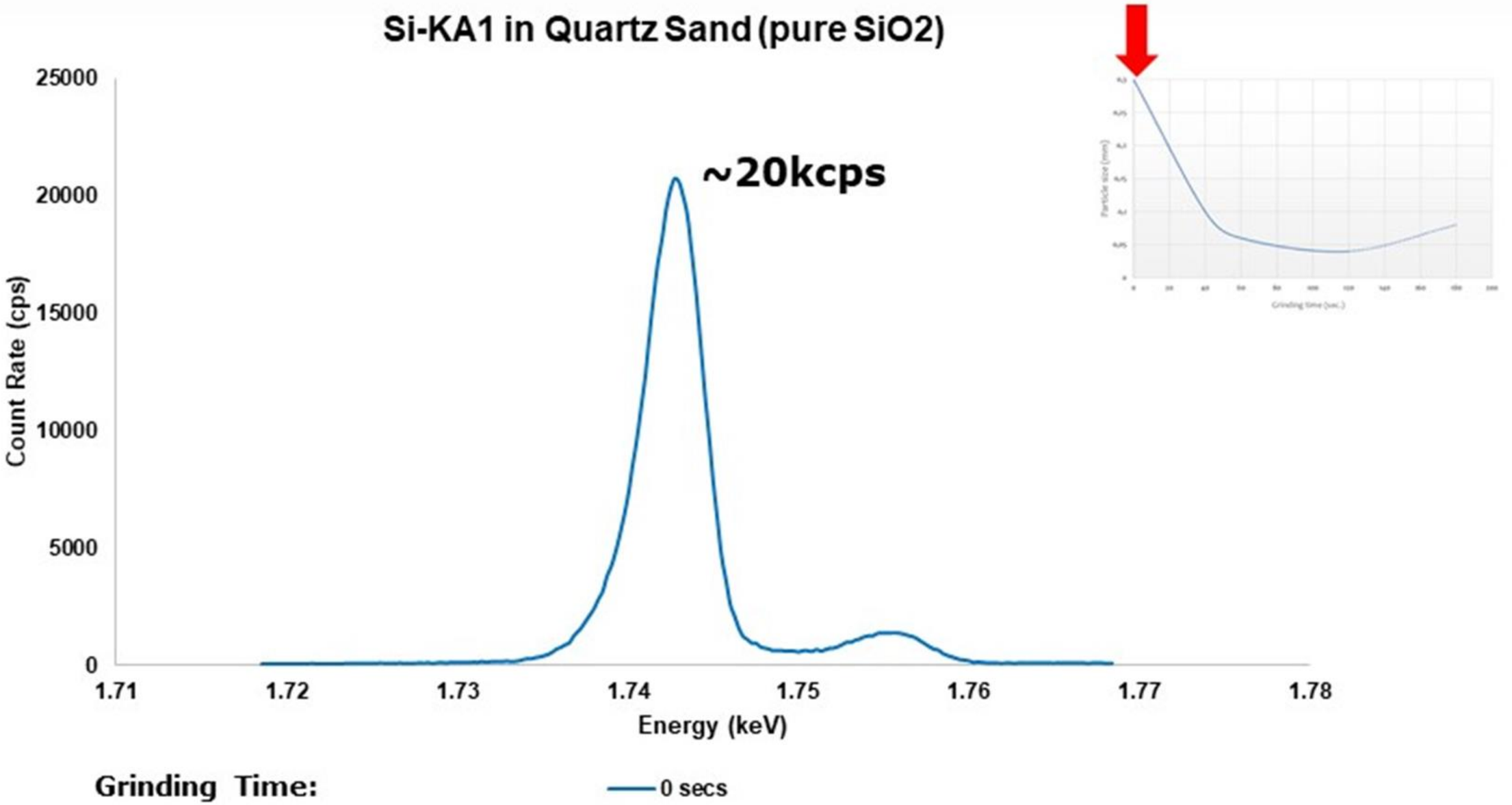
- Prepared Specimens
  1. Quartz Sand, "Out of the Box"
  2. Quartz Sand, 10s Grinding
  3. Quartz Sand, 20s Grinding
  4. Quartz Sand, 60s Grinding

Audience Poll

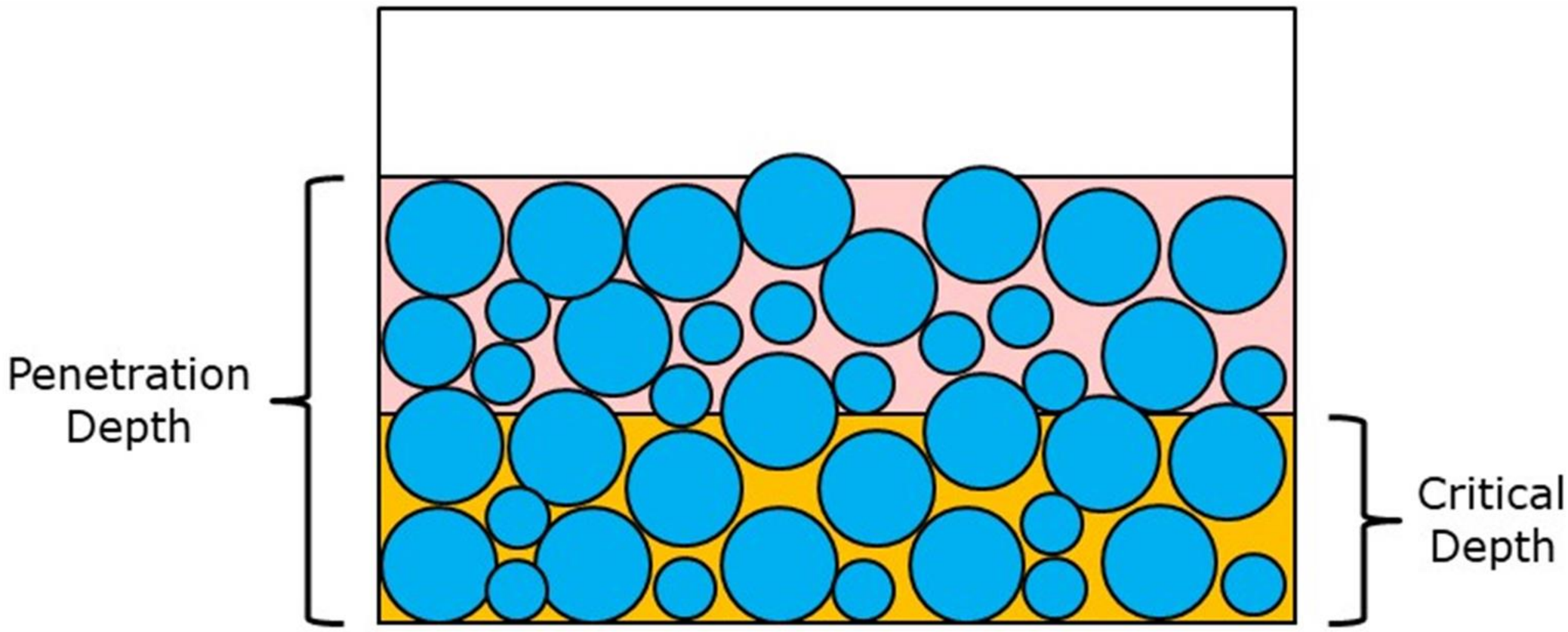
What difference do you think 60 seconds of grinding will make to the signal intensity?

a) -20%
b) No change
c) +20%
d) +50%
e) +70%

Sample Preparation Effects



Sample Preparation Effects



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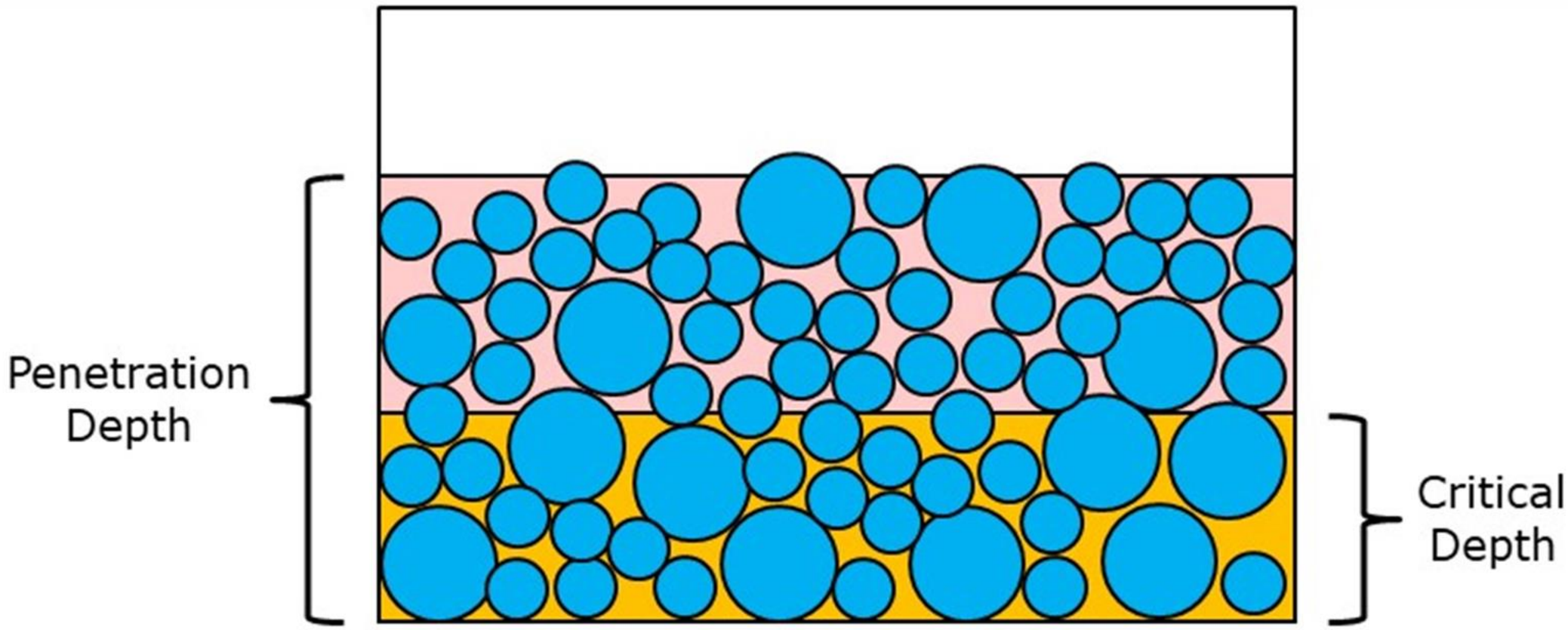
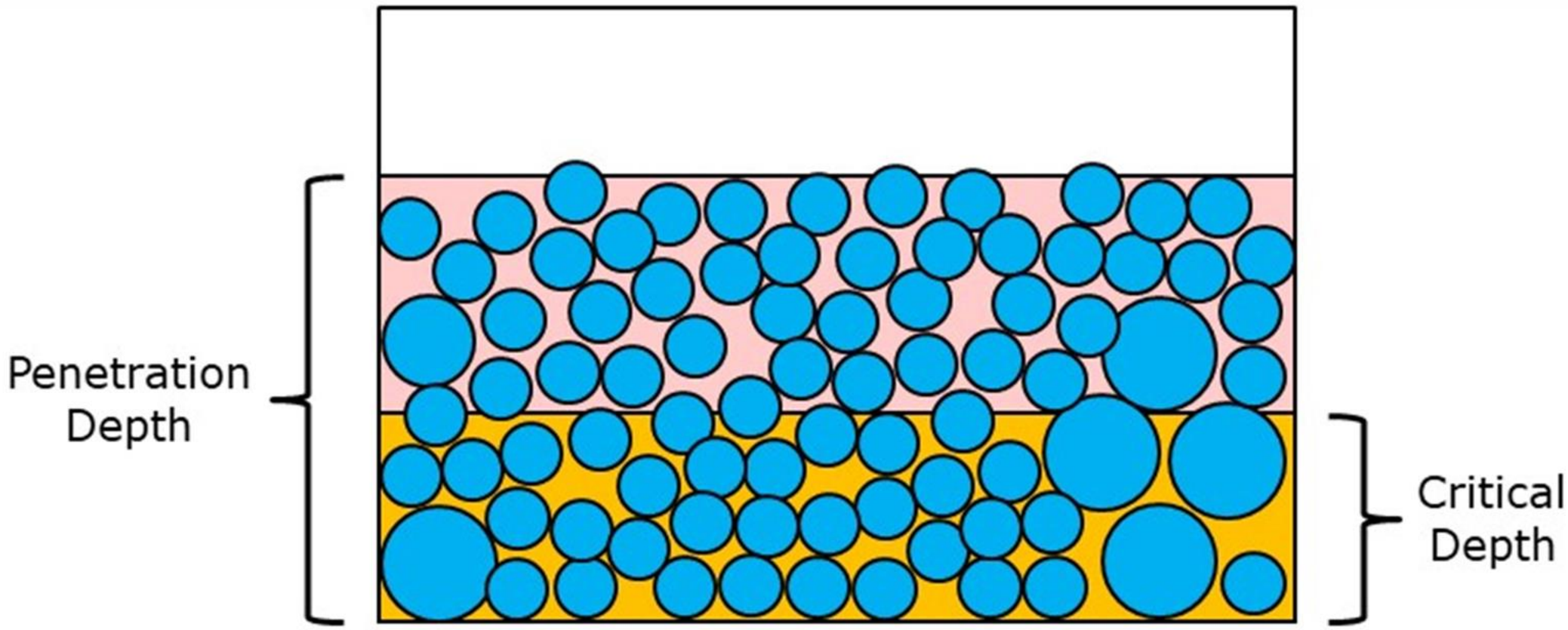
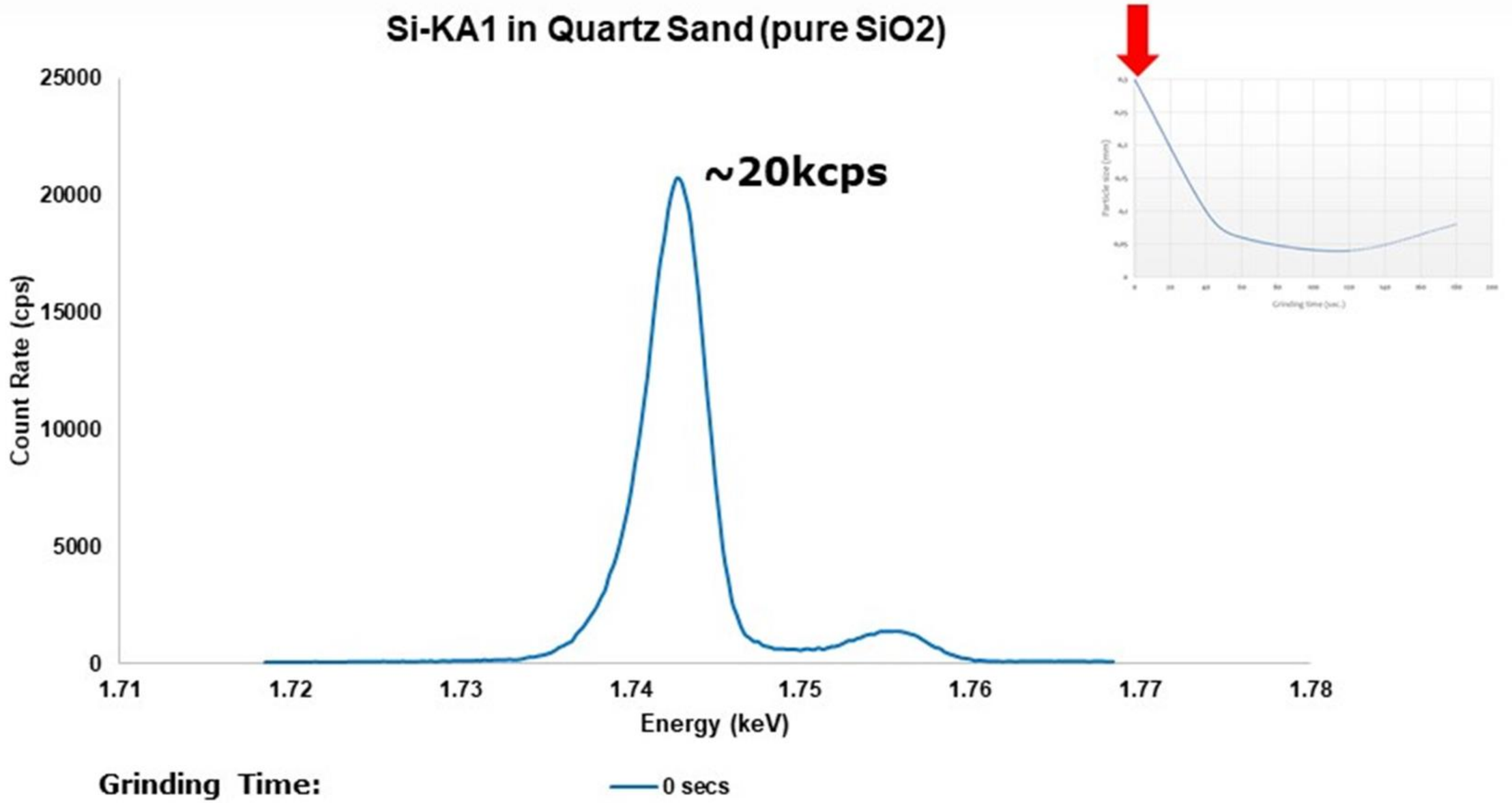


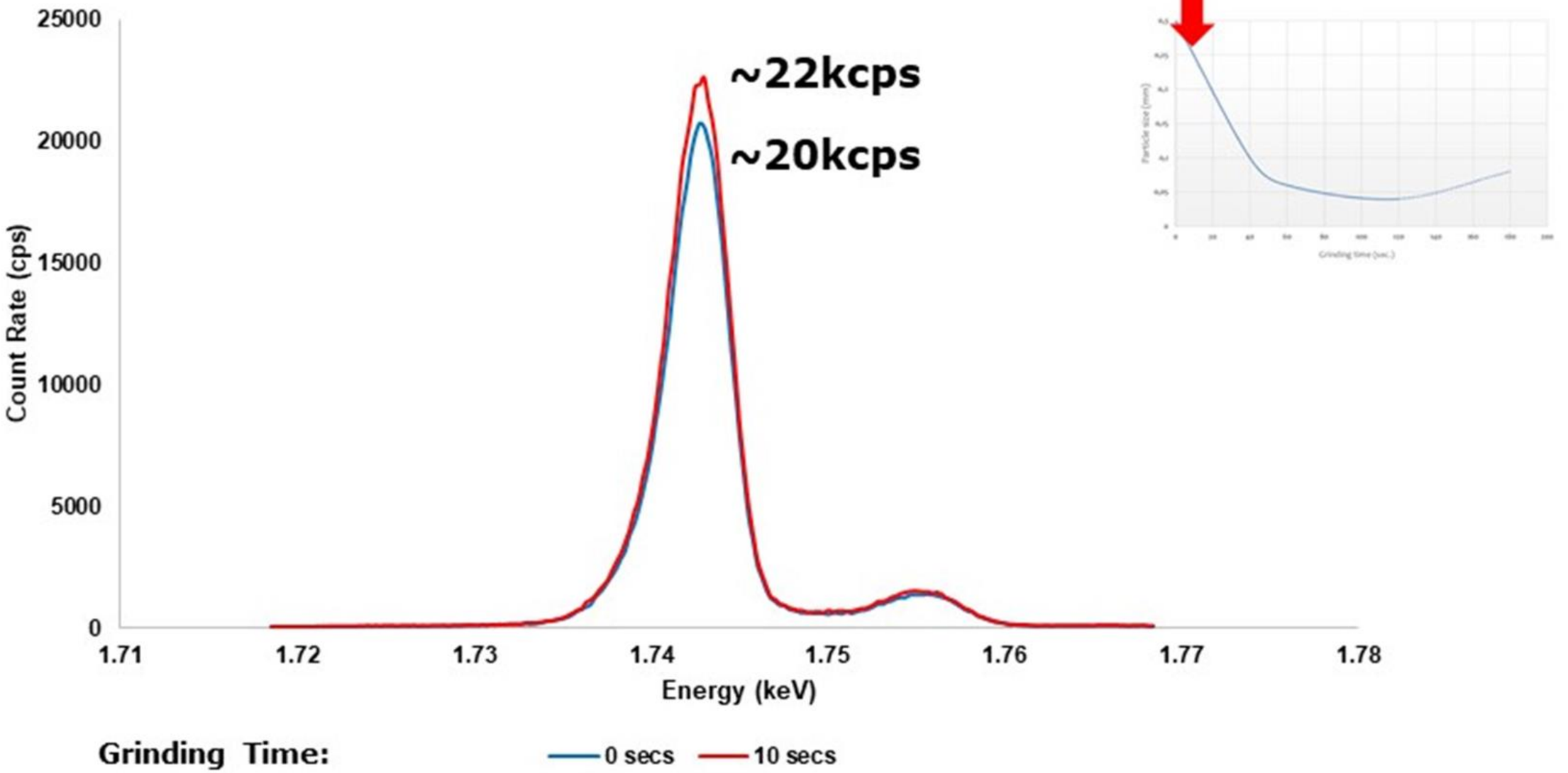
Sample Preparation Effects



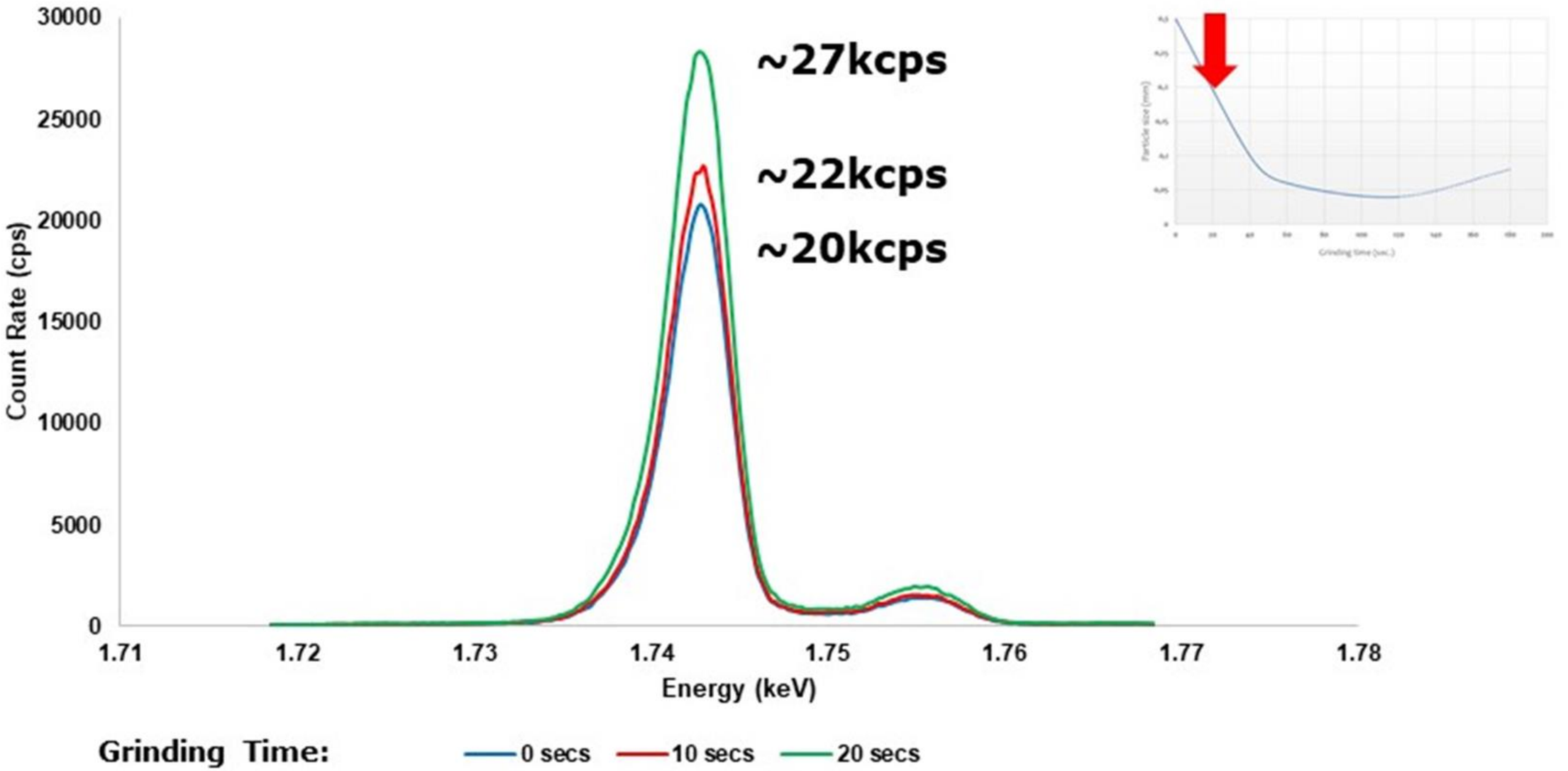
Sample Preparation Effects



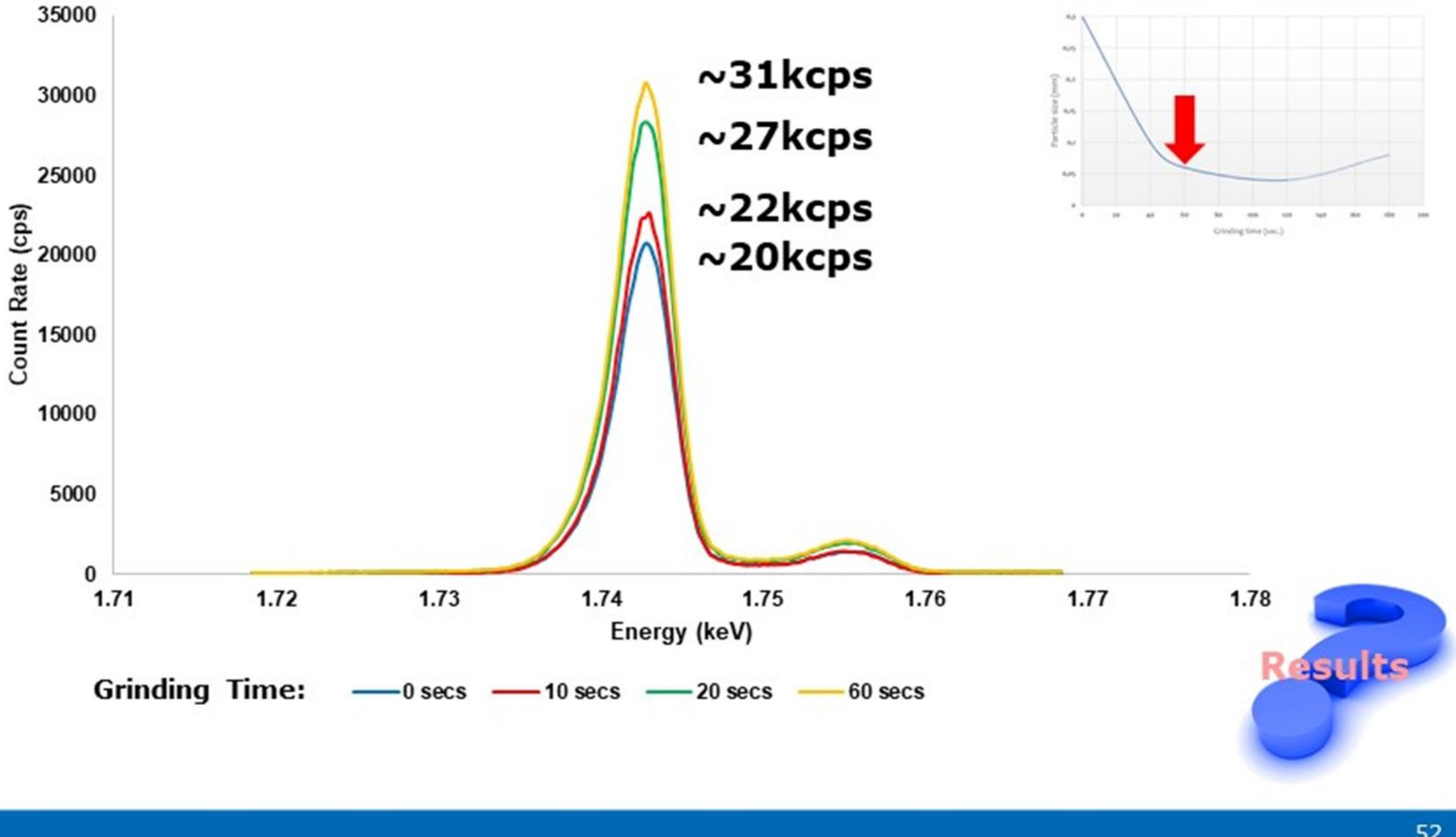
Sample Preparation Effects



Sample Preparation Effects



Sample Preparation Effects



Sample Preparation Effects

> “… so for how long should I grind my sample and how small do I need to make the particles to get the best result?

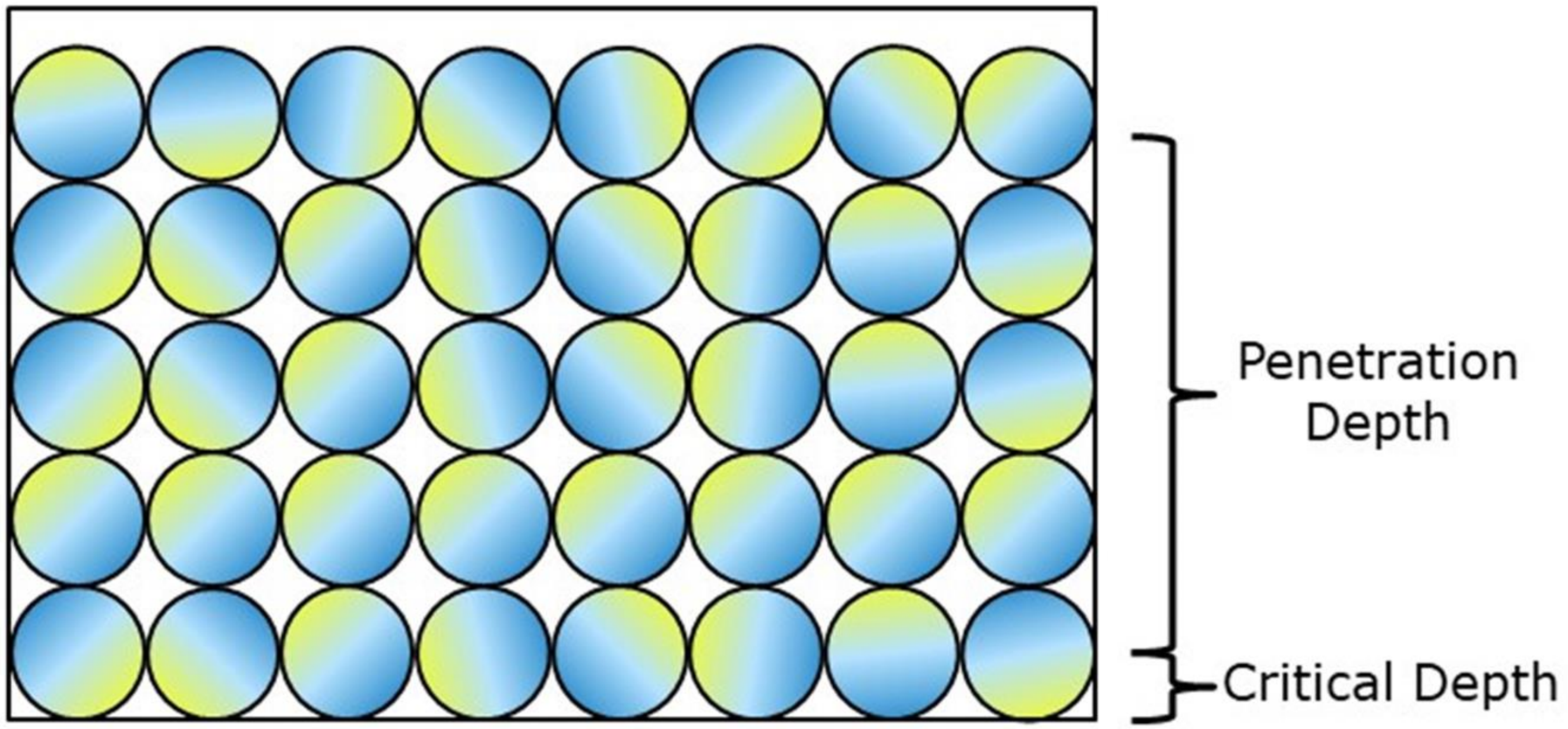
Sample Preparation Effects

> “ … so for how long should I grind my sample and how small do I need to make the particles to get the best result?

> “ **… it depends!**

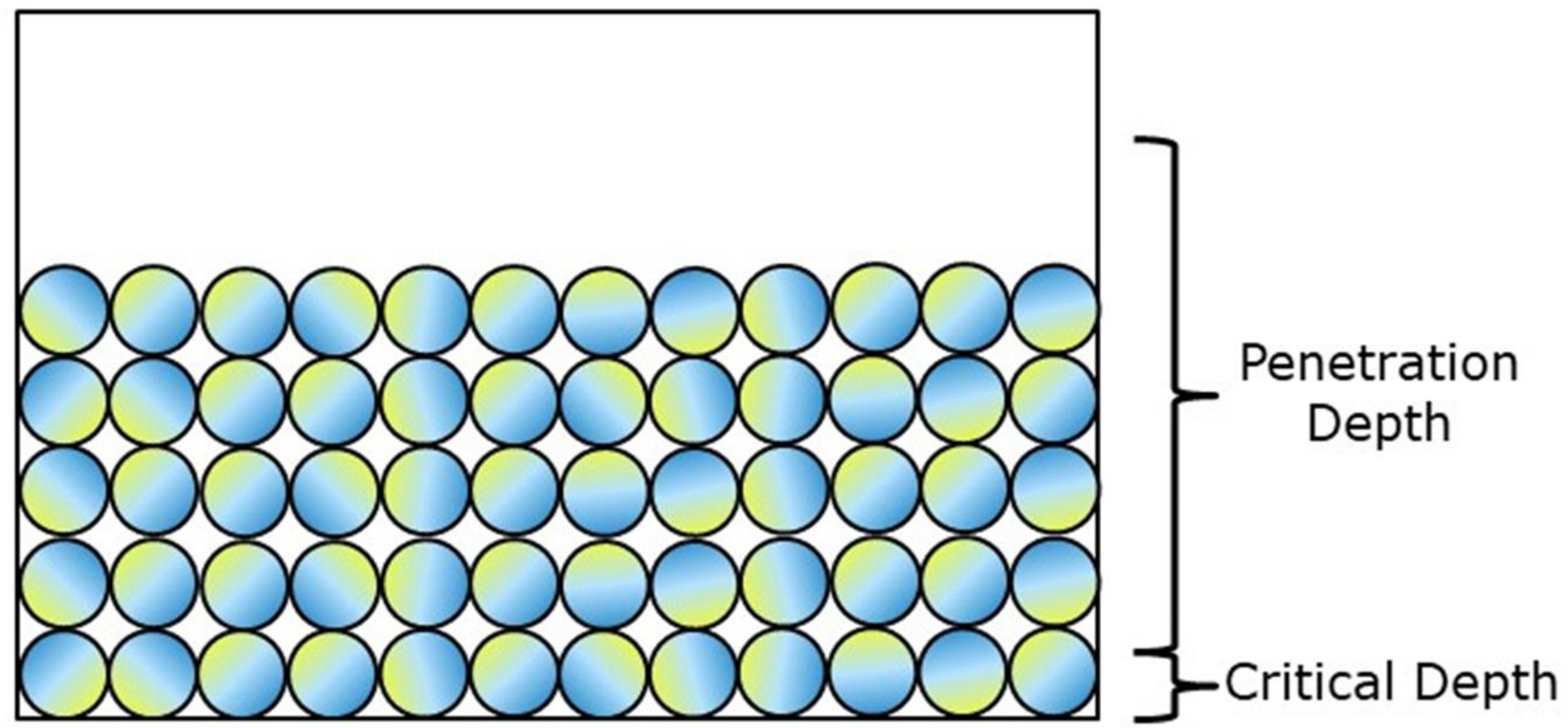
Sample Preparation Effects

Light elements are affected more than heavier elements by particle size variation



Sample Preparation Effects

Light elements are affected more than heavier elements by particle size variation



Sample Preparation Effects

> “ … so for how long should I grind my sample and how small do I need to make the particles to get the best result?

> “ **… it depends!**

Sample Preparation Effects

> … so for how long should I grind my sample and how small do I need to make the particles to get the best result?

> **… it depends which elements you are interested in measuring and what the rest of the sample is made up of**

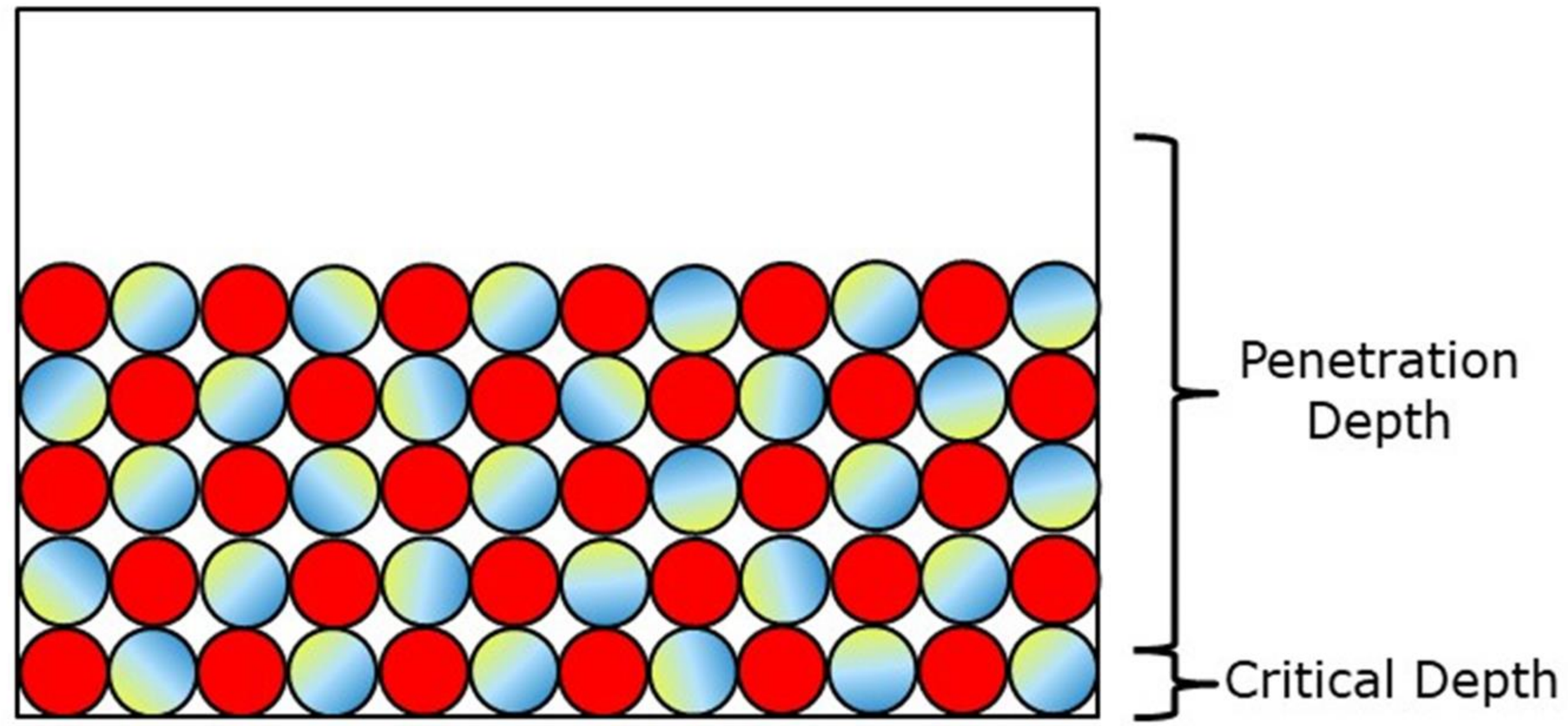
Sample Preparation Effects

Z	Element	Line	Energy (keV)	Graphite	SiO2	Fe	Pb
5	B	KA1	0.1833	5.0	1.0	0.3	0.1
9	F	KA1	0.6768	5.0	3.0	0.4	0.3
11	Na	KA1	1.0419	16.0	10.0	0.9	0.9
13	Al	KA1	1.4875	45.0	26.0	2.0	2.0
14	Si	KA1	1.7412	72.0	40.0	4.0	3.0
20	Ca	KA1	3.6910	684.0	88.0	28.0	4.0
56	Ba	LA1	4.4640	989.0	98.0	43.0	5.0
22	Ti	KA1	4.5104	1.3	156.0	47.0	6.0
25	Mn	KA1	5.8981	2.8	338.0	96.0	12.0
26	Fe	KA1	6.4031	3.6	430.0	119.0	15.0
29	Cu	KA1	8.0481	7.1	838.0	28.0	26.0
74	W	LA1	8.3976	8.0	949.0	31.0	29.0
82	Pb	LA1	10.5512	15.0	1.9	57.0	52.0
40	Zr	KA1	15.7749	4.4	6.0	176.0	43.0
42	Mo	KA1	17.4791	5.5	8.0	234.0	47.0
45	Rh	KA1	20.2158	7.3	12.0	355.0	70.0
47	Ag	KA1	22.1630	8.5	16.0	460.0	89.0
56	Ba	KA1	32.1929	13.1	4.0	1.3	242.0

= cm = mm = µm

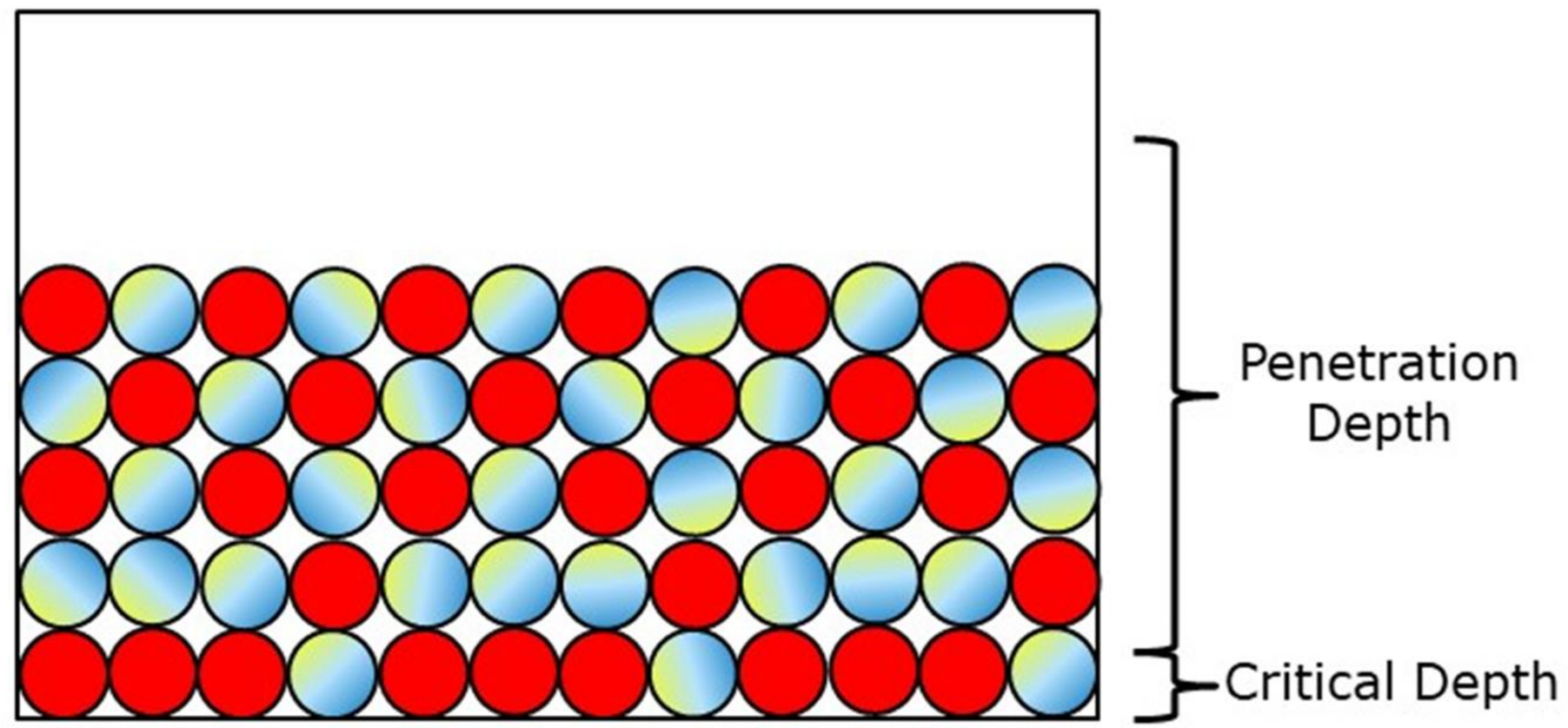
Sample Preparation Effects

Pressing effects, e.g. segregation, are also critical



Sample Preparation Effects

Pressing effects, e.g. segregation, are also critical



Sample Preparation Effects

Pressing effects, e.g. segregation, are also critical

When the sample prep is done correctly…

10 individually prepared fusion beads of a NIST Cement CRM, run against a calibration constructed with NIST CRMs

	CaO (%)	SiO2 (%)	Al2O3 (%)	Fe2O3 (%)	MgO (%)	K2O (%)	Na2O (%)	SO3 (%)
Fused Pead 01	65.950	20.560	5.087	1.977	0.952	0.760	0.159	3.299
Fused Pead 02	65.970	20.530	5.099	1.978	0.946	0.760	0.163	3.334
Fused Pead 03	65.930	20.560	5.107	1.972	0.946	0.760	0.156	3.362
Fused Pead 04	65.900	20.540	5.088	1.974	0.943	0.770	0.158	3.363
Fused Pead 05	65.960	20.530	5.098	1.975	0.949	0.760	0.152	3.313
Fused Pead 06	65.940	20.570	5.092	1.979	0.945	0.760	0.164	3.301
Fused Pead 07	65.890	20.620	5.100	1.981	0.941	0.770	0.161	3.373
Fused Pead 08	65.910	20.520	5.105	1.974	0.952	0.760	0.155	3.356
Fused Pead 09	65.930	20.580	5.101	1.979	0.945	0.770	0.152	3.353
Fused Pead 10	65.960	20.550	5.084	1.974	0.951	0.760	0.156	3.308
MIN	65.890	20.520	5.084	1.972	0.941	0.760	0.152	3.299
MAX	65.970	20.620	5.107	1.981	0.952	0.770	0.164	3.373
MEAN	65.934	20.556	5.096	1.976	0.947	0.763	0.158	3.336
ST.DEV	0.027	0.030	0.008	0.003	0.004	0.005	0.004	0.029

Fusion data presented at Denver X-Ray Conference 2020 by A Mehling, M. Lischka and S. Durali-Müller "High-precision borate fusion using induction heating furnaces with calibrated platinum-gold crucibles"

When the sample prep is done correctly…

10 individually prepared raw meal specimens, run against a calibration constructed using secondary standards also of raw meal.

	CaO (%)	SiO2 (%)	Al2O3 (%)	Fe2O3 (%)	MgO (%)	K2O (%)	Na2O (%)	Cl (%)	SO3 (%)
Pressed Pellet 01	42.820	14.630	2.860	0.950	0.426	0.480	0.051	0.020	0.210
Pressed Pellet 02	42.820	14.620	2.870	0.950	0.427	0.480	0.048	0.020	0.209
Pressed Pellet 03	42.840	14.620	2.880	0.950	0.424	0.480	0.031	0.020	0.208
Pressed Pellet 04	42.860	14.610	2.850	0.940	0.425	0.480	0.039	0.020	0.208
Pressed Pellet 05	42.860	14.620	2.860	0.950	0.430	0.480	0.035	0.020	0.207
Pressed Pellet 06	42.830	14.610	2.860	0.950	0.423	0.480	0.018	0.020	0.205
Pressed Pellet 07	42.840	14.650	2.880	0.950	0.428	0.480	0.034	0.020	0.209
Pressed Pellet 08	42.800	14.600	2.850	0.940	0.424	0.480	0.038	0.020	0.209
Pressed Pellet 09	42.850	14.580	2.850	0.950	0.421	0.480	0.028	0.020	0.209
Pressed Pellet 10	42.800	14.610	2.850	0.950	0.427	0.480	0.033	0.020	0.210
MIN	42.800	14.580	2.850	0.940	0.421	0.480	0.018	0.020	0.205
MAX	42.860	14.650	2.880	0.950	0.430	0.480	0.051	0.020	0.210
MEAN	42.832	14.615	2.861	0.948	0.426	0.480	0.036	0.020	0.208
ST.DEV	0.022	0.018	0.012	0.004	0.003	0.000	0.009	0.000	0.002

Questions and Answers

Any questions?

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

Thank you!

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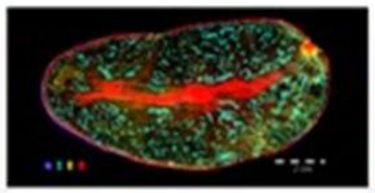
July 9, 2020 UPCOMING

Elemental Analysis for the Iron, Steel and Metal Industry by OES, XRF and Combustion/Fusion

During this webinar, specialists from Bruker will introduce three major techniques for elemental analysis: OES, CS/ONH, XRF.

Session 1 › Session 2 ›



July 8, 2020 UPCOMING

Seeing the world through other eyes

Scanning micro-XRF has developed into a highly informative analytical tool for the study of complex samples across multiple disciplines.

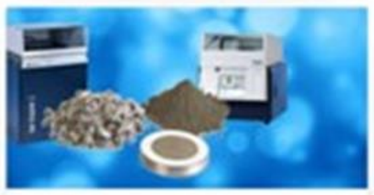
Session 1 › Session 2 ›

June 24, 2020 UPCOMING

TRACER: The benchmark in handheld-XRF for Cultural Heritage (NEW-Third session)

The Bruker TRACER handheld-XRF is the go-to portable analytical tool for elemental analysis in cultural heritage studies.

Register ›



June 24, 2020 UPCOMING

The Importance of Good Sample Preparation

An educational webinar hosted jointly by Bruker UK and Herzog, in partnership with Datech Scientific

June 17, 2020 ON-DEMAND

TRACER: The benchmark in handheld-XRF for Cultural Heritage

The Bruker TRACER handheld-XRF is the go-to portable analytical tool for elemental analysis in cultural heritage studies.

June 10, 2020 ON-DEMAND

Quickly analyze nutrients, additives, and contaminants in food, feed, supplements, and beverages with pXRF and TXRF

This webinar will demonstrate the powerful combination of portable and benchtop XRF spectrometers and its application to food samples and beverages.

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