“AMAP P4 Melt Cleanliness” in a Nutshell

J. Morscheiser

24.05.2018

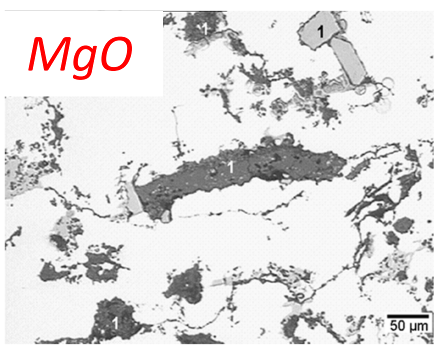
Content

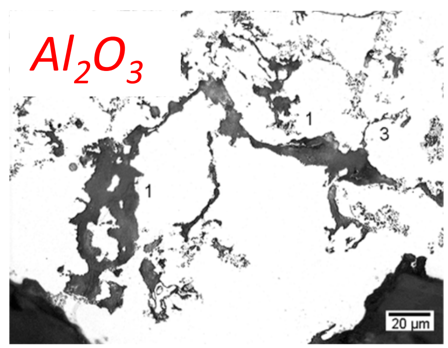
- Motivation and introduction incl. state of the art
- Project organization
- Reference setup
- Fundamental understanding of settling and agglomeration
- Results regarding detector development and testing
  - Ultrasound
  - LIBS
- Summary
- Publications within AMAP P4

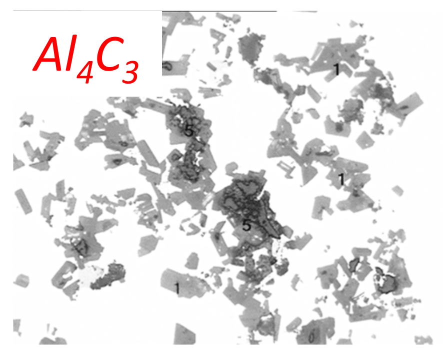
MOST WANTED

- Particles in aluminium
  - Non-metallic, undissolved impurities in liquid or solid aluminium
- Typical sources
  - Metal sources: Alloying material, potroom metal, scrap, …
  - Melt oxidation
  - Particle reaction
  - Linings, tools, …
- Particle characteristics
  - Various shapes: Spherical, flakes, films, clusters
  - Sizes from 1-1000µm
  - Density often close to Al

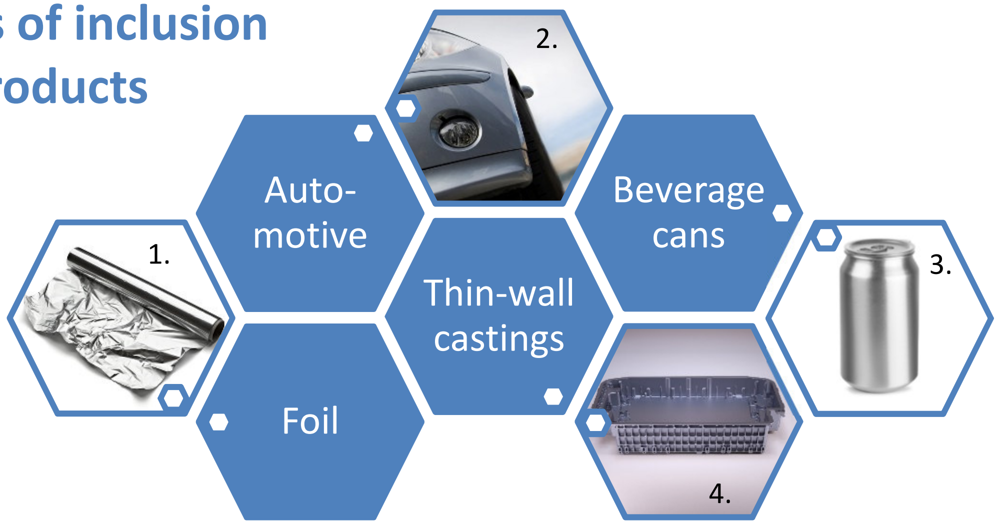
Particle Type	Chemistry
Magnesium Oxide	MgO
Spinel	$MgAl_2O_4$
Refractory Inclusions	Oxides and Silicates
Spinel like	
Thick Oxide Films	a-Al_2O_3 ,MgO, $MgAl_2O_4$
Cuboid	$MgAl_2O_4$
Alpha Alumina	a-Al_2O_3
Thin Oxide Films	a-Al_2O_3 ,MgO, $MgAl_2O_4$
Aluminium carbide	Al_4C_3
Grain refiner	TiB_2, TiC
Spinel Crystal	MgO, $MgAl_2O_4$





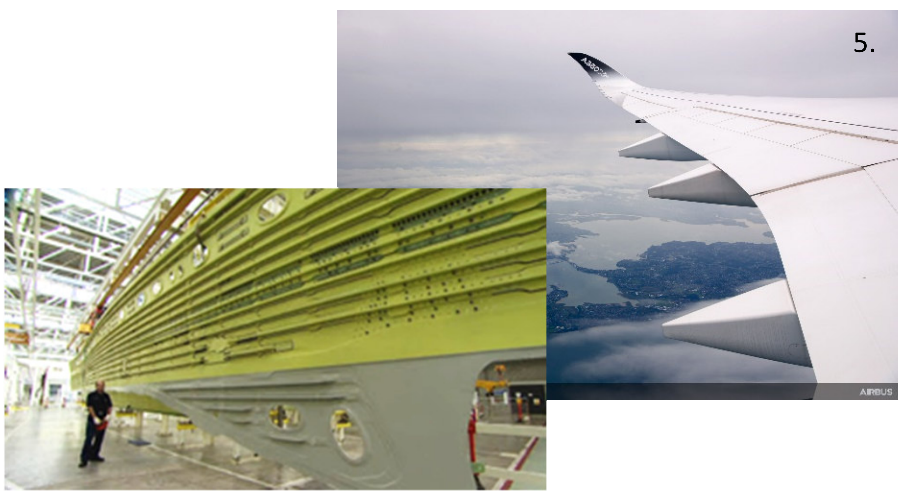


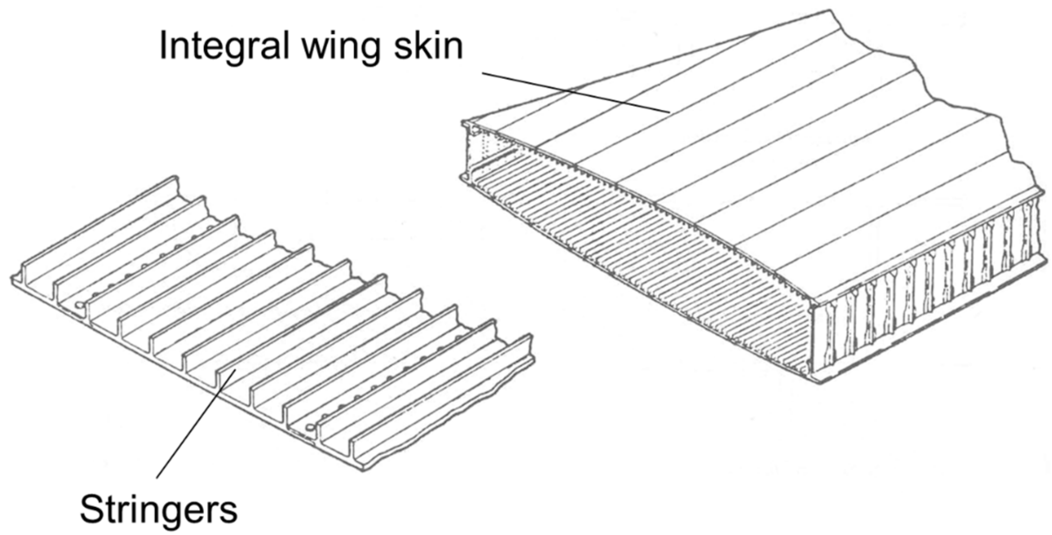
Examples of inclusion critical products



1. https://www.boldsky.com/img/2014/11/x24-foil1.jpg.pagespeed.ic.hcf-8gx1ZR.jpg
2. www.Aleris.com
3. www.homecanning.co.uk
4. Nemak / Deutsche ACCUmotive GmbH & Co. KG
5. www.Airbus.com

Short excursion on wing plates

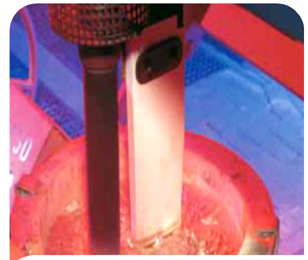




Inclusion Control in Production

Raw Material

Melting



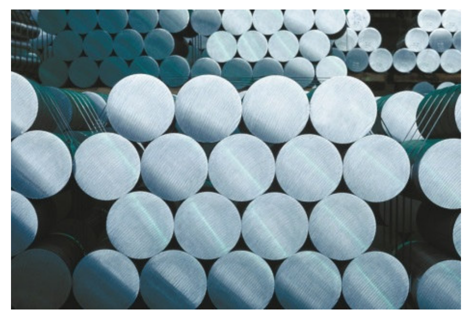
Melt Treatment

Filtration

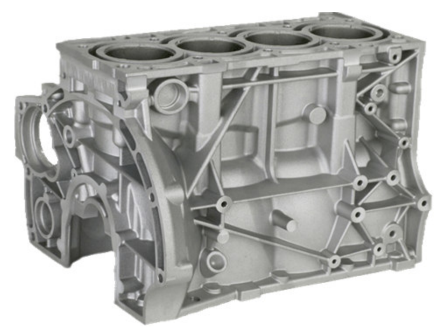
Casting

Rolling Ingots

Extrusion billets

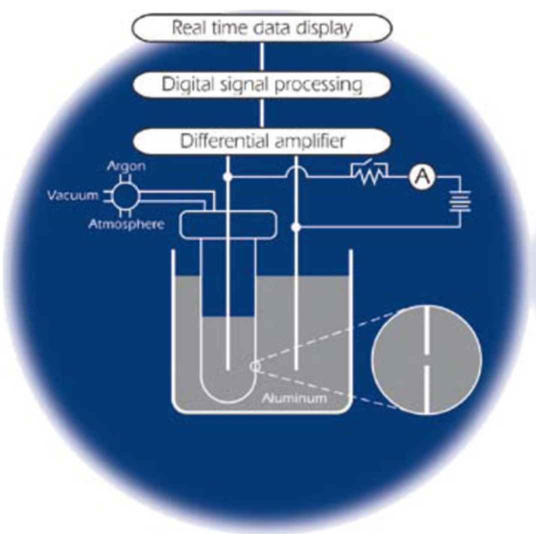


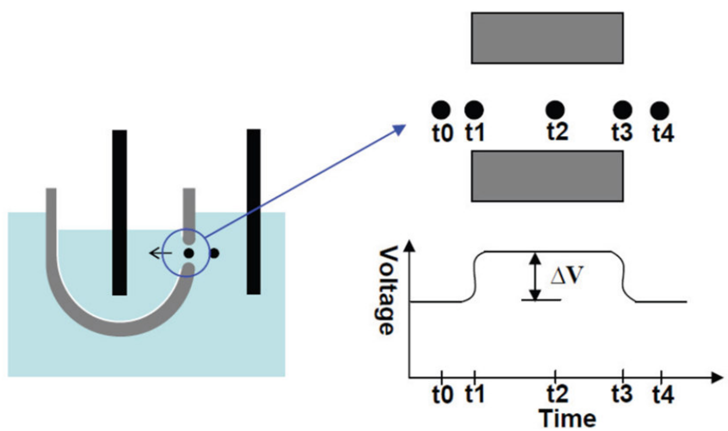
Finished castings

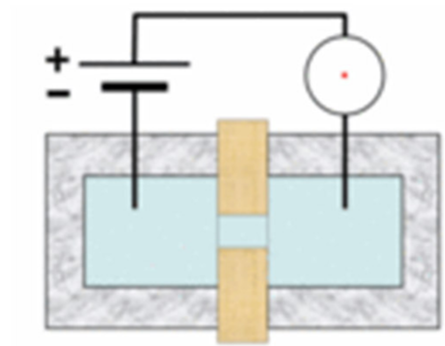


Proper control you want, properly measure you must

- Based on the Coulter Counter Principle (ESZ)
- Measurement range: approx. 20 – 150 µm
- Measurement location: mainly launder
- Semi-continuous measurement: approx. 1 sample per minute
- Volume representativity of the analysis: about 0.01% of the cast volume
- Equipment availability: limited (1 producer)







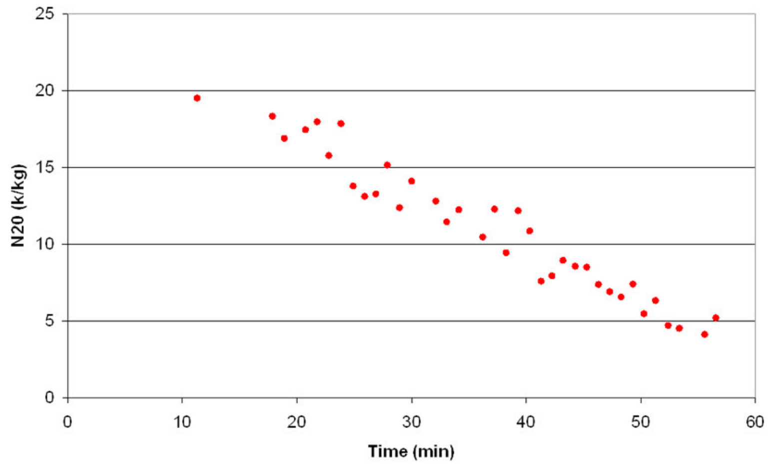
Technologies in use – LiMCA

Mobile unit (LiMCA III)

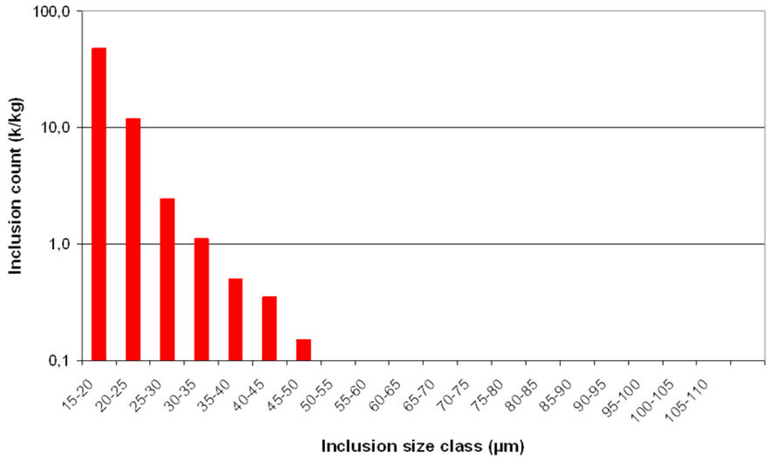
Stationary unit (LiMCA CM)

Close-up of LiMCA head

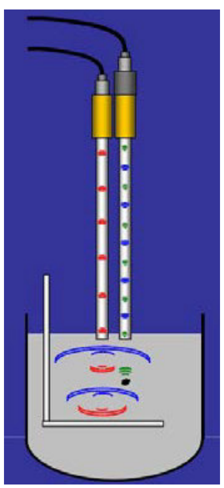
Overall N20 inclusion trend

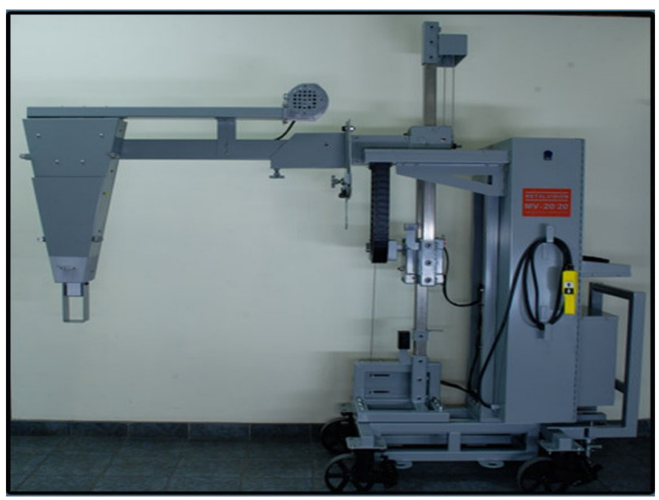


Histogram of single data point

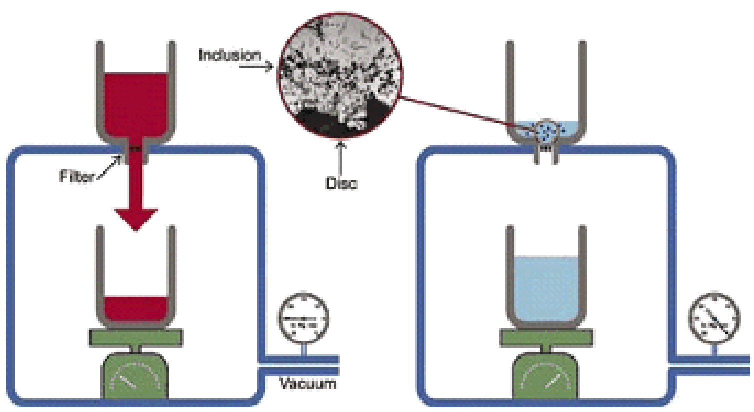


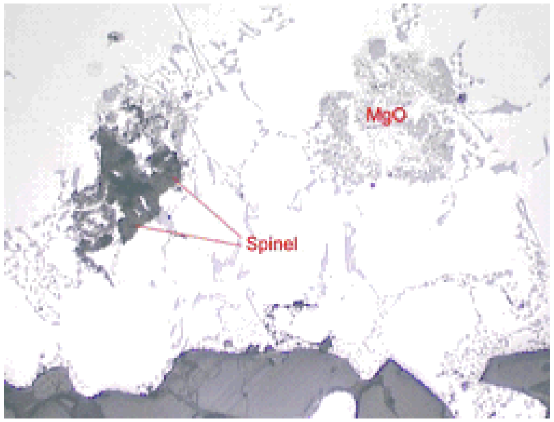
- Based on the attenuation of the signal and on the reflection generated by discontinuities in the liquid phase
- Measurement location: mainly launder
- Measurement range: > 20 (?) µm
- Continuous measurement
- Volume representativity of the analysis: about 0.1-1 % of the cast volume
- Equipment availability: 1 commercially available unit





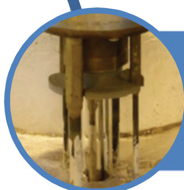
- Inclusion concentration by filtration
- Analysis of filter residue based on visual observation
- Measurement location: any
- Measurement range: > 1 µm
- Discontinuous measurement (often 2 – 3 / drop)
- Volume representativeness of the analysis: about 0.01 % of the cast volume
- Equipment availability: 1 producer





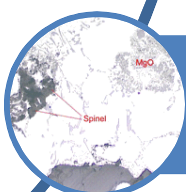
Summary Inclusion Detection

Challenging task due to high temperatures, opacity and reactivity of Aluminium



Few established methods available on the market

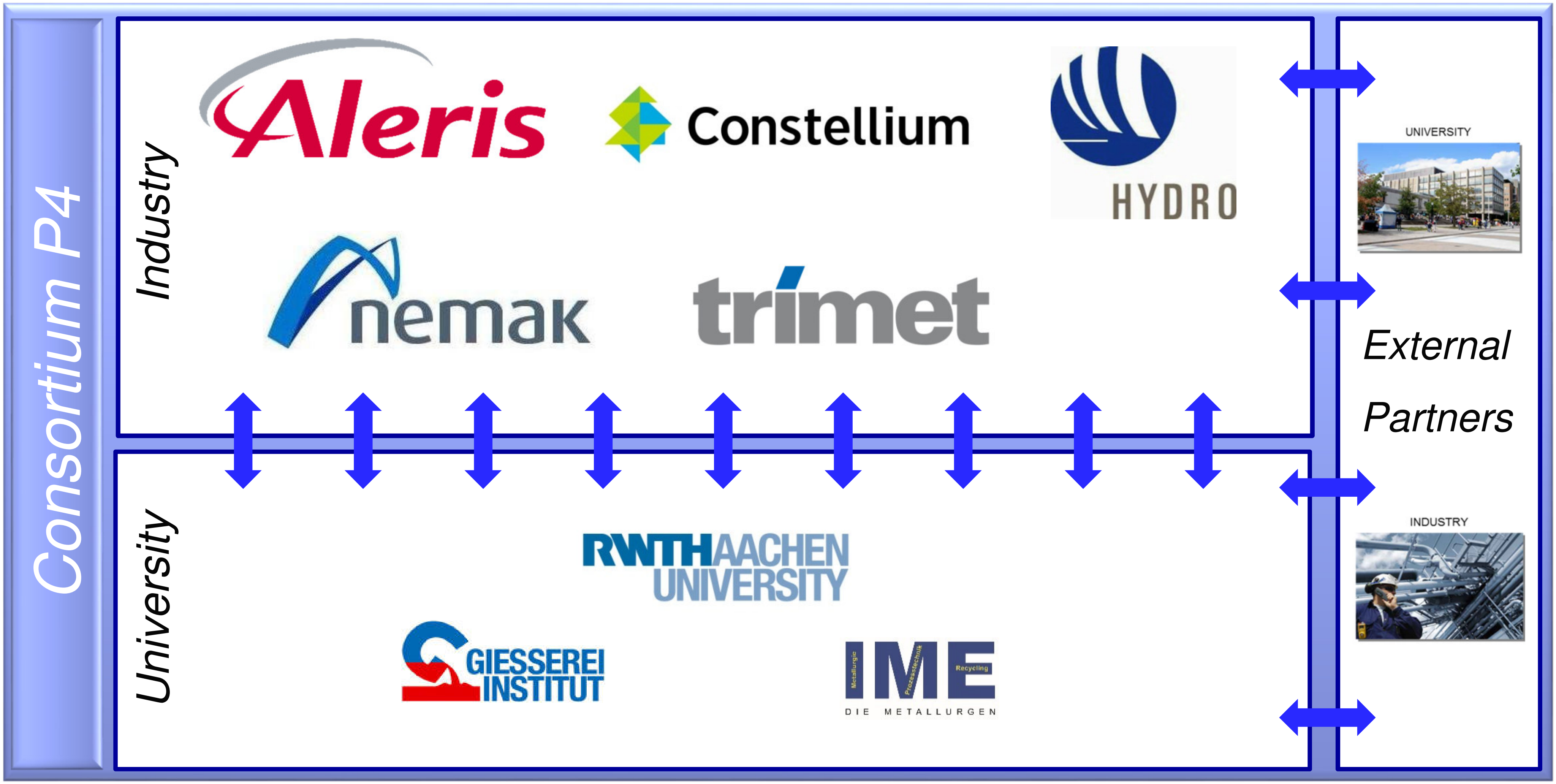
Often high capital cost involved



Different methods have different drawbacks (e.g. sampling volume, time until result is available, analysis of chemical composition, etc.)

→ Need for optimized technology

P4 – International Consortium – Melt Cleanliness



- Title: AMAP P4 Melt Cleanliness
- Duration: 01.07.2013 – 31.12.2016
- Objectives: - Development of a prototype detector for non-metallic inclusions
  - Fundamental understanding of settling & agglomeration of inclusions

2013	2014	2015	2016
Finalization of contract	Development of reference setup	Lab scale settling and benchmark tests	Industrial field test of ultrasound
Expert and technology scouting	Feasibility study of different technologies	Detailed study of LIBS on wrought alloys	Industrial field test of LIBS

PhD Thesis Mertol Gökelma on settling and agglomeration of inclusions

Support of AiF Project on ultrasound detector

Minimum General Requirements

- Unit must be
  - safe to operate
  - temperature-resistant
  - moveable

- able to measure in launder & crucible / ladle (typical melt velocity in launder 10 cm/s)

- Minimum resolution:
  - For small sample volume: 20-50 µm (LiMCA)
  - For large sample volume (near 100 %): >50 µm
- Temperature resistance up to 750°C
- If possible: differentiation between microbubbles and non-metallic inclusions

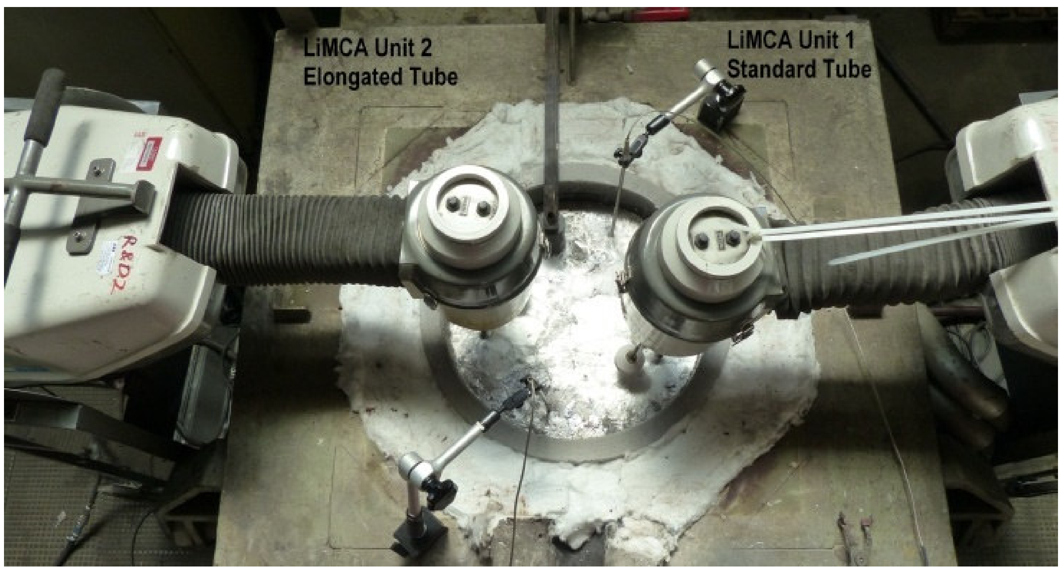
- Definition of basic requirements for detector (e.g. detection limit, max. price, size, weight, etc.)
- Very broad survey on different potential partners and technologies
- Feasibility studies on most promising methods with solid reference samples

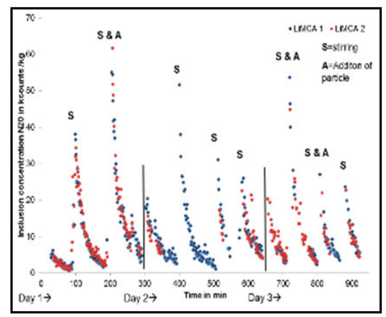
Method	Feasibility
Ultrasound (US)	Already established in MetalVision
Laser induced breakdown spectroscopy (LIBS)	First results from lab and industrial foundry tests promising; gives additional info on composition
X-ray radioscopy	Trials with selected samples at HZDR not successful; insufficient contrast of oxides in Al
Ultrasound Doppler velocimetry	With current technology insufficient sensitivity for small non-metallic inclusions in Al melts
Contactless inductive flow tomography	Insufficient sensitivity
X-ray tomography	Insufficient resolution in moving molten metal
Automated analysis of PoDFA samples	Software not capable of differentiation between various particle types and filter particles

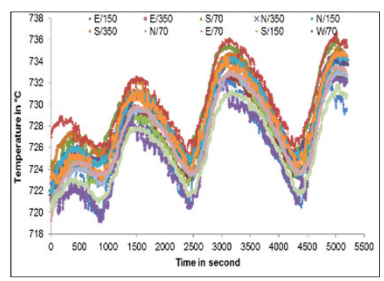
Reference Setup

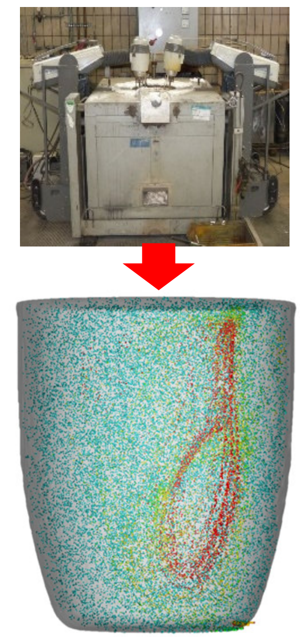
AMAP
Advanced Metals and Processes

- Reference setup for testing of potential detectors parallel to LiMCA (state of the art): Resistance heated crucible furnace at foundry institute (GI)
- Symmetry of measurement positions validated by double LiMCA trials
- Numerical modelling of reference setup for deeper understanding of flow behaviour
- Comparison of model to results from extensive temperature measurements
- Inclusion load adjusted by manual addition of MMC
- Monitoring of settling curves for different inclusion types

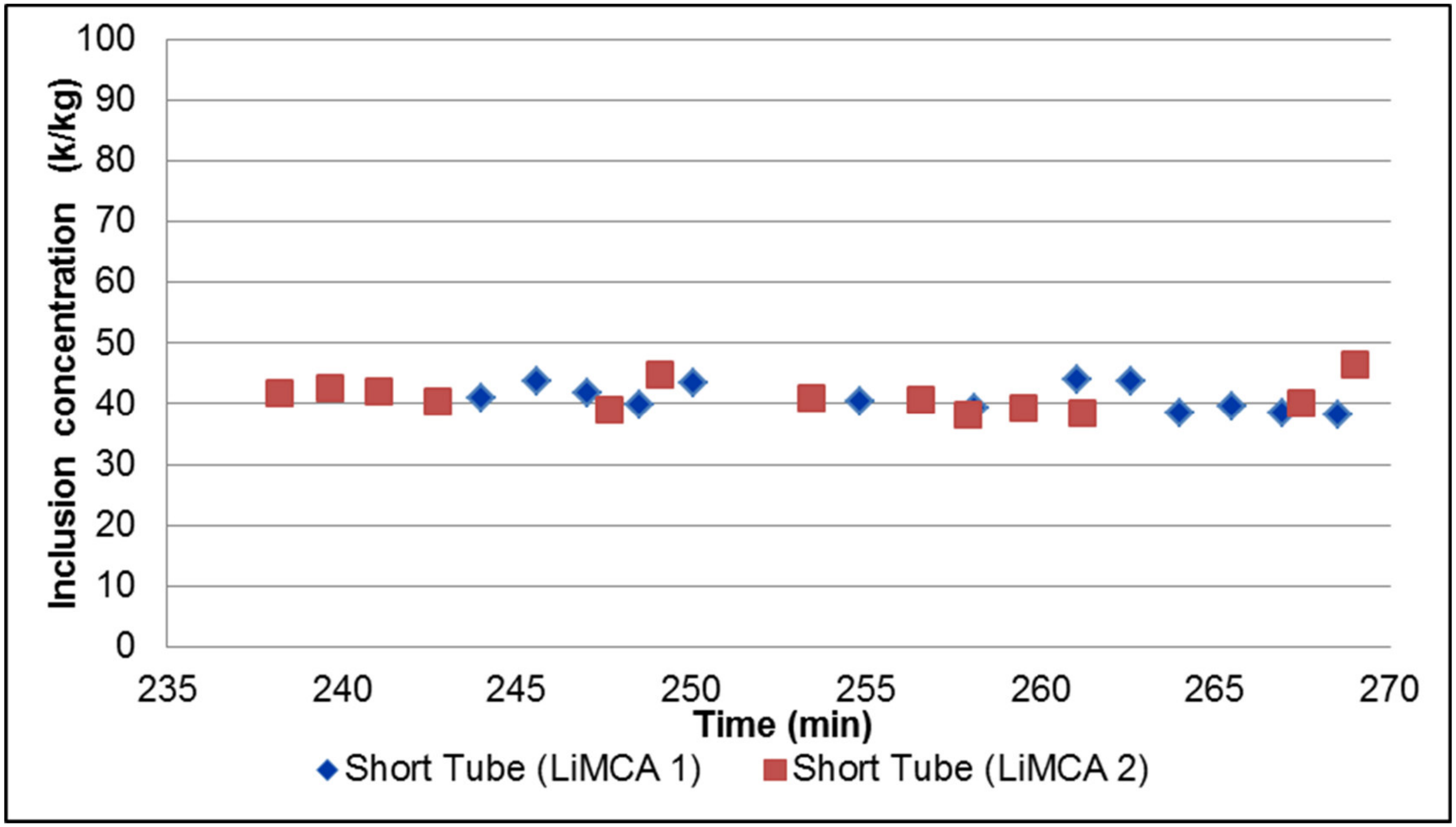


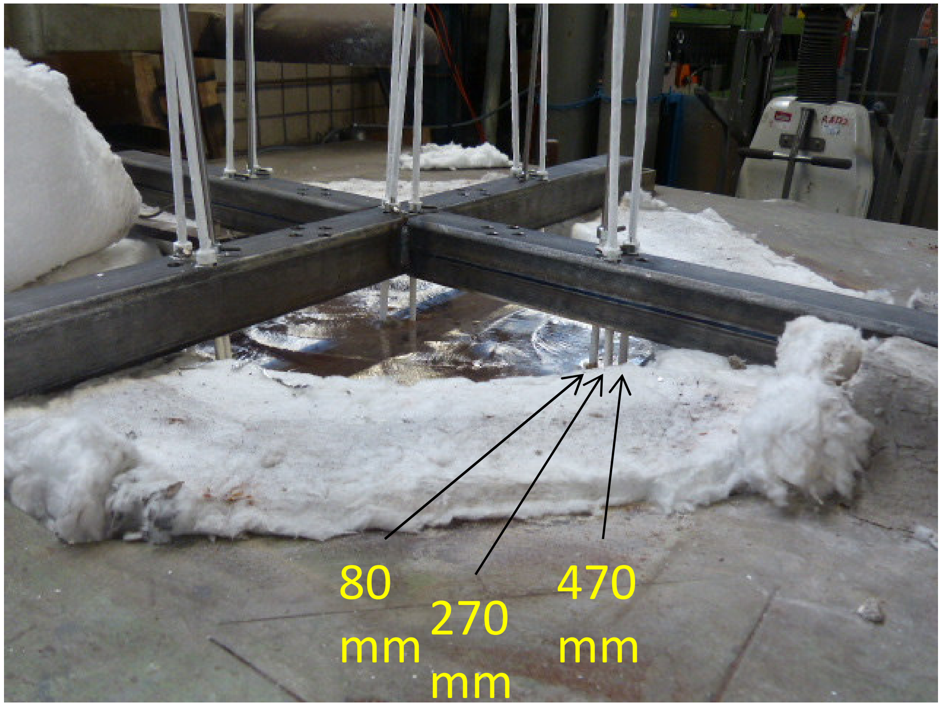


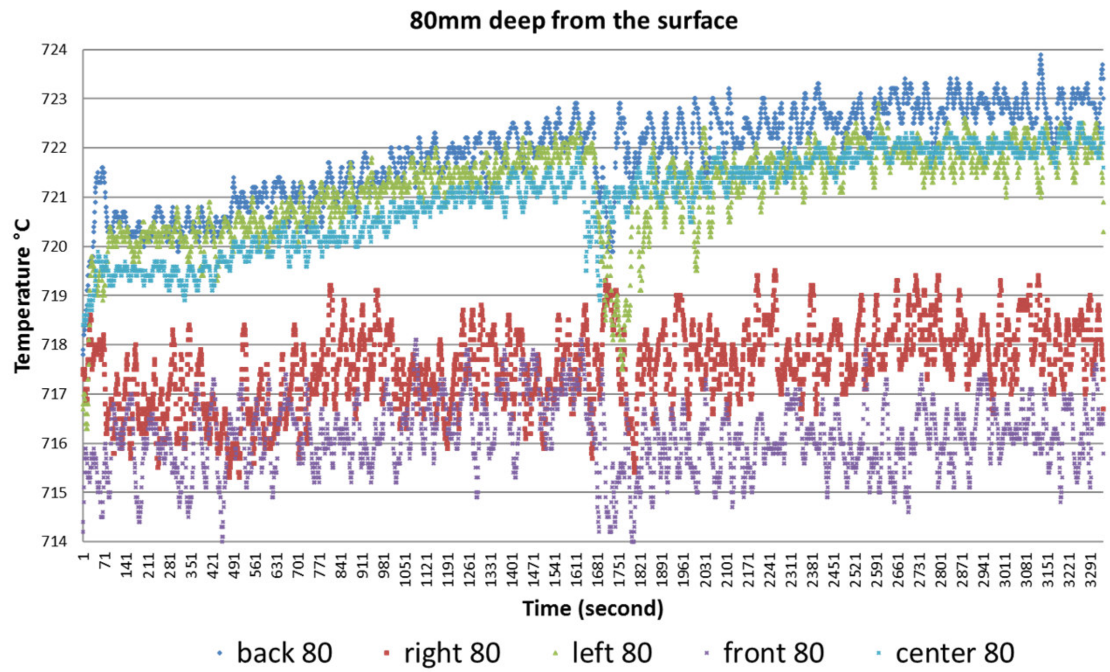




Both LiMCAs measured with standard tubes simultaneously in almost same conditions



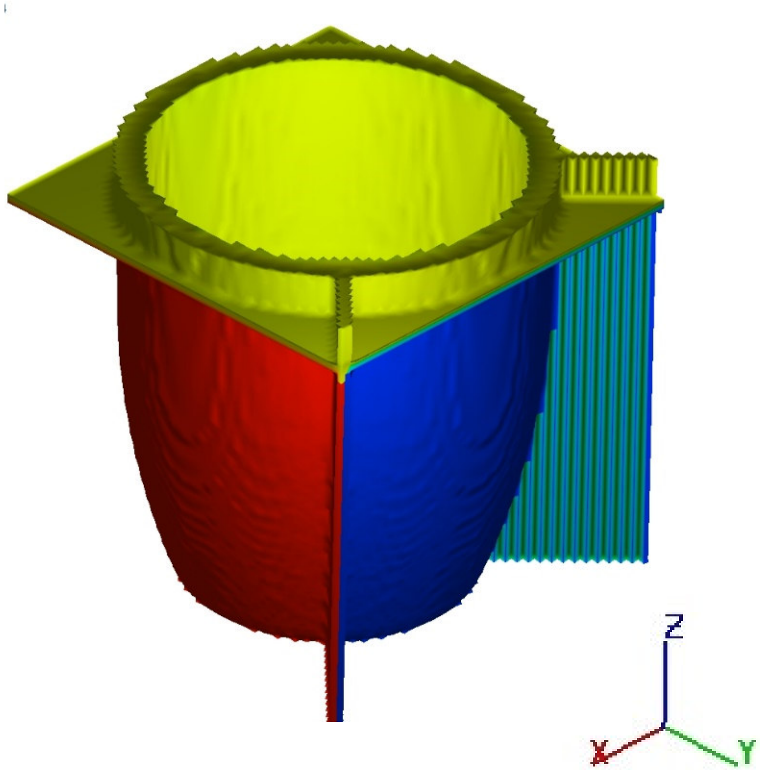




Experimental setup

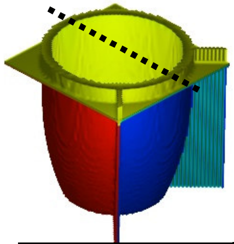
Exemplary Results

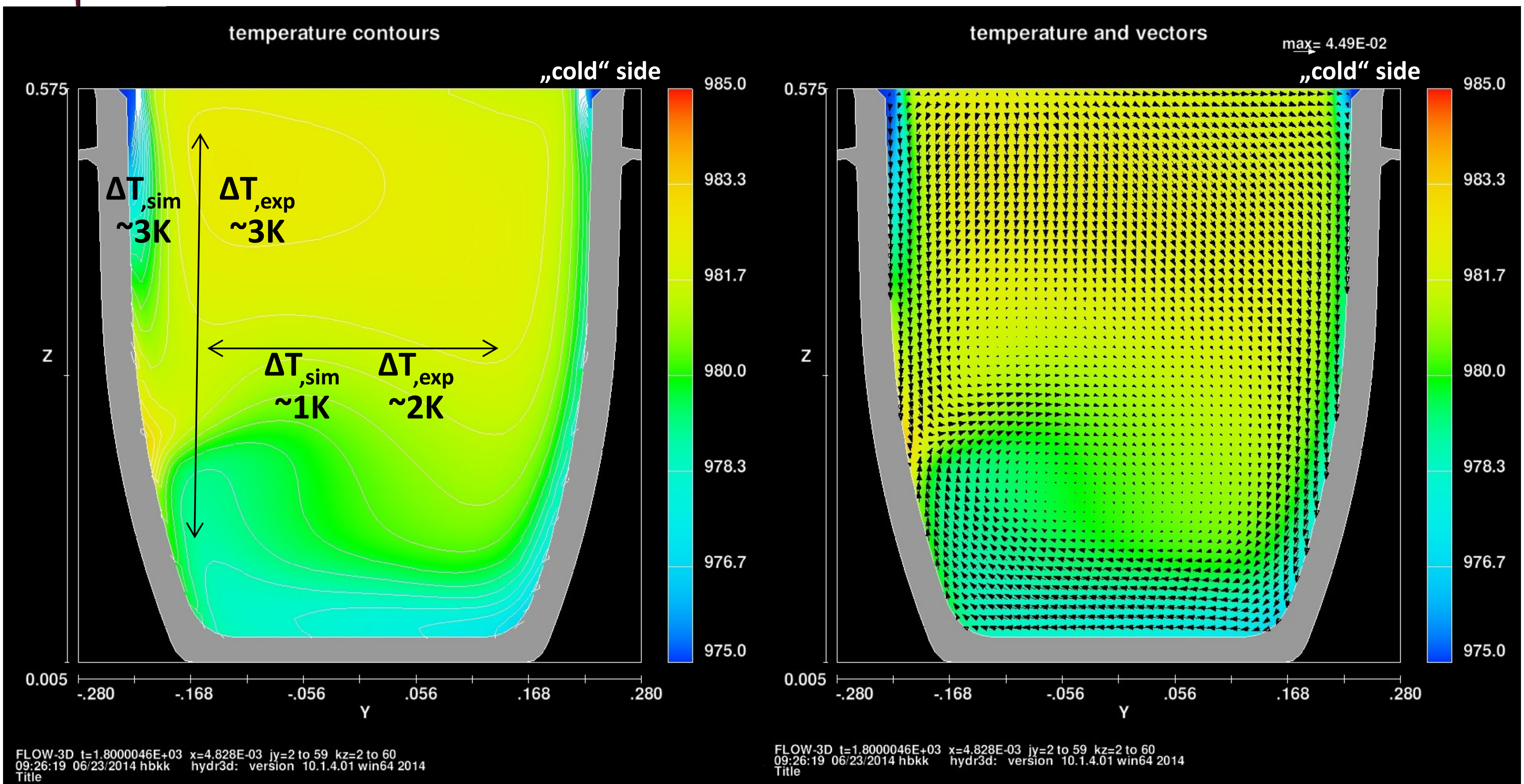
- Furnace is heated on three sides
- Top of the crucible is not heated
- Heat transfer from crucible walls to voids by radiation model
- Heat loss / transfer above melt bath to ambient air: 20 W/m²K



Comparison of Temperature Measurements with Numerical Results and Calculation of Flow Fields

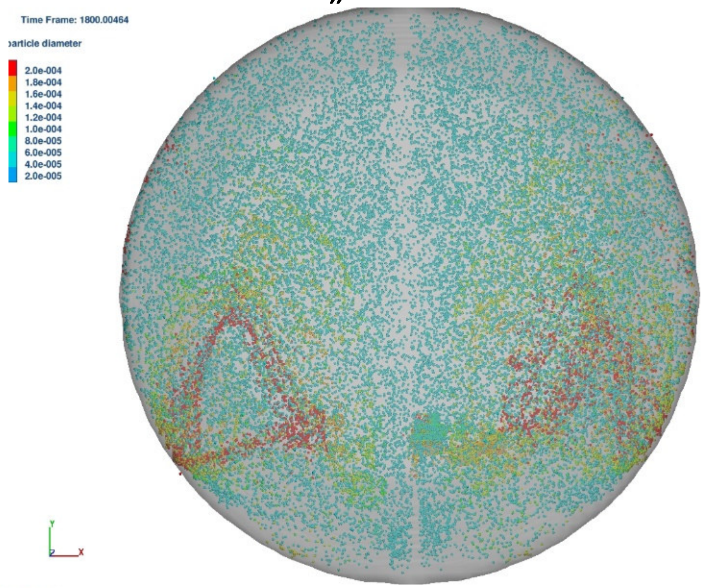
AMAP Advanced Metals and Processes



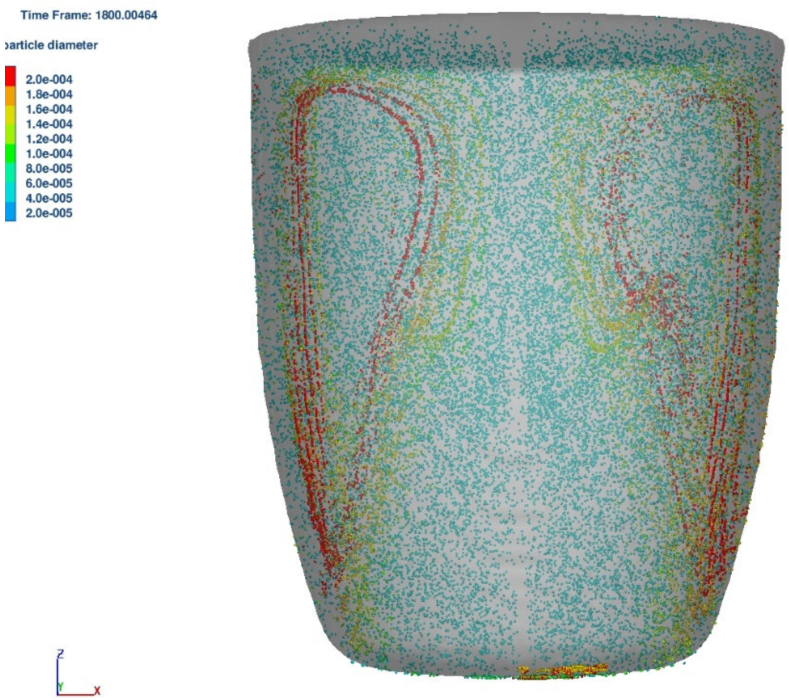


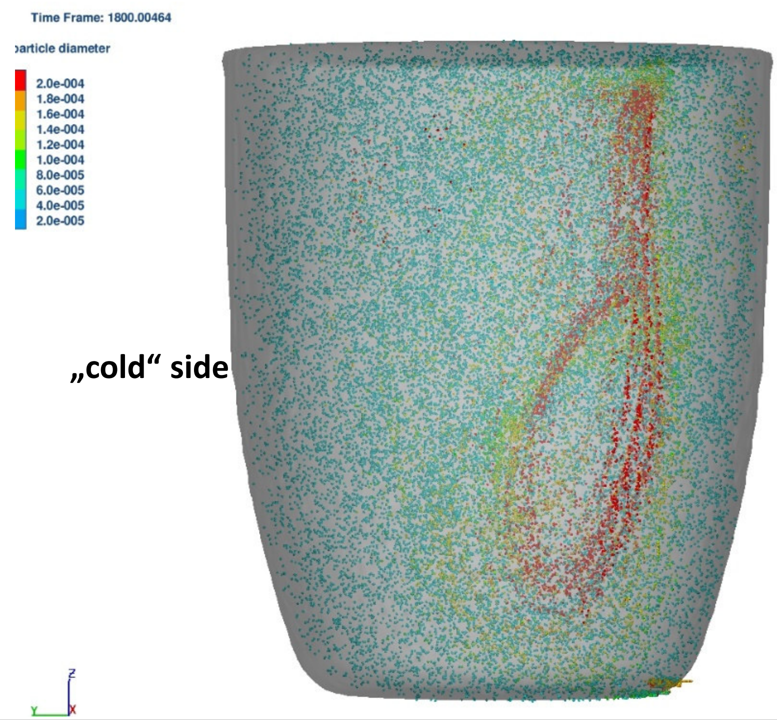
Particle tracking loops

„cold“ side



Density: 3950 kg/m³, simulating Al_2O_3 particles
Size: spherical, diameter from 20µm up to 200µm
Number of particles: 150.000
Initial condition: homogenous/random distributed
Fluid motion is not affected by particles

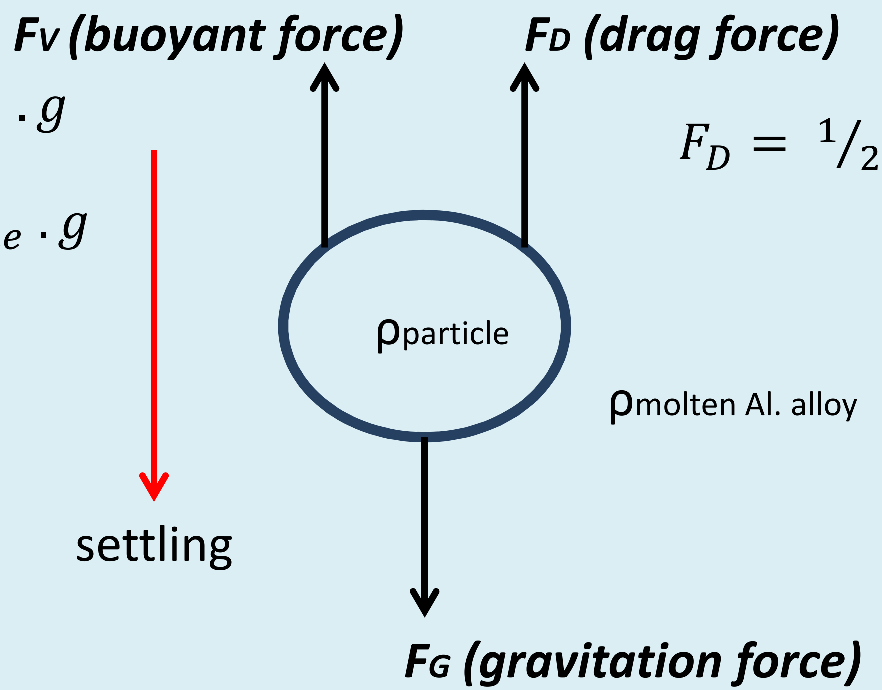




„cold“ side

$$F_V = m_{fluid\ displaced} \cdot g$$

$$= \rho_{fluid} \cdot V_{particle} \cdot g$$

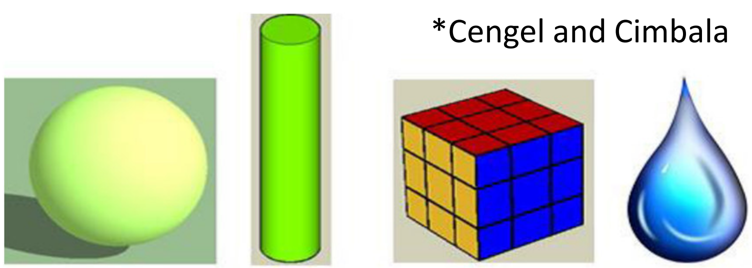


$$F_D = {}^{1}/_{2} \cdot \rho_{fluid} \cdot v^2{}_{\text{particle}} \cdot C_D \cdot A$$

$$F_G = m_{particle} \cdot g$$

$$= \rho_{particle} \cdot V_{particle} \cdot g$$

*Cengel and Cimbala



***C_D:** 0.5 0.8 1.0 0.04

C_D: Drag coefficient

Most studies assume individual spherical particles

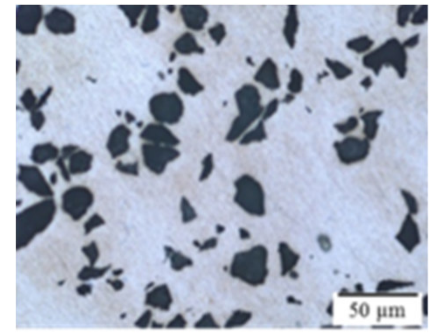
Contribution: analysis of inclusion data vs particle shape

Settling and agglomeration of particles in crucible furnaces

Experimental Setup

Resistance heated crucible furnace, 100kg molten aluminum (99.7%), LiMCA & PoDFA

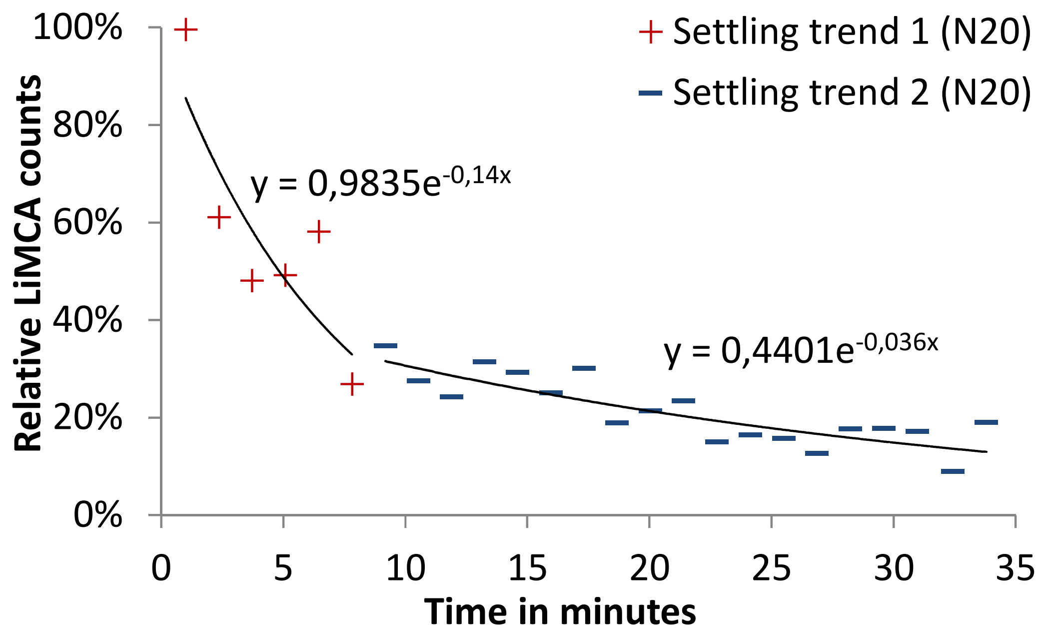
Experimental Procedure



- Controlled addition of MMC material containing Al_2O_3 particles
- Manual stirring of the particles
- Monitoring the settling behavior of the inclusions
- Post-processing of the slopes of settling curves

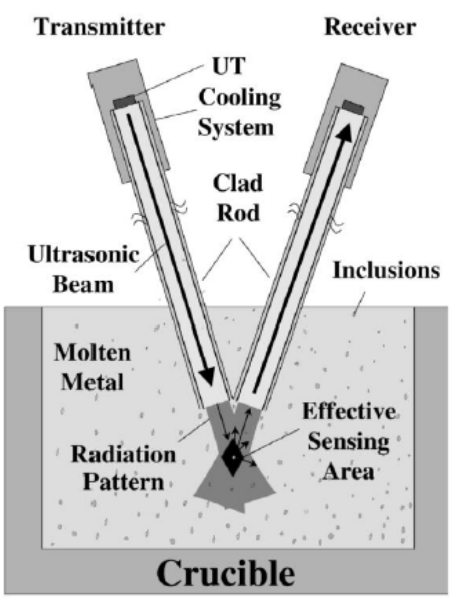
Data analysis

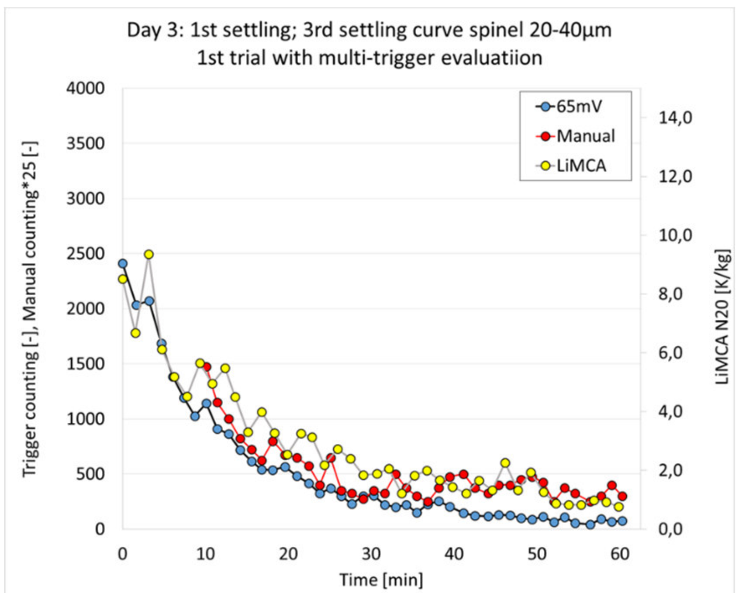
- Exponential regression
- Modeling :
  - Size (F_G)
  - Density (F_G)
  - Shape factor (F_D)



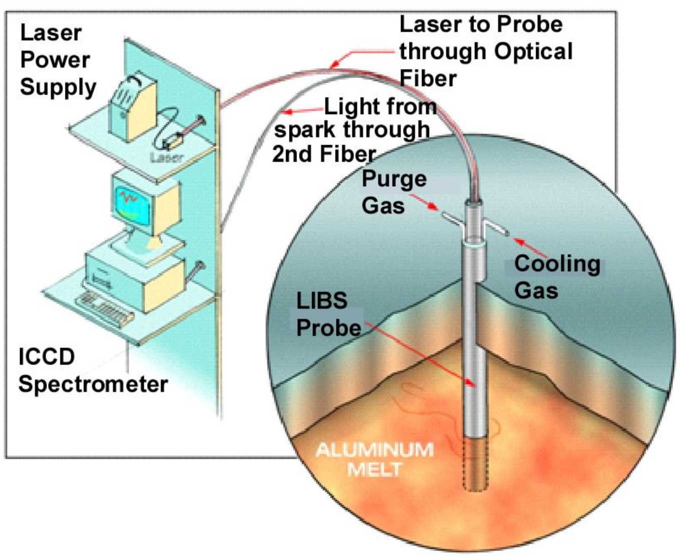
Particle Detection by Ultrasound (US)

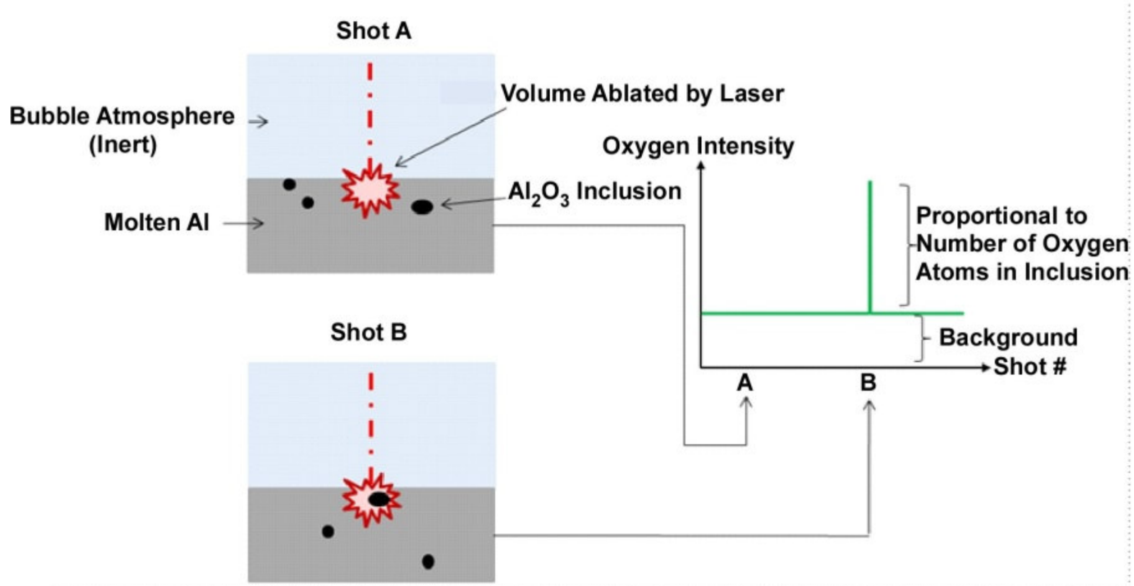
- Known concept for inclusion detection in liquid Al alloys (e.g. Metalvision 20/20); advantage compared to LiMCA: larger sample volume
- Parallel to AMAP P4: AiF project on US with all AMAP P4 partners, other companies, Fraunhofer IZPF and IfG (later foundry institute RWTH Aachen university)
- Focus on basic setup, guide rod materials, signal analysis, etc.
- Support of AiF project by AMAP P4 especially after change from IfG to GI
- Experimental trials with prototype in reference setup parallel to LiMCA and in Nemak plant in Dillingen showed promising results but also further challenges





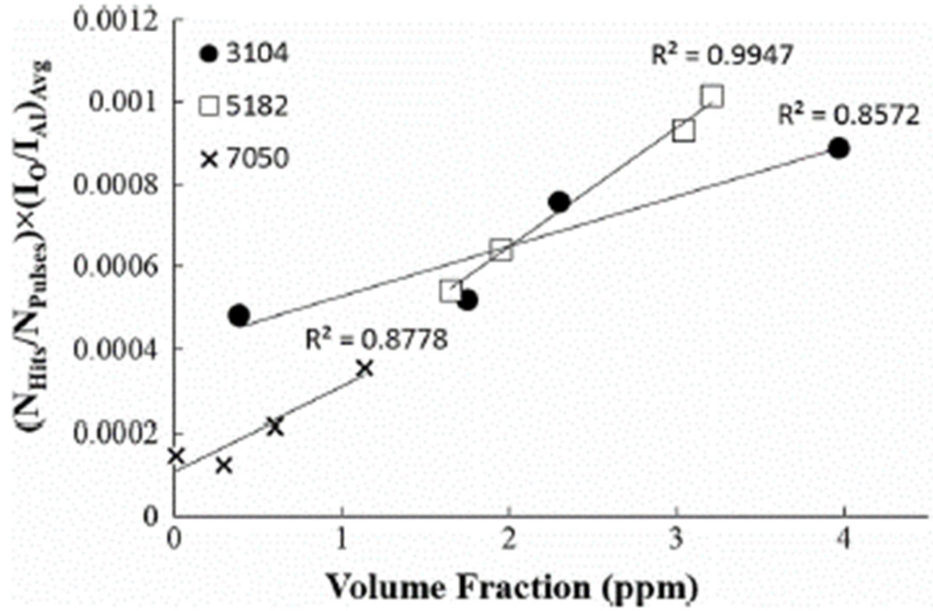
- Principle: Identification of non-metallic inclusions based on analysis of local chemical composition by laser ablation and spectrometer analysis
- Potential advantage: shows not only number but also composition of inclusions
- Parallel to AMAP P4: Work on detection of non-metallic inclusions in Al melts by LIBS in ACRC project together with WPI Worcester (Prof. Diran Apelian) and ERCo; funding by several industrial companies → focus on foundry alloys
- Extension of ACRC project regarding wrought alloys funded by AMAP P4 in 2015





- Feasibility test on three different wrought alloys in lab scale crucible furnace:
  - AA3104
  - AA5182
  - AA7050
- Addition of synthetic inclusions for different levels of cleanliness
- LIBS shows certain sensitivity for inclusions

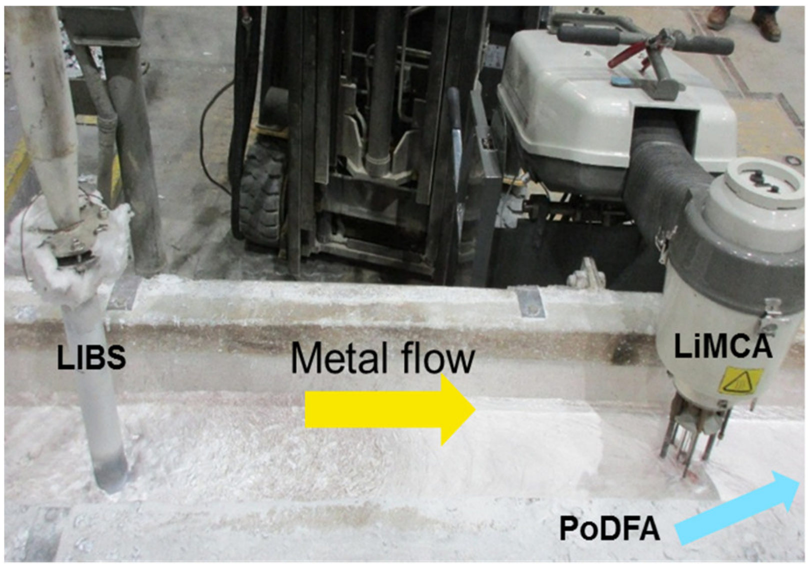
→ Decision to carry out plant trials



Calibration curve relating LIBS signal-to-oxide volume fraction in wrought alloys

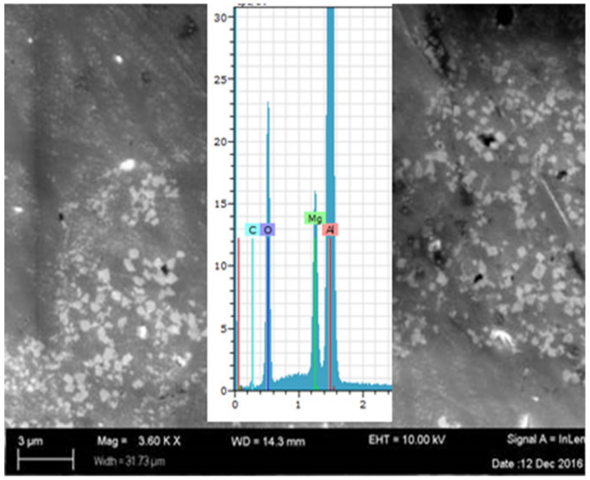
S. HUDSON, J. CRAPARO, R. DESARO, D. APELIAN
Inclusion Detection in Aluminum Alloys via Laser-Induced Breakdown Spectroscopy
Metallurgical and Materials Transactions B, Published on-line on Jan 10, 2018

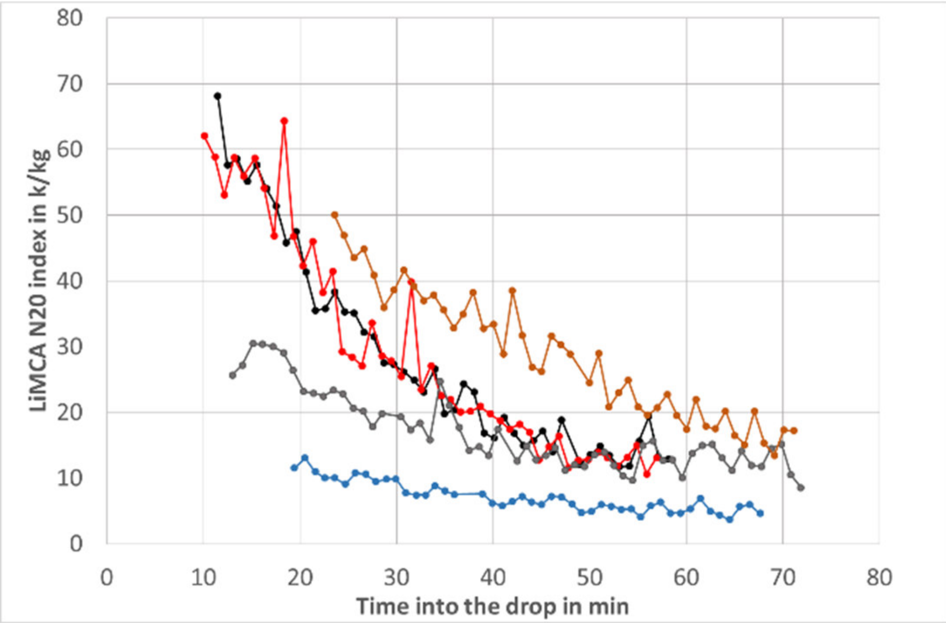
- Industrial test organized at Constellium Muscle Shoals, Alabama.
  - 5 test drops (3xxx) with 2 target inclusion loads
  - Measurement: casting furnace exit
  - LiBS, LiMCA (reference), PoDFA/Prefil, chemistry

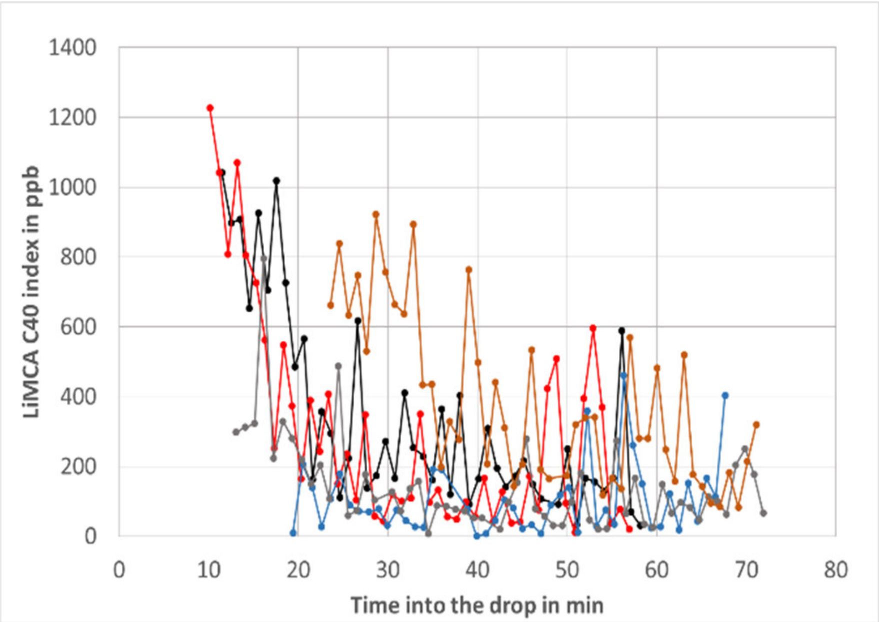


	Inclusion loading	Position	Characterisation
Drop 1	High	Holder exit	LIBS, LiMCA, chemistry
Drop 2	High	Holder exit	LIBS, LiMCA, chemistry
Drop 3	Low	Holder exit	LIBS, LiMCA, PoDFA, chemistry
Drop 4	Low	Holder exit	LIBS, LiMCA, PoDFA, chemistry
Drop 5	High	Holder exit	LIBS, LiMCA, PoDFA, chemistry

- LiMCA: Successful in covering a broad inclusion range:
  - LiMCA N20 counts from 10 to 60 k/kg
  - C40 below 1000 ppb
  - Observation of O-containing inclusions (PoDFA/Prefil)



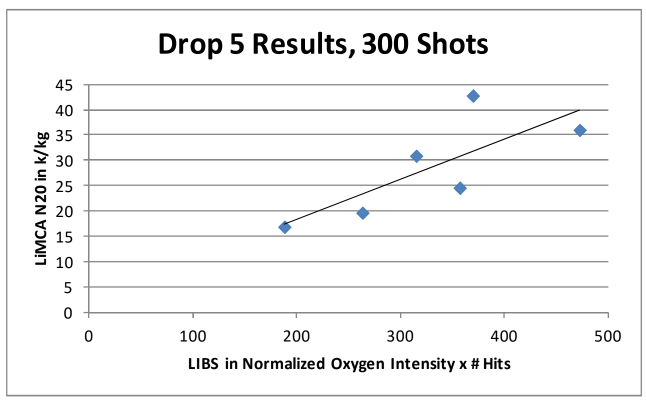




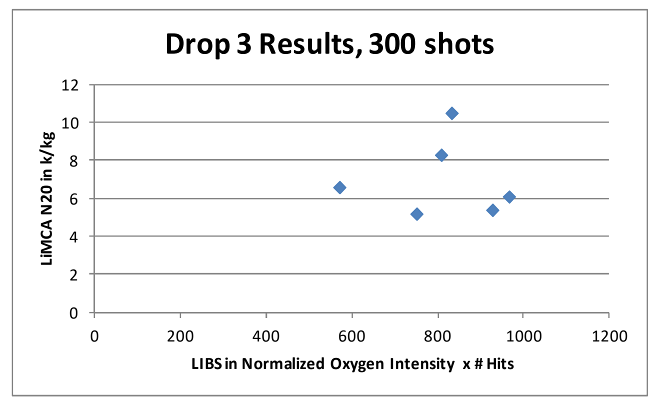
LIBS – Test under Industrial Conditions

- Robustness of the prototype/principle proven (100 % uptime during trials)
- Some oxygen peaks were successfully measured
- Only in some casts correlation between LiMCA and LIBS
- Sensitivity not proven to be sufficient for demanding applications
- Chemical composition data potentially of interest
- Currently no further common activities on LIBS planned within the P4 consortium

Good correlation LiMCA/LIBS



No correlation LiMCA/LIBS



- Good collaboration and open discussions between project partners
- Very similar background of industrial participants was both
  - advantageous, because of good common understanding and common target
  - disadvantageous, due to same area of specialization and therefore not a very broad range of different skill sets
- Fundamental understanding of settling and agglomeration of non-metallic inclusions in aluminium melts improved and simple models for inclusion behaviour developed
- Initial target of plant trials with US and LIBS prototypes fulfilled
- Both technologies showed promising results but also open issues
- Potentially further development of US detector together with IZFP in AMAP P20
- AMAP project P4C on deeper understanding of particle behaviour at IME with Aleris, Constellium, Hydro, Magma, Nemak, Trimet and Foseco has started recently

- 5 full text publications
  - Bath Movement Effect on Agglomeration of Inclusions in Aluminium Melts, IMMC 2014
  - Study of Particle Settling and Sedimentation in a Crucible Furnace, TMS Light Metals 2015
  - Observation on inclusion settling by LiMCA and PoDFA in aluminium melts, Int. Al Journal April 2015
  - Assessment of Settling Behavior of Particles with Different Shape Factors by LiMCA Data, TMS Light Metals 2016
  - A Review on Prerequisites of a Set-up for Particle Detection by Ultrasonic Waves in Aluminium Melts, Open Journal of Metal

- 2 presentations at conferences without manuscripts
  - Leichtmetallfachausschuss (light metals expert committee), GDMB, Neuss, March 2015
  - Lagrangian transport: from complex flows to complex fluids, Lecce Italy, March 2016

- 1 PhD thesis
- 4 master theses
- 3 mini theses

Thank you very much for your attention!

Questions?