Analysis and possible sources of dust particles extracted from the LHC

A. T. Pérez Fontenla CERN EN-MME-MM

Workshop on Dust Charging and Beam-Dust Interaction in Particle Accelerators 14.06.2023

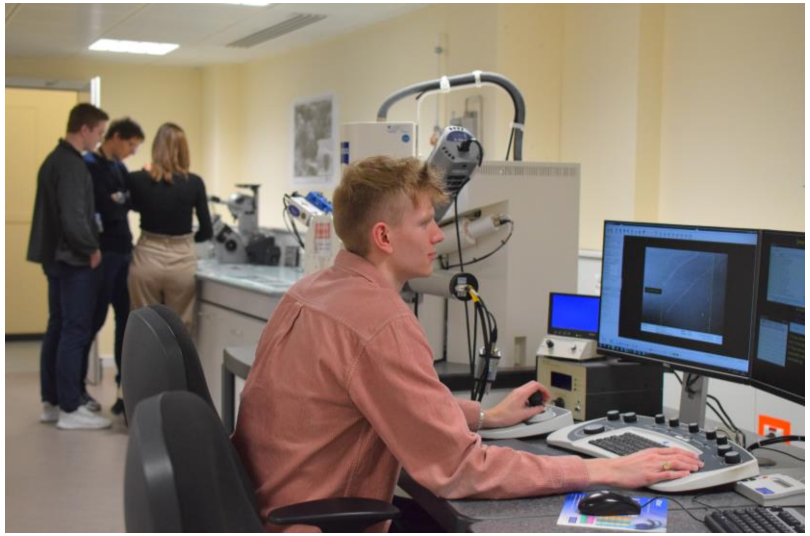
Materials science at CERN: MM Section

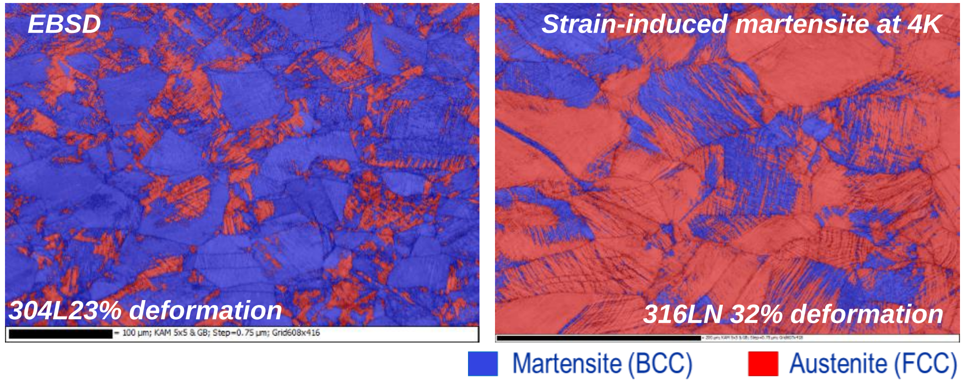
MM Section have an extensive experience in materials characterization and our equipment covers conventional and advanced techniques in microstructural and mechanical testing, dimensional control and NDT

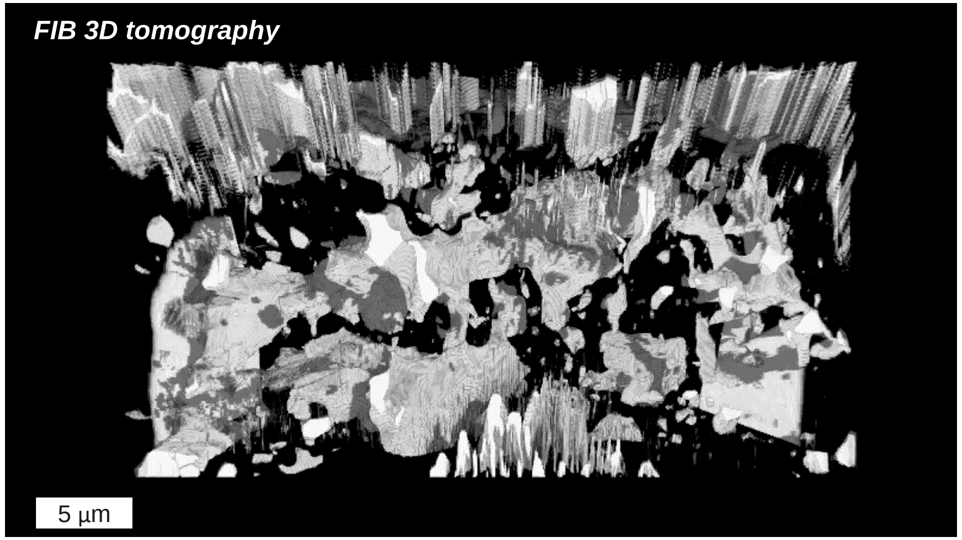
OM, SEM, FIB-SEM, STEM, EDS, EBSD, APA, XRD, residual stress, hardness, tensile testing, fatigue, FT, nano-indentation, µCT, UT, RT…

UHV, magnets, cryogenics, irradiation zones…

Materials selection, technical specifications, researching, qualification, failure analysis…







Particles analysis applications at CERN

Raw material cleanliness:

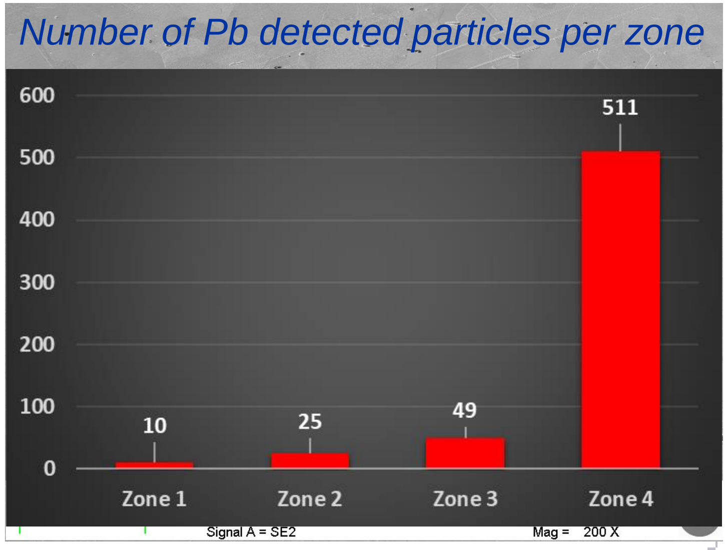
Analyse the type and density of non-metallic inclusions in metals and steels, or impurities in pristine powder for additive manufacturing…

Technical cleanliness:

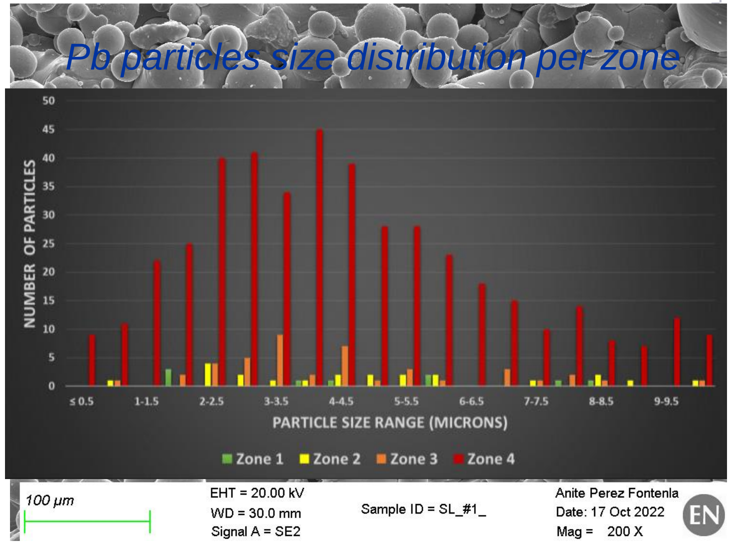
Identify the source of contaminants in manufacturing processes: after precision machining, thermal treatments, comparison before and after service/cleaning…

Environmental analysis:

Pollution study of a certain contaminant in a localized working area by analysing particles in air filters or from specific locations sampling



Non-metallic inclusions in Ni–Co ferrous alloy



3D printed Ti-alloy spiral load

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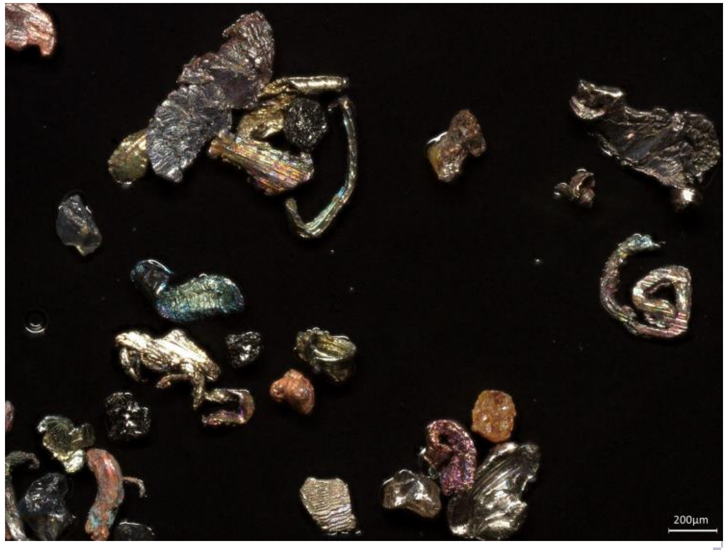
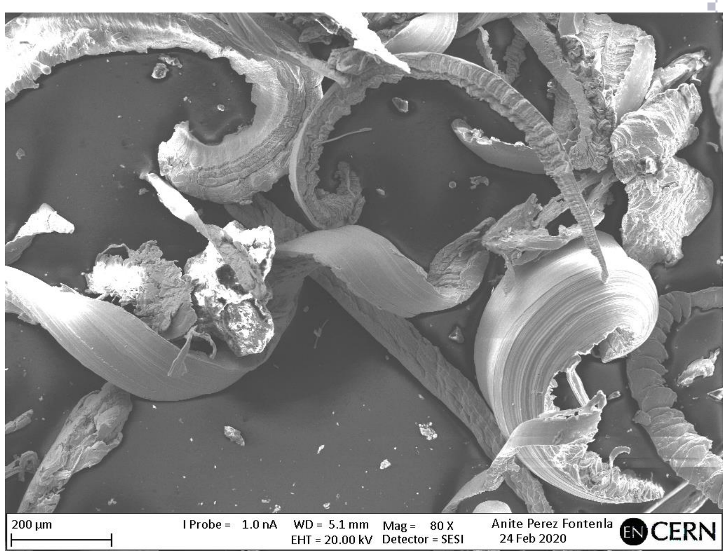
Cooling system filters:

Monitor air quality and pollution by analysing particles in air or water filters



Residue in cryo-line



Residue in water cooling circuit after works

Particles analysis applications at CERN

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Monitor air quality and pollution by analysing particles in air or water filters

Unidentified Falling Object (UFO):

Dust particles of unknown nature and origin extracted from the LHC components

Sampling extension:

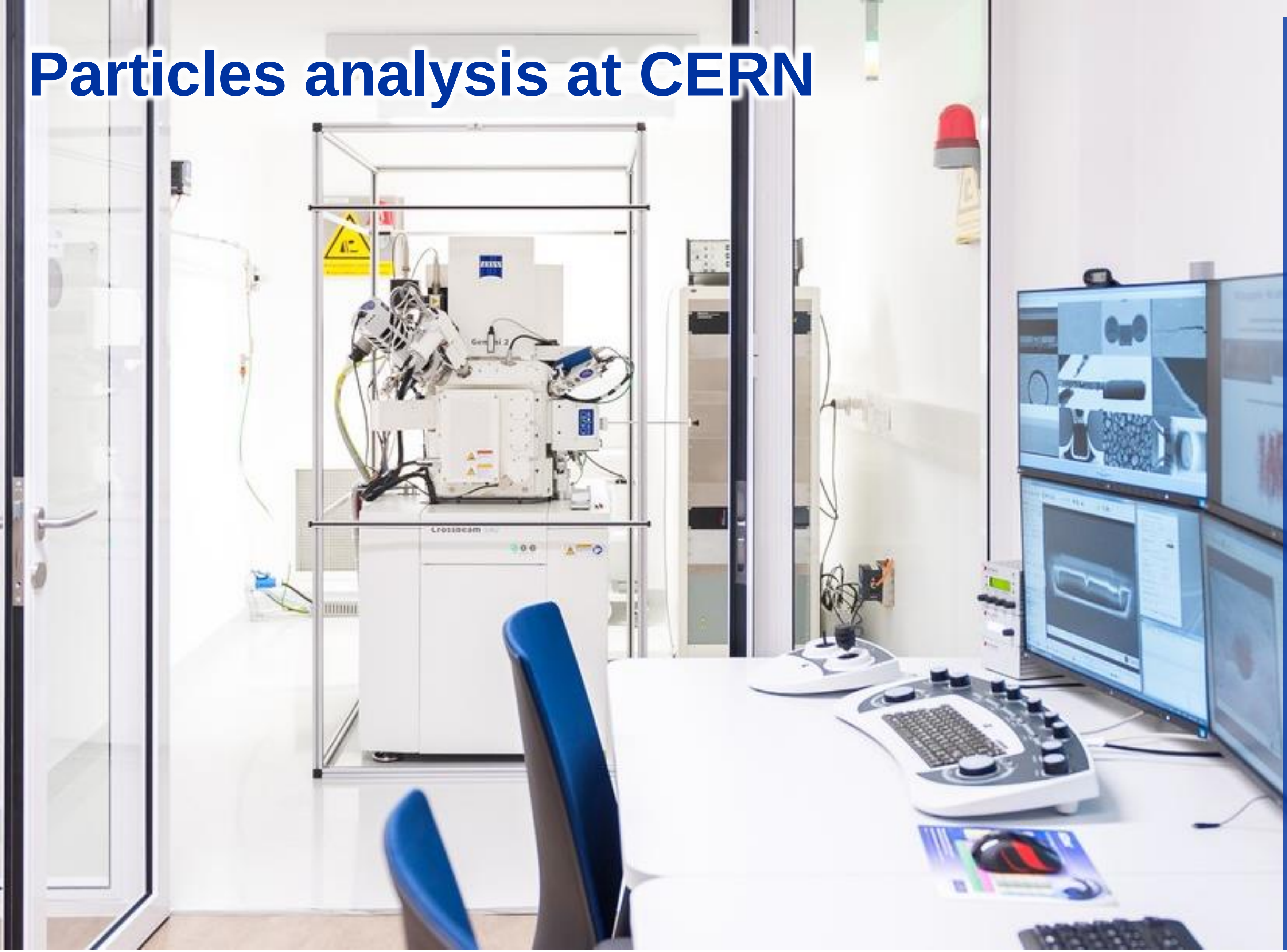
Specimen

Room/ laboratory

Circuit

LHC

Particles analysis at CERN



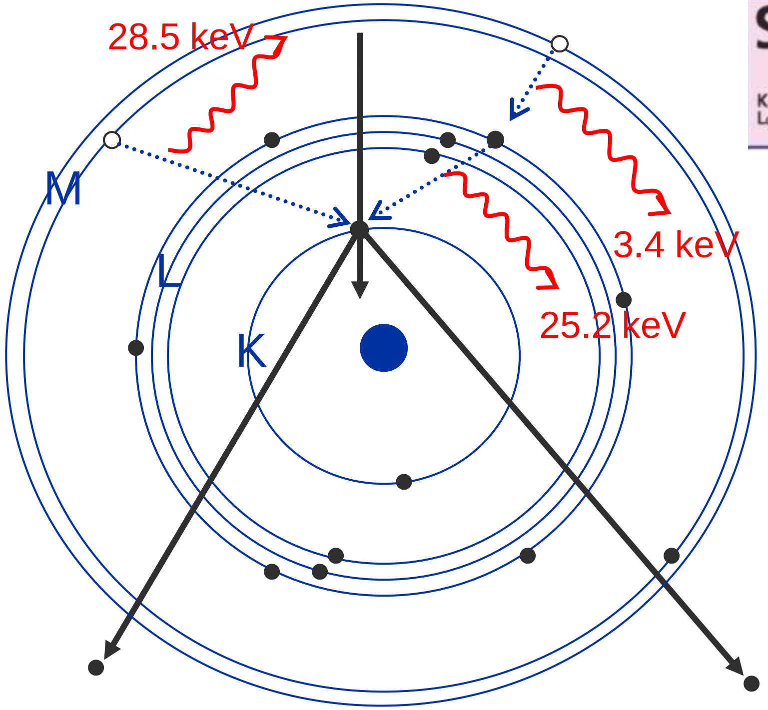
SEM-based microanalysis: Energy Dispersive X-ray Spectroscopy (EDS)

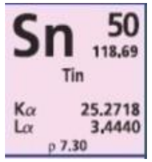
Requirements:

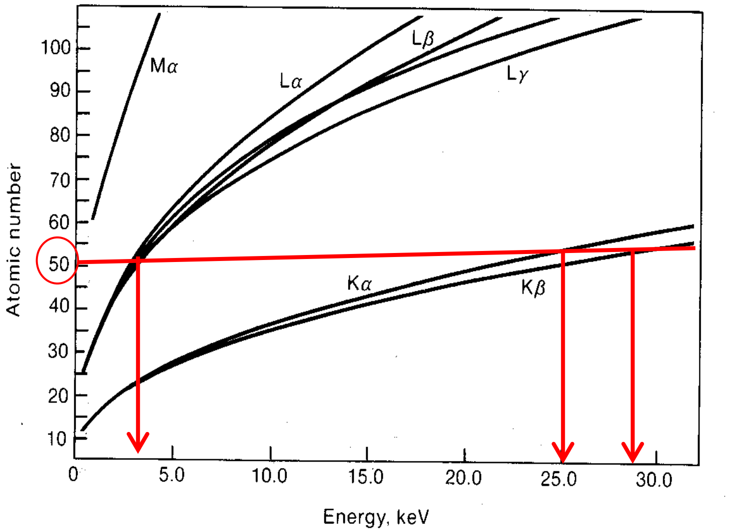
- Specimen SEM inspectable
- Non volatile/ hazarous particles
- Nanometric to micrometric

Particles analysis at CERN

- Analysis of X-rays emitted by the matter in response to being hit with charged particles
- The energies of the emitted photons are characteristic for each element







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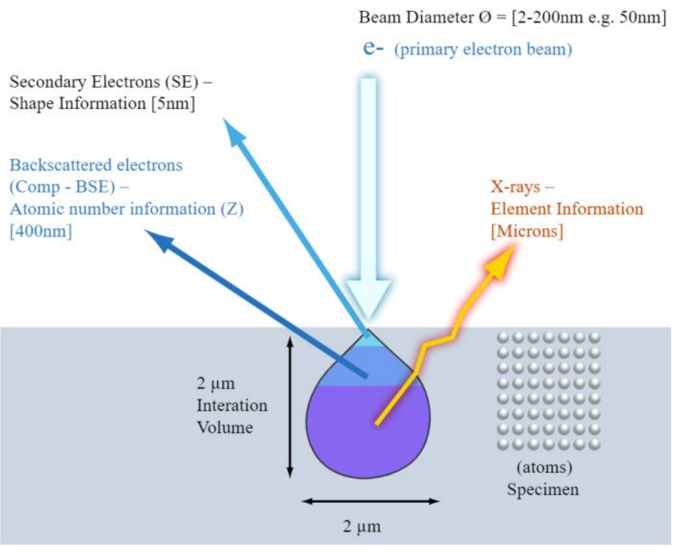
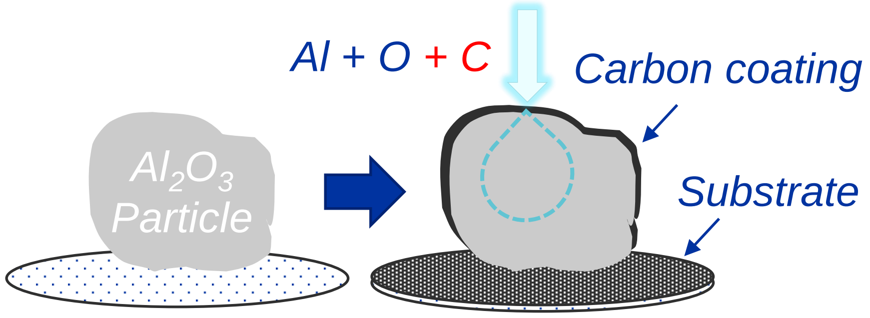
Particles analysis at CERN

- Analysis of X-rays emitted by the matter in response to being hit with charged particles
- The energies of the **emitted photons are characteristic for each element**
- **Detection limits** with conventional systems makes impossible to detect presence of elements below around 0.2 wt. % (the value depends on the weight and the matrix around), or light elements
- **Conductive coating is necessaire** (carbon is the most appropriate for EDS studies)





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Particles analysis at CERN

- The evaluation can be done directly on the **sample surface** with minimum preparation, extracted with **adhesive tabs or filters/membranes** (normally C-based)
- A good alternative is to perform a **liquid-based extraction** where all the particles present in the liquid are recovered in a filter or membrane

SEM

Coating
+
SEM

SEM-based microanalysis: Energy Dispersive X-ray Spectroscopy (EDS)

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Sampling is key:

- Good contrast with the substrate (background)

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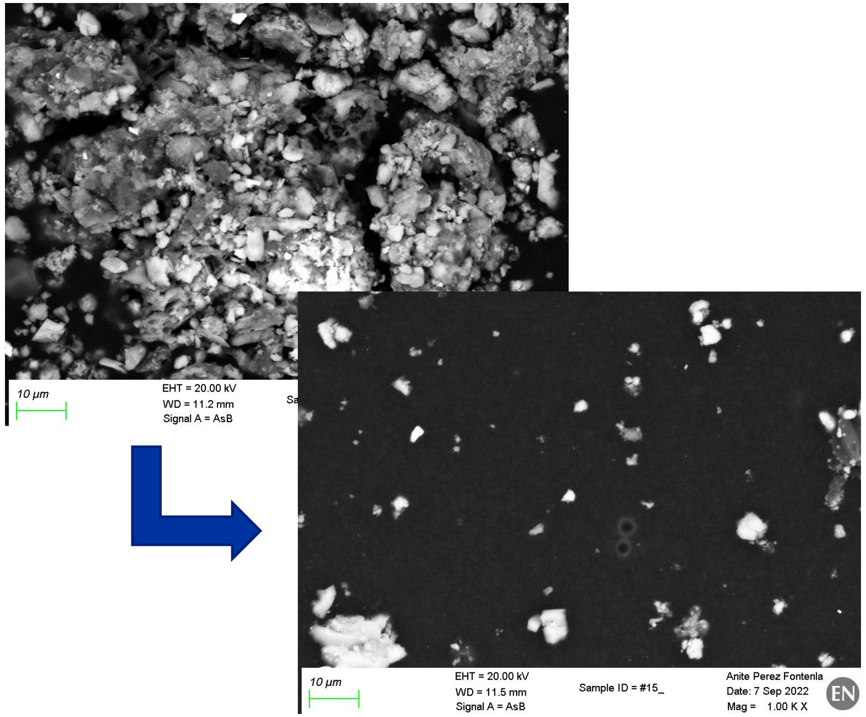
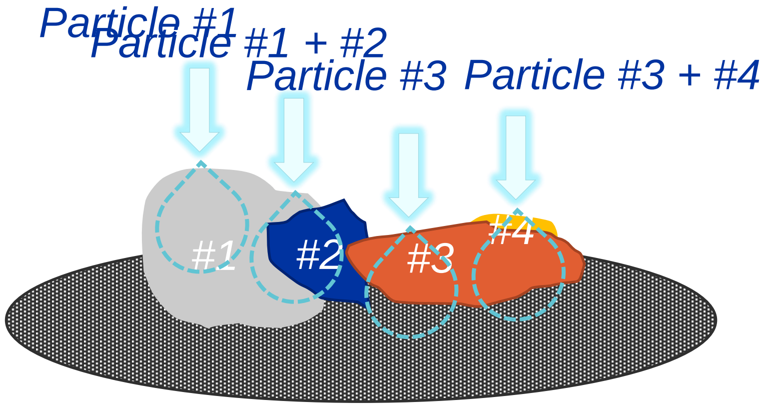
- **Overlapping** will lead to size overestimation and non-representative chemical results





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Sampling is key:

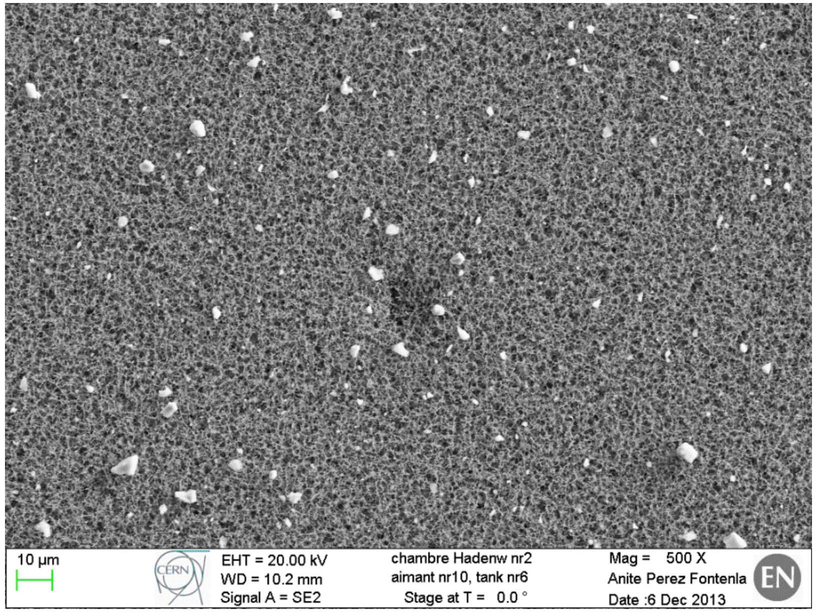
- Good contrast with the substrate (background)
- Not-overlapping between particles

SEM-based microanalysis: Energy Dispersive X-ray Spectroscopy (EDS)

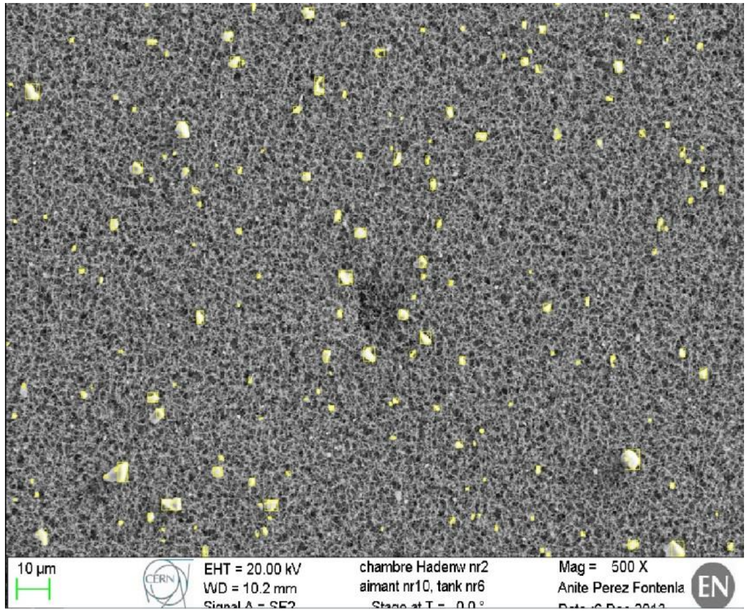
UFO analyses at CERN: manual process

- By using a detector that provides the best possible contrast between the particles and the filter, the counting was done initially by hand in at least 5 representative (500x) fields and since 2013 by image analysis
- The number of particles per filter were then extrapolated to the full filter surface

Original SEM image



SEM image after particle analysis



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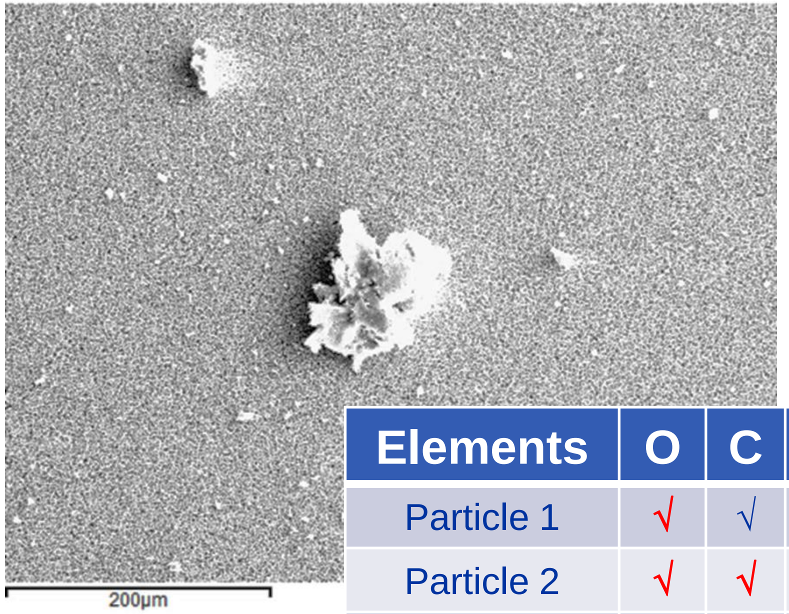
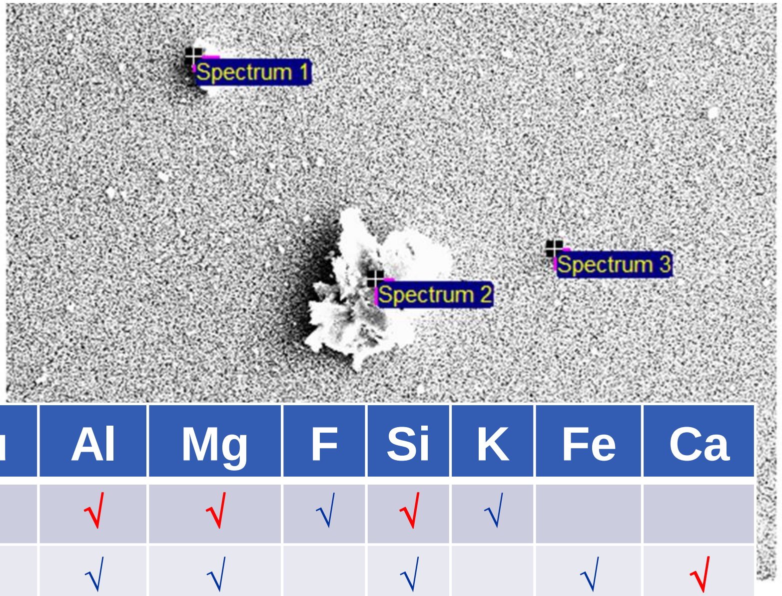
- The nature of the particles was evaluated in tens of them

Original SEM image



EDS spectra identification



Elements	O	C	Au	Al	Mg	F	Si	K	Fe	Ca
Particle 1	√	√	√	√	√	√	√	√		
Particle 2	√	√	√	√	√		√		√	√
Particle 3	√	√	√	√			√			

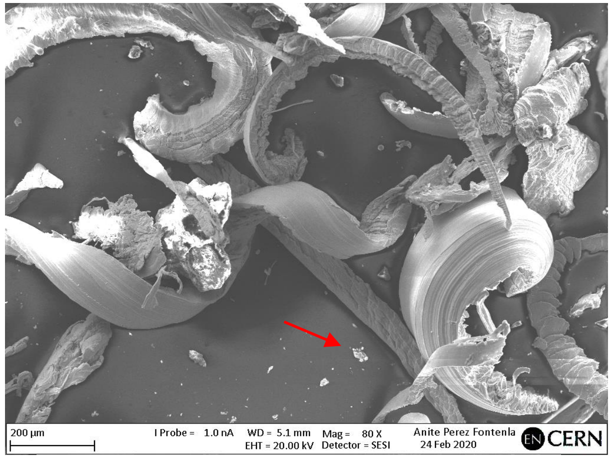
SEM-based microanalysis: Energy Dispersive X-ray Spectroscopy (EDS)

Automatic Particle Analysis (APA)

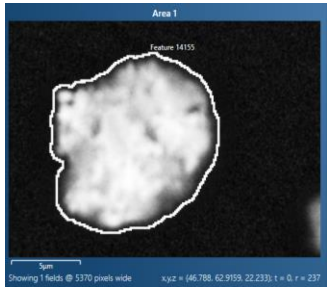
UFO analyses at CERN: APA process

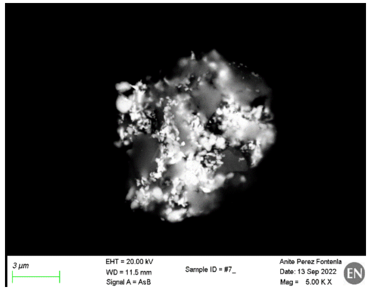
- This technique is widely used in crime forensics to identify gunshot residue and for identifying precious metals in ore samples
- By processing the electron image, candidate features can be identified rapidly in contrast with the background of the field under study
- Since 2017: AzTEC-features from Oxford Instruments analyses each particle in seconds and automatically measures composition and morphological parameters

Sampling and definition of the test parameters are critical

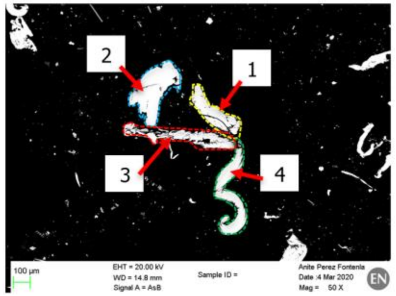


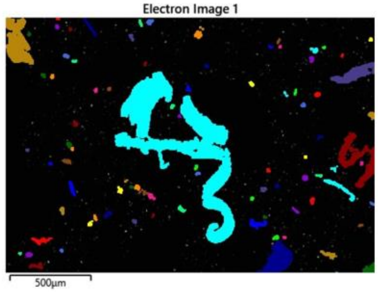
Which minimum size we define?





Agglomerated particles can lead to artefacts





UFO analyses at CERN: APA process

SEM-based microanalysis: Energy Dispersive X-ray Spectroscopy (EDS)

Automatic Particle Analysis (APA)

Composition and morphology

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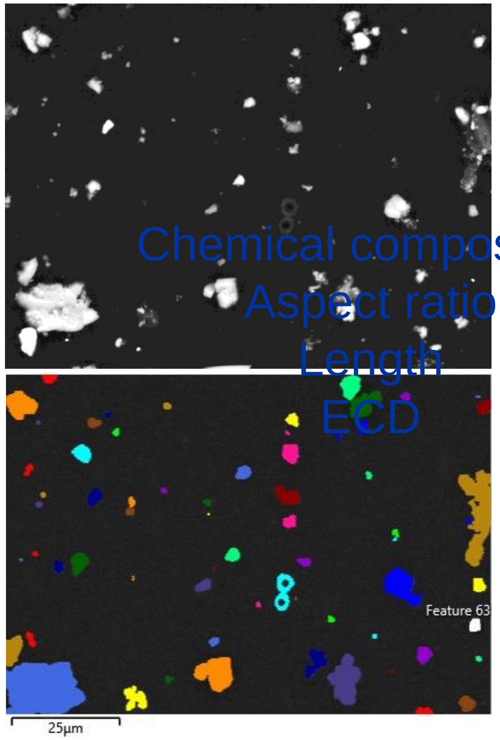

Chemical composition
Aspect ratio
Length
ECD

SEM-based microanalysis: Energy Dispersive X-ray Spectroscopy (EDS)

Automatic Particle Analysis (APA)

Composition and morphology

Thousands of particles characterized

Results can be incorporated into particle classification schemes

Particles with 100 wt. % C and/or O are not classified in UFO studies

UFO analyses at CERN: APA process

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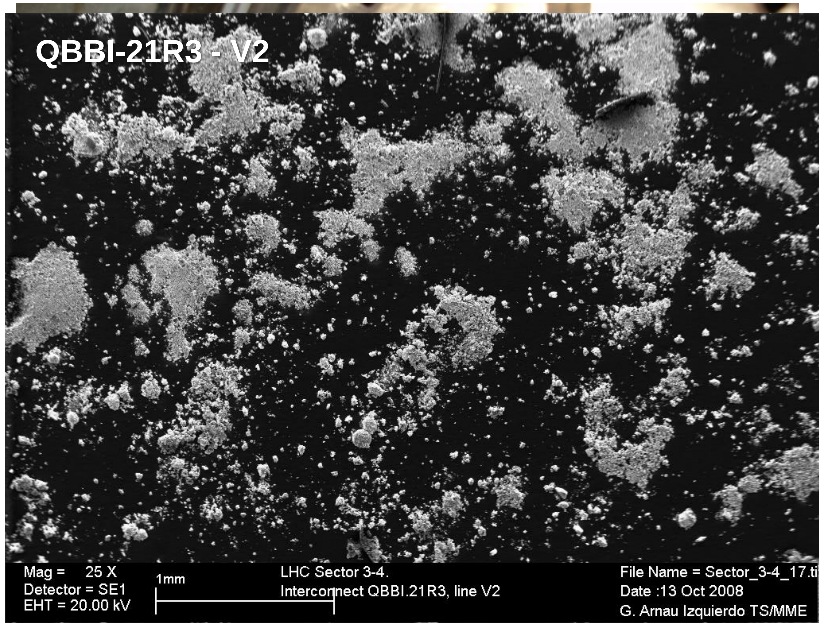
- By processing the electron image, candidate features can be identified rapidly in contrast with the background of the field under study
- Since 2017: AzTEC-features from Oxford Instruments analyses each particle in seconds and automatically measures composition and morphological parameters
- From tens of particles analysed with the manual process to thousands with APA. The analysis run normally during the night and weekends
- The data collected is stored and saved per particle and can be exported to other formats

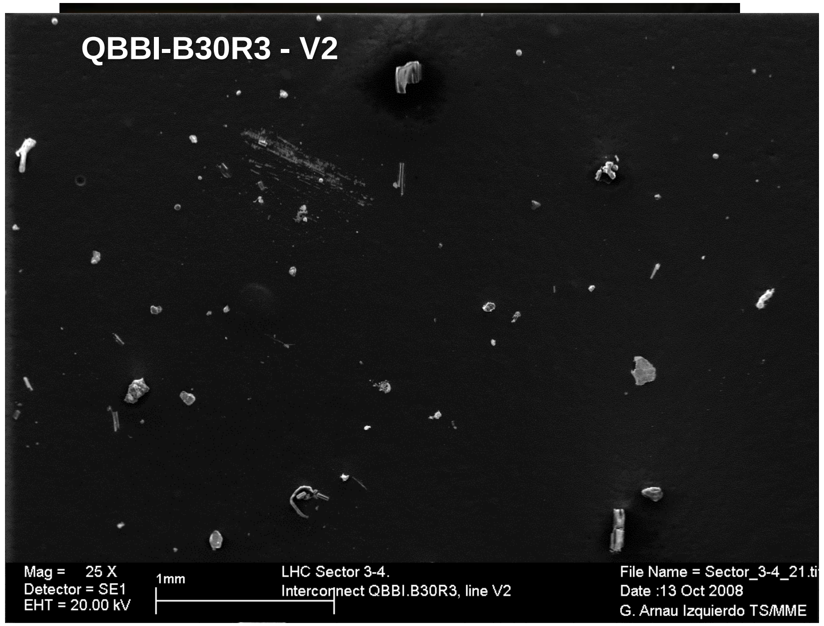
Category	Features	% Total Features	Class Features Area (sq. µm)	% Features Area
All features	20137	100	9.99E+04	100
Classified	11501	57.11	9.2E+04	92.08
No Classification	8636	42.89	7.91E+03	7.92
Marked	0	0	0E+00	0

UFO analyses at CERN: LHC components

- **2008**: a series of interconnects were opened in sector 3-4
- Dust extraction from the vacuum components: plug-in modules (**PIM**) and beam screens (**BS**)
- Particles sampled with **adhesive tabs** put against the inner wall of the components → **Composition and morphology**
- QBBI-21R3-V1 and QBBI-B30R3-V2 presented few individual particles (polymer chip and particle that contains In+Cu)

EDMS 972804 G. Arnau Izquierdo





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- QBBI-21R3-V1 and QBBI-B30R3-V2 presented few individual particles (polymer chip and particle that contains In+Cu)
- QBBI-21R3-V2 presented macroscopic clusters covered with sub-micrometric particles and many with droplet shape. The composition corresponds to materials melted in the interconnection due to an isolated incident: (Cu rich + Sn and Si and Fe+Cr+Ni+Mo)

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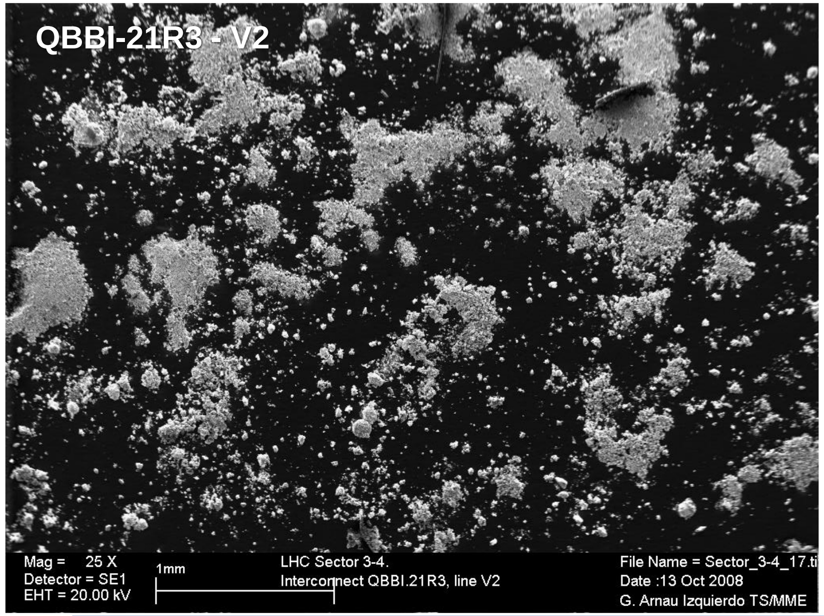
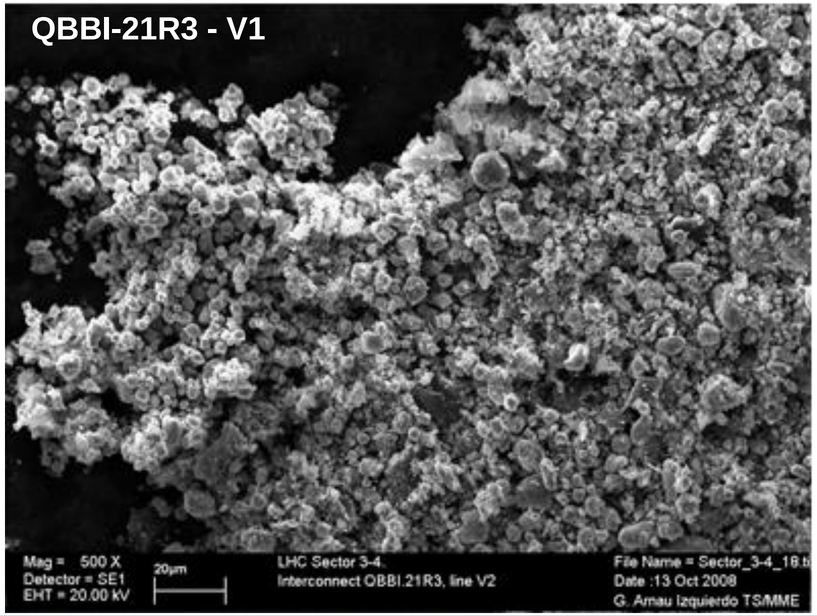
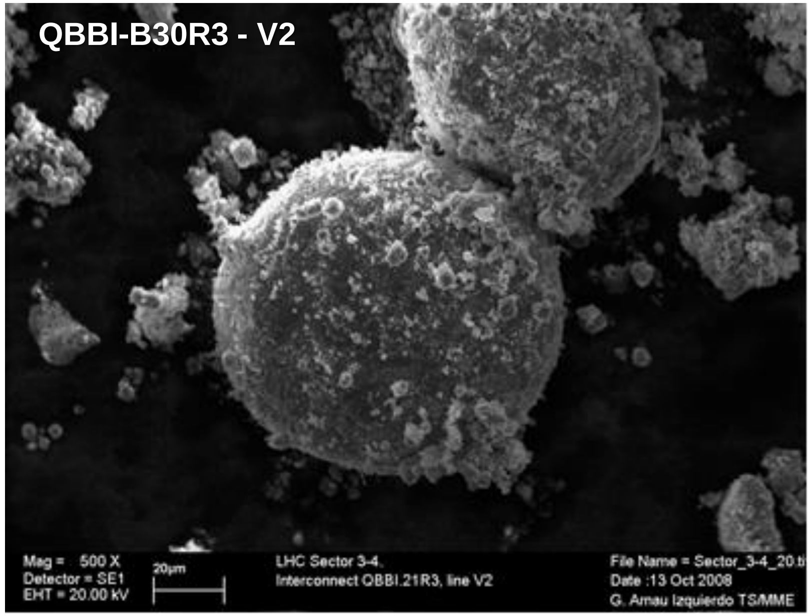
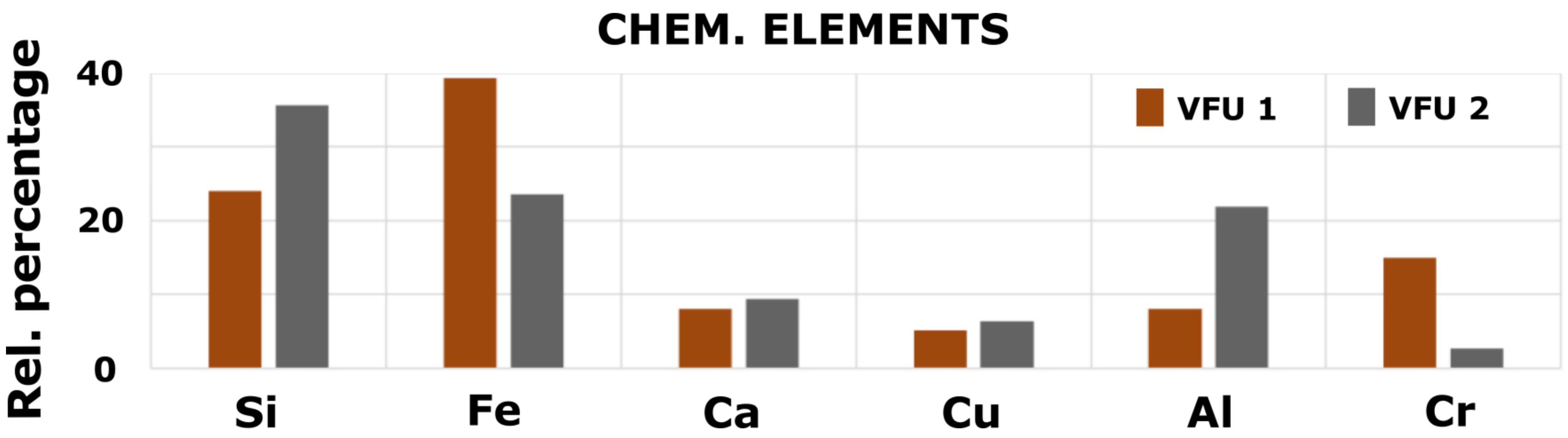






UFO analyses at CERN: LHC components

- **2016:** a dipole magnet was removed from the LHC (sector 12)
- Vacuum filter units **(VFU)** with pore size <10 µm were installed during ball-test to collect the transported dust → **Composition**
- **Particles < 5 µm were excluded** from the analysis, since their impact on the LHC operation is considered **negligible**
- **Fe, Cr and Cu** are **intrinsic materials** of the LHC vacuum system
- **Si, Ca and Al** are considered to have an **external origin**: airborne particles in the tunnel containing concrete dust, which might have entered during installation and maintenance

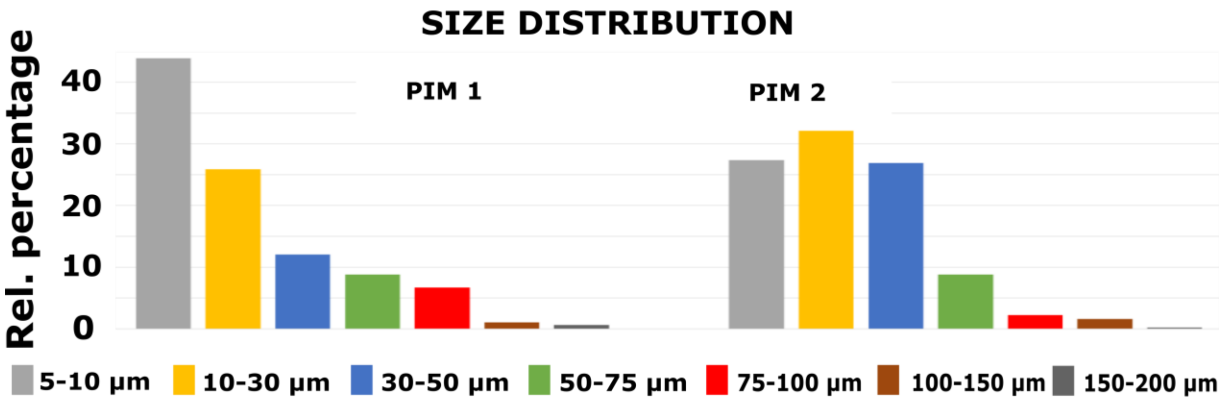


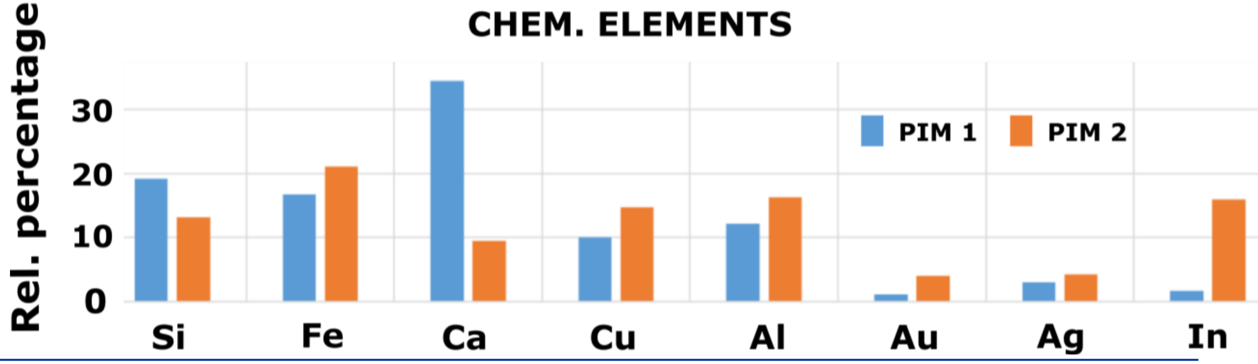
Results from "Dust analysis from LHC vacuum system to identify the source of macro particle-beam-interactions. L.K. Grob et al."

UFO analyses at CERN: LHC components

Plug-in module

- **2016:** a dipole magnet was removed from the LHC (sector 12)
- 4x **PIM →** compensate expansion and contraction during warm-up and cool-down
- Gold-coated RF fingers and static RF contacts, which are vacuum brazed to the copper structure
- Particles evaluation between 5 and 200 µm → **Size distribution and Composition**
- More than 80% of the analysed particles present less than 50 µm
- **Fe, Cr, Cu, Au, In and Ag** from the are **intrinsic materials** of the LHC vacuum system and PMIs
- **Si, Ca and Al** are considered to have an **external origin**: airborne particles in the tunnel containing concrete dust, which might have entered during installation and maintenance

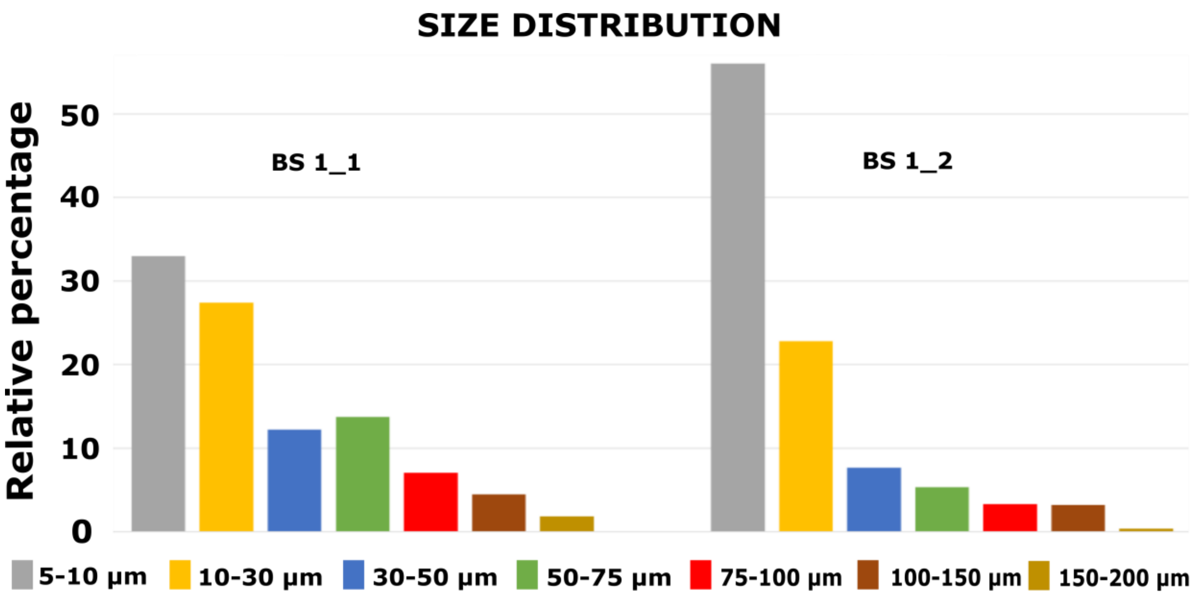


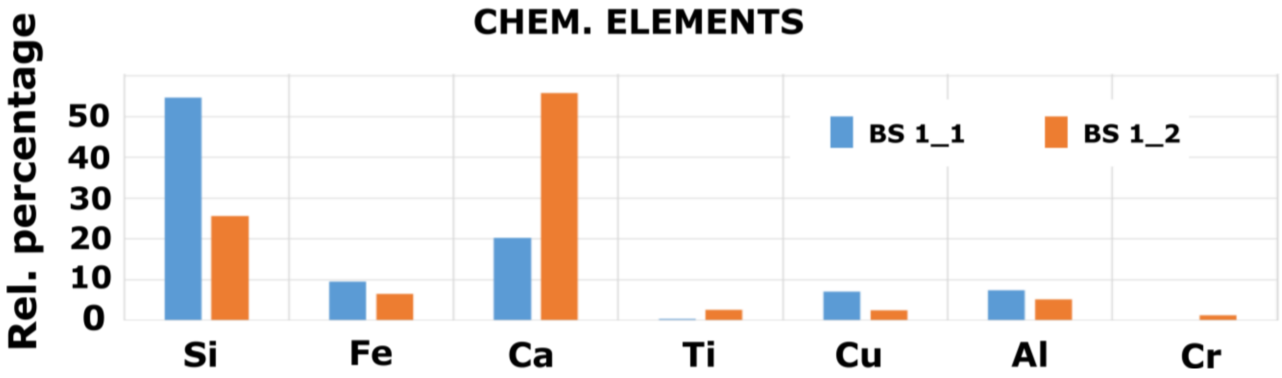


UFO analyses at CERN: LHC components


Beam screen & cold bore

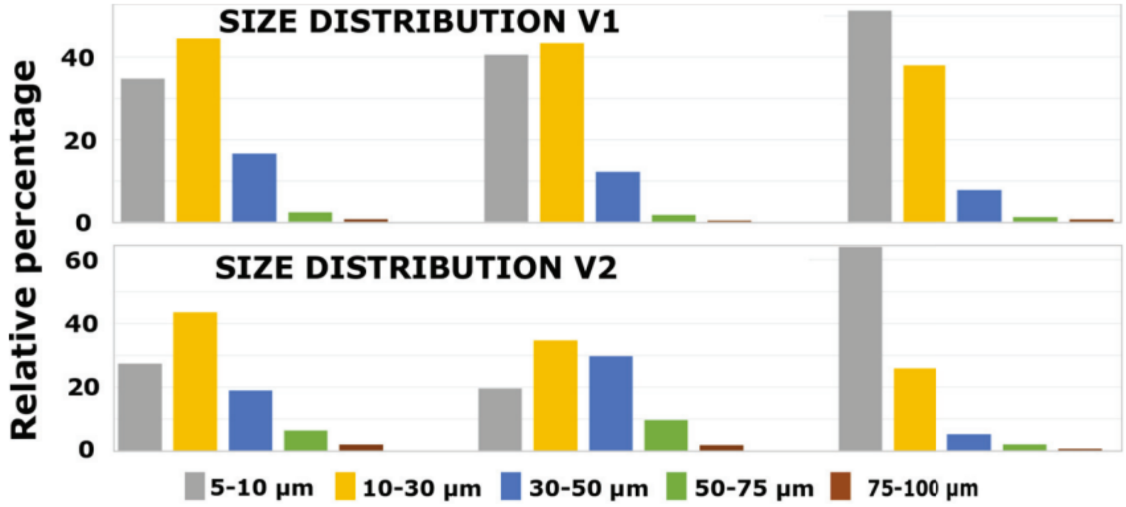
- **2016:** a dipole magnet was removed from the LHC (sector 12)
- 2x **BS** → Co-laminated low permeability 1mm thick austenitic stainless steel strip with a 75 µm copper
- Particles evaluation between 5 and 200 µm → **Size distribution and Composition**
- The second part (BS1_2) exhibits a larger amount of small sized particulates in the range below 10 µm
- Similar elemental composition for the dust found in both parts, but the peaks in **Si** and **Ca** seem complementary
- Also remarkable the presence of **Ti** only detected in the form of traces on previous samples

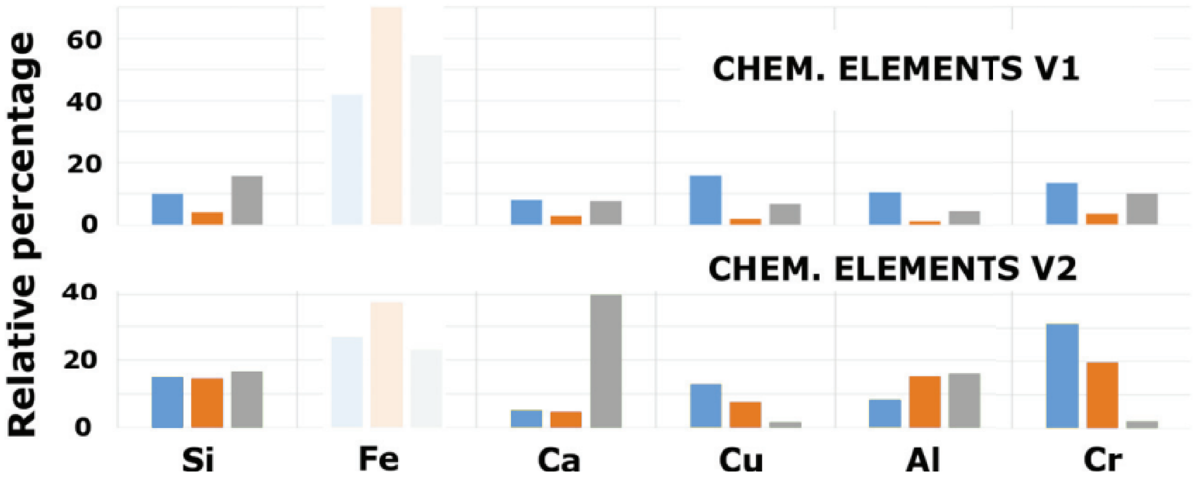




UFO analyses at CERN: LHC components

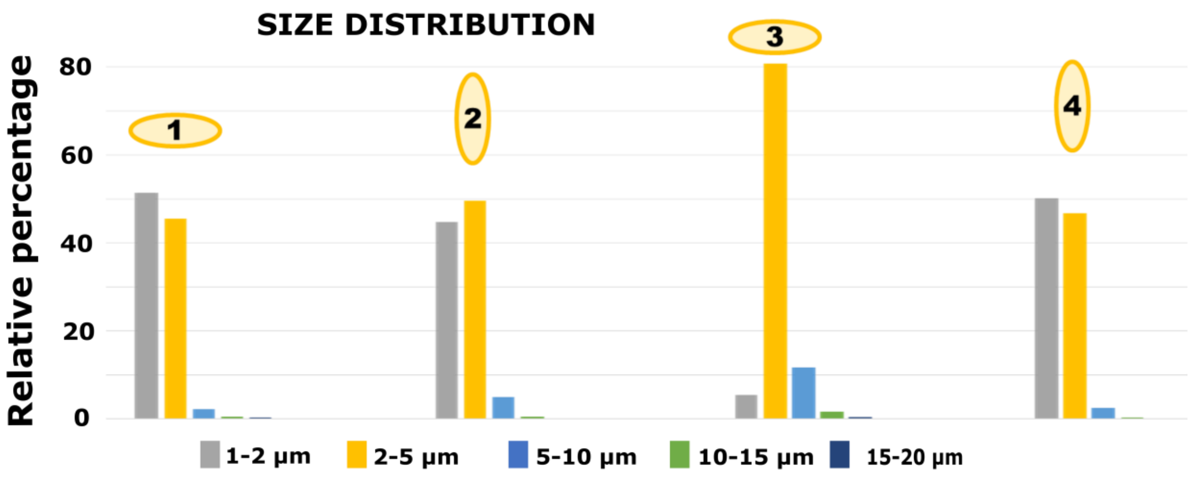
- **2016:** a dipole magnet was removed from the LHC (sector 12)
- 2x **CB** → Seamless AISI 316 LN. External diameter 53 mm, wall thickness 1.5 mm and 16 m long pipe
- Particles evaluation between 5 and 100 µm → **Size distribution and Composition**
- A train of **three cleaning electrostatic dusters** was pulled through the pipe and subsequently packed for particle extraction
- Similar elemental composition for the dust found in both parts, but higher presence of **Si, Ca** and **Al** in V2 (opened before cleaning for magnetic measurements)
- More particles in V2 but with same size than V1 → external contamination brought in by the measurement is comparable with the one already present inside the cold bore

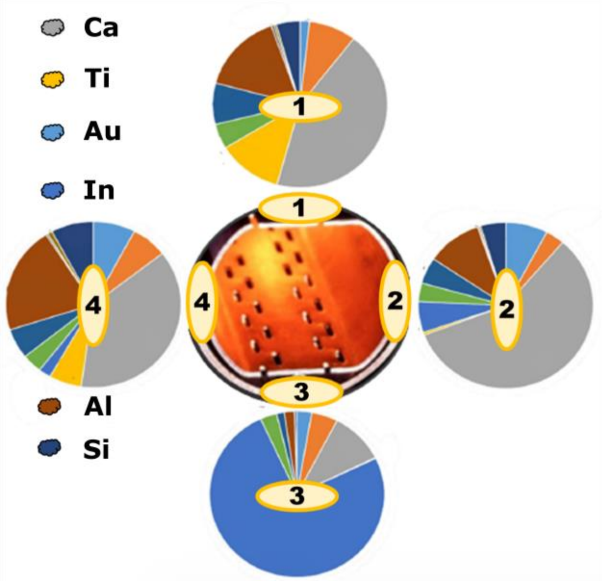




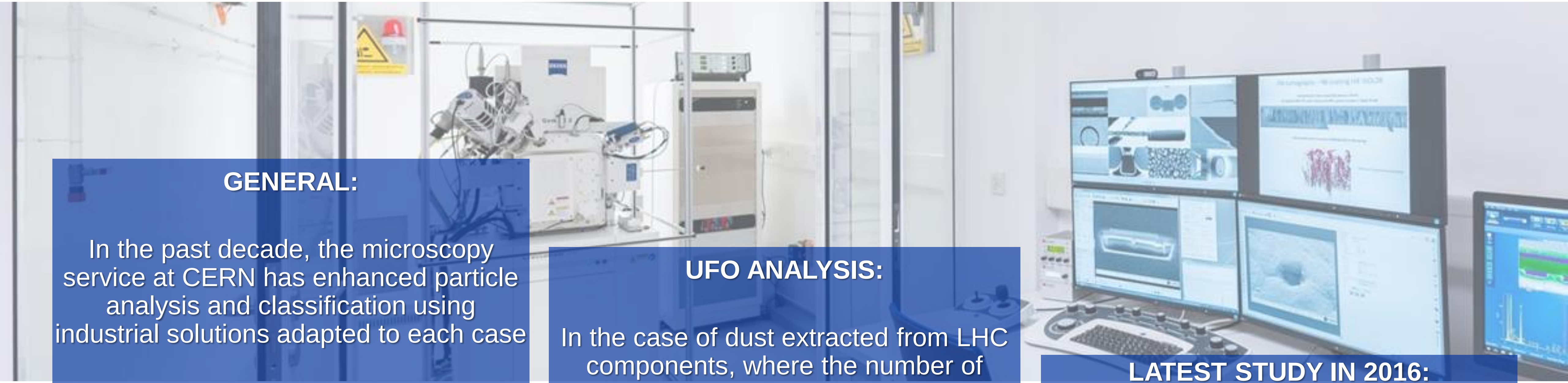
UFO analyses at CERN: LHC components

- **2016:** a dipole magnet was removed from the LHC (sector 12)
- **BS →** Position-Sensitive Wipe Test (gravity influence)
- Sampling included the apertures of the adjacent magnets remaining inside the ring. Figure shows data analysis for left aperture of QBBI.A31L2
- Particles evaluation between 1 and 20 µm → **Size distribution and Composition**
- The size distribution shows that most of the particles have a diameter below the defined limit of 5µm.
- The bottom position (#3) presents a higher amount of **In** while the three other positions mainly exhibit **Al, Si** and **Ca** and only small fractions of **Au** and **In.**





Summary



GENERAL:

In the past decade, the microscopy service at CERN has enhanced particle analysis and classification using industrial solutions adapted to each case

The new automatic SEM-based tool generates large amounts of data compared to manual methods and enables storing detailed information for each particle

The sampling procedure is crucial for obtaining representative specimens and valuable data

UFO ANALYSIS:

In the case of dust extracted from LHC components, where the number of specimens is limited, the ability to analyze thousands of particles per sample is crucial

The analysed UFOs mostly consists of small sized macro-particles and the nature varies over the components (different manufacturing processes and materials)

LATEST STUDY IN 2016:

Ca-, Al- and Si-based contaminants were present in all samples and could be originating from the concrete dust inside the tunnel or manufacturing environments

Additionally, for the PIM, also wear and tear could explain the relatively high amount of indium macro-particles found

Thanks for your attention!

Acknowledgements:
G. Arnau, E. García-Tabarés, J. Busom, L. K. Grob, C. Neves,
C. Charvet, B. Teissandier, A. Apollonio, H. Kos and R. Schmidt

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