

Cleanliness for Electron Microscopy

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 The world leader in serving science



Agenda

- Introduction
- Electron Microscope
- Parts, Material Aspects, Contamination and Cleanliness for Electron Microscopy
- New QEO-11-070/QEO-11-061
- Examples
- Q&A

We take pride in our Mission:

We enable our customers to make the world healthier, cleaner and safer

Customers worldwide trust Thermo Fisher Scientific products and services to help them accelerate innovation and enhance productivity. Together, we are advancing science to make a real difference. We do that by providing an unmatched combination of innovative technologies, purchasing convenience and comprehensive support through these and other product and service brands:



>125,000
colleagues



7,000
R&D scientists
and engineers



\$1.5B/YR
invested in R&D



\$44B
in revenue



>1,000,000
products

thermo
scientific

applied
biosystems

invitrogen

 fisher
scientific

unity
lab services

patheon

PPD

Complementary segments

Life Sciences Solutions

Genetic Sciences
SeqStudio CE System
QuantStudio 7 Real-Time PCR Systems
TaqPath COVID19 rtPCR test kit
Genexus Integrated Sequencer

Biosciences
GeneArt CRISPR Gene Editing
Cell Culture Media & Reagents

BioProduction
TruBio Discovery Automation System
Single-use Bioreactors

Specialty Diagnostics

Clinical Diagnostics
AcroMetrix Coronavirus 2019 RNA Control
B-R-A-H-M-S PCT Biomarkers

ImmunoDiagnostics
Phadia 200
ImmunoCAP Allergy and EliA Autoimmunity Tests
Viral Transport Media and Collection
Antimicrobial Susceptibility Testing Solutions

Microbiology

Transplant Diagnostics
AllType NGS Assays High-resolution Genotyping

Healthcare Market Channel

Analytical Instruments

Chromatography & Mass Spectrometry
Vanquish Core UHPLC
Orbitrap Exploris 480 MS

Electron Microscopy
KriosG4 Cryo-TEM

Chemical Analysis
Niton Apollo Handheld LIBS Analyzer

Instrument and Enterprise Services

Laboratory Products and Services

Lab Equipment & Consumables
TSX Series ULT Storage
Nalgene/ Nunc Labware
GP PRO Centrifuge
E1 Cliptip Electronic Pipette System

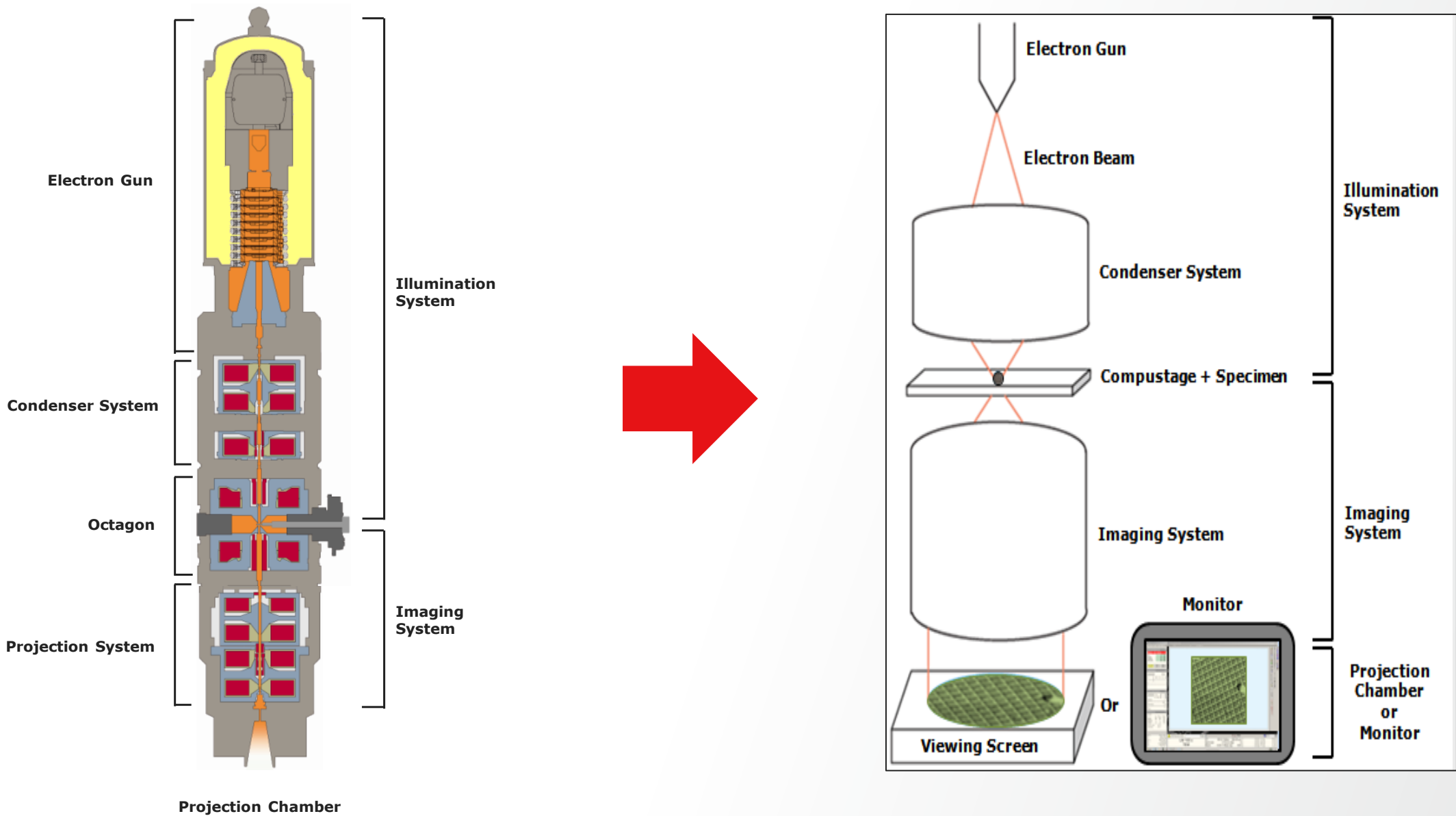
Pharma Services

Research & Safety Channel

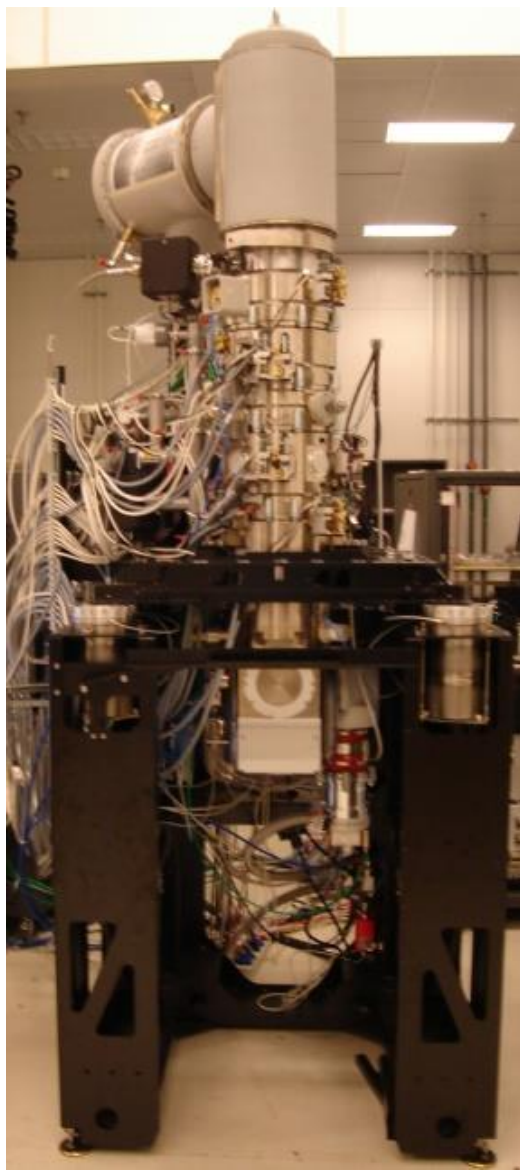
Laboratory Chemicals

Electron Microscope

Transmission Electron Microscopy (TEM)



Transmission Electron Microscopy



TEM system and vacuum

The main three vacuum volumes:

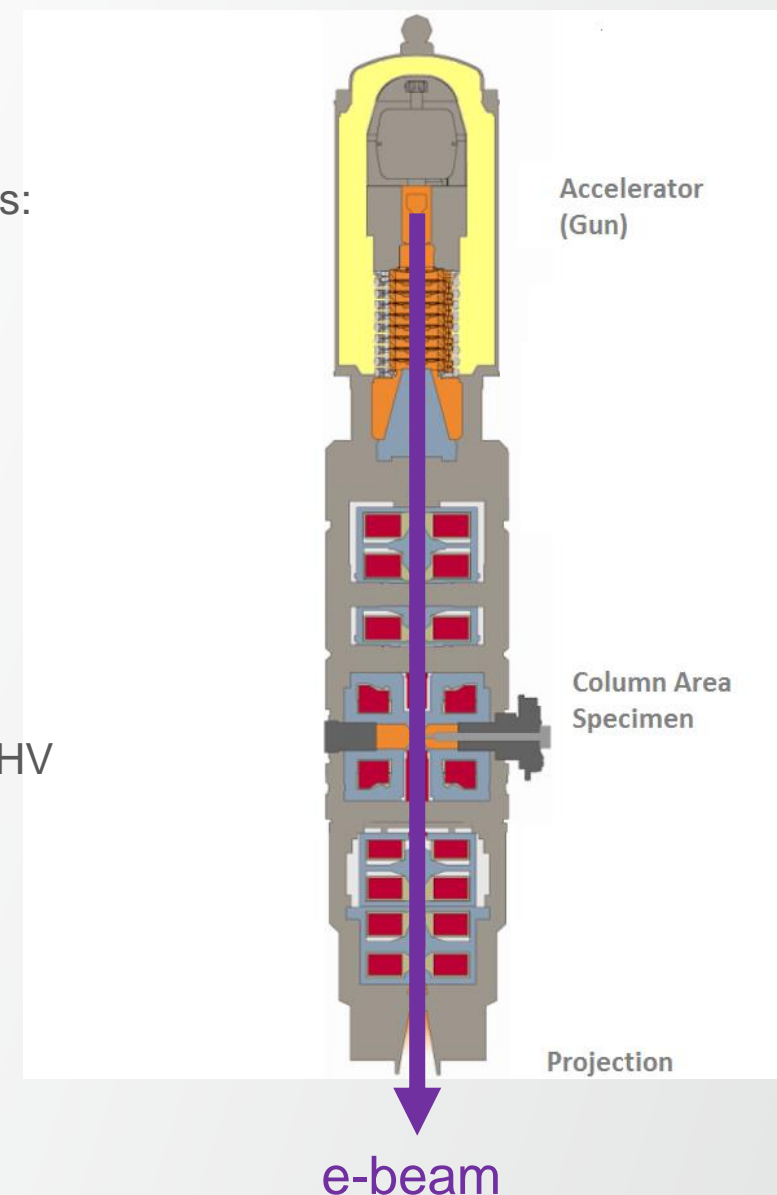
- The '**Gun**' area, around the emitter and the accelerator:
 $<1\text{E-}8\text{ Pa} \rightarrow \text{UHV}$ (new CFEG $<1\text{E-}10\text{ Pa} \rightarrow \text{XHV}$)
- The **Column area**, with beam pipes, specimen manipulator and detectors:
 $1\text{E-}4.. 1\text{E-}6\text{ Pa} \rightarrow \text{HV}$
- The **Projection** Chamber, with cameras and detectors:
 $1\text{E-}4.. 1\text{E-}5\text{ Pa} \rightarrow \text{HV}$

Why is vacuum important for TEM?

- For electron microscopy the '**mean free path**' for electrons needs to be $> 60\text{ mm} \rightarrow \text{HV}$
- To **prevent oxidation** of the electron source $\rightarrow \text{UHV/XHV}$
- For the electron generation through High Voltage (20....300 keV) $\rightarrow \text{HV/UHV}$

Trend: lower keV \rightarrow more sensitive for contaminants

Important : We need a CLEAN and DRY Vacuum!



TEM Clean and Dry Vacuum

Why is a Clean and Dry Vacuum important for TEM and what do we mean with it?

Chemical Clean

Prevent sample etching by beam interaction with

- Oxygen (O_2)
- Water (H_2O)

Prevent sample contamination (carbon growth) by beam interaction with

- Hydrocarbons (C_xH_y)

Particle Clean

Prevent particle contamination in or near the beamline to prevent

- Electron beam blocking
- High Voltage discharges
- Electron beam deflections due to electrostatic charging of particles

Surface Clean

- Prevent vacuum wall contamination (oxides) in or near the beamline to prevent electron beam deflections due to electrostatic charging
- Prevent release of top layers of surfaces

Dry Vacuum

Reduce the water content in the vacuum to prevent ice growth on Cryogenic samples (Biological samples)

‘A Vacuum is as good as the weakest link’

Not only applicable on the ultimate pressure,
but also,

to the Quality of the Vacuum

**Parts,
Material Aspects,
Manufacturing,
Contamination
and
Cleanliness
for Electron Microscopy**

Materials used in, and specification for Electron Microscopes

Material used:

- Stainless Steel (316, 304)
- Aluminum
- Titanium
- Copper
- Aluminum-Copper
- Phosphor bronze (Cu-Sn-P)
- Nickel-Iron
- Cobalt-Iron
- Graphite
- Ceramics
- Platinum
- Gold plated copper
- Lead
- Engineering Plastics
- PCB
- Elastomeric seals
-

Not used:

- Cadmium
 - Zinc
 - Brass (Copper-Zinc)
 - Grease
 - ..
- (low vapor pressure materials)

Manufacturing:

- Processing, treatment, handling and cleaning is depending on the material and shape.

Specification:

- The requirements are specified via **Technical Product Documentation** (TPD), sheet 110, 160 or 199.
- The required cleanliness is specified via the **Quality of Electron Optics (QEO)** documents:
 - **QEO-11-070 describes the required cleanliness level**
 - QEO-11-074 describes packaging of cleaned parts

It's the responsibility of the supplier to achieve the required specification

Cleanliness grades Thermo Fisher Scientific According QEO-11-070 Rev.G

- Specification of the cleanliness grade is related to the application in which the part is used (e.g. outside vacuum, inside vacuum, HV, UHV/XHV, High Voltage area,...)

- **Cleanliness Grades for suppliers:**

- **No cleaning**
- **General cleanliness** (used in clean room, not a vacuum or High Voltage part)
- **Pre-cleaning for final cleaning** (final cleaning by Thermo Fisher Scientific)

No Particles >50 µm - like dust, hair, fibers, burrs, metallic micro-fragments

No Thin Films on surfaces – organic and inorganic, like skin oil, greases, processing and cutting fluids, surfactant/chemical residues, cleaning and rinsing agents' residues, water marks, oxides, fluxes, polishing paste, chemical treatment against corrosion, silicones, other unwanted thin films on surface and in holes

- **Low Vacuum Cleaning**

Part used in vacuum environments with pressure range 1E-1 to 1E-4 Pa.

No particles >5 µm , no thin film contamination

Process evaluation procedure: RGA test cube wet processed: $C_xH_y-v < 1E-8$ and $C_xH_y-nv < 1E-9$ mbar*l/s*cm²

- **High Vacuum (HV) Cleaning**

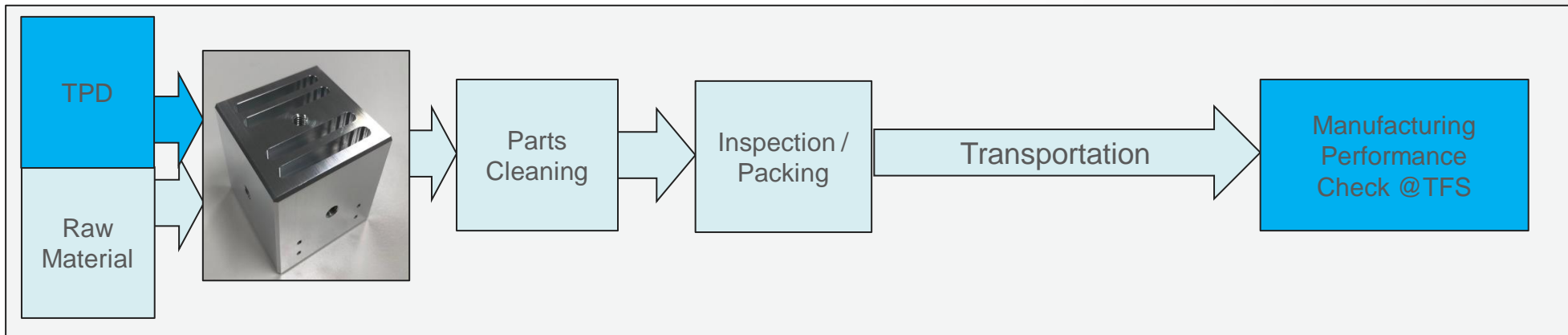
Part used in vacuum environment with pressure range 1E-4 to 1E-10 Pa, of with High Voltage up to 300 kV

- **No particles >3 µm ,**
- **No thin film contamination,**
- **Process evaluation procedure: RGA test cube “wet” processed: $C_xH_y-v < 2E-9$ and $C_xH_y-nv < 1.2E-10$ mbar*l/s*cm²**

Audit: Evaluation procedure for EM parts manufacturing according QEO-11-070

Evaluation procedure:

- Part (Test Cube or part) manufacturing & cleaning process at the supplier (material vacuum suitable Stainless Steel, Copper, Aluminum, Titanium,...)
- Supplier Cleaning Evaluation is based on so-called “WET Processing”, meaning no bake-out step!



Evaluation by Thermo Fisher Scientific (or authorized body) on

- particles >3 μm ,
- visual appearance,
- $\text{C}_x\text{H}_y\text{-v}$, $\text{C}_x\text{H}_y\text{-nv}$ values (RGA-spectrum)



(based on QEO-11-070)

Particle contamination and visual appearance

Visual inspection tools

Bright light



UVA-light



Loupe (M=9x)

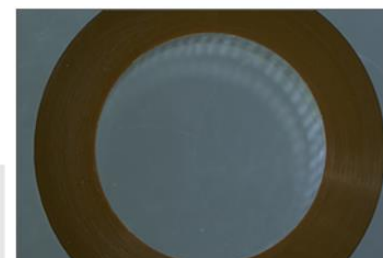


Microscope (M=40x)



Endoscope

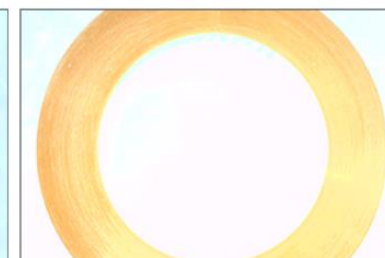
Microscope illumination



Too little light

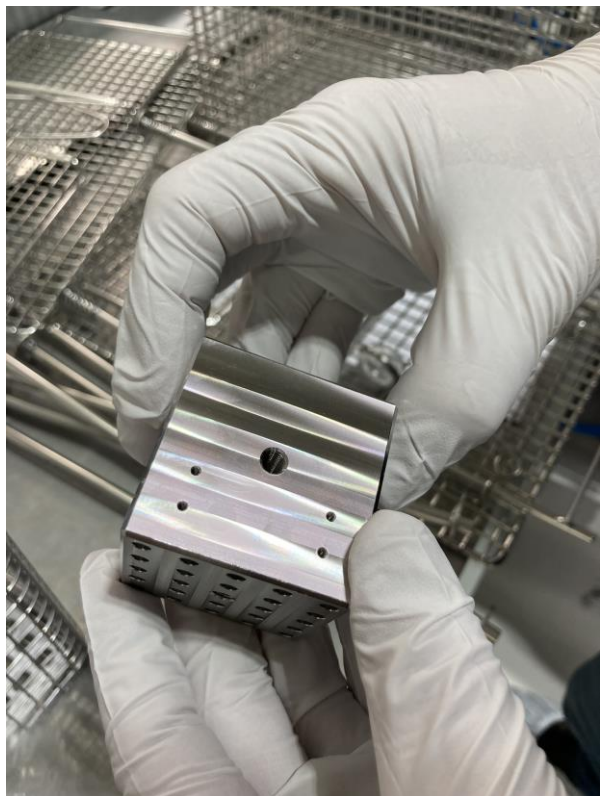


Good illumination

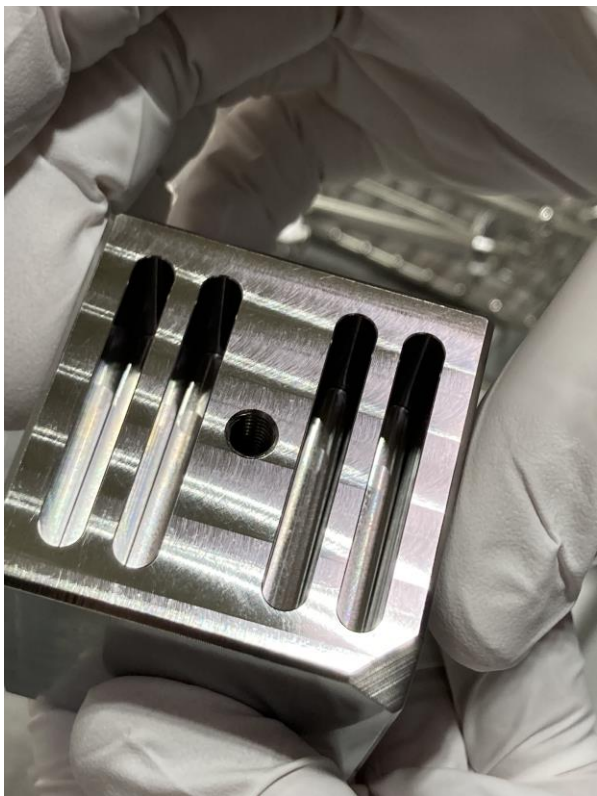


Too much light

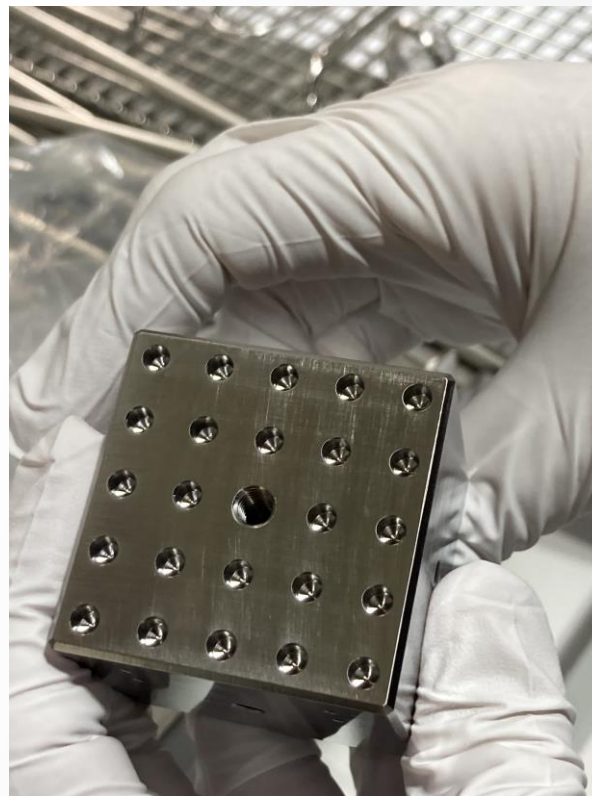
Example Test Cube visual inspection @ Thermo Fisher Scientific



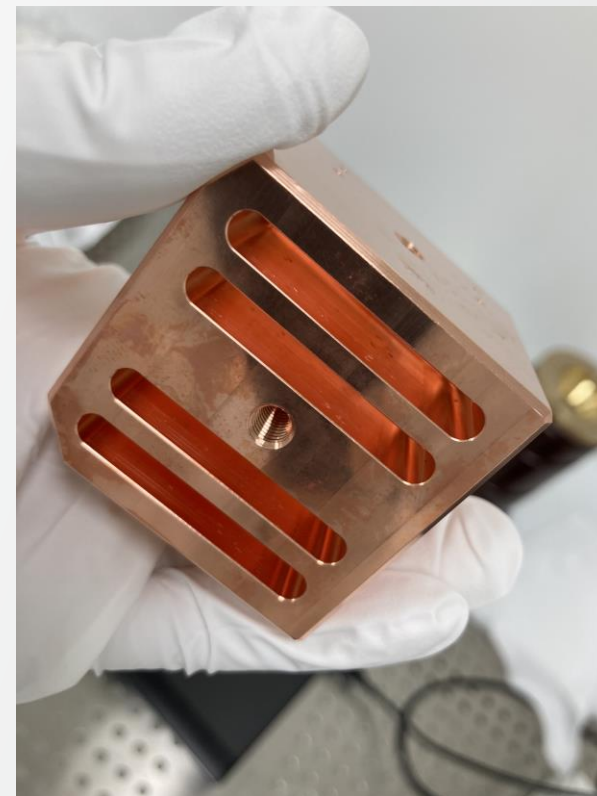
Stainless steel version



Stainless steel version

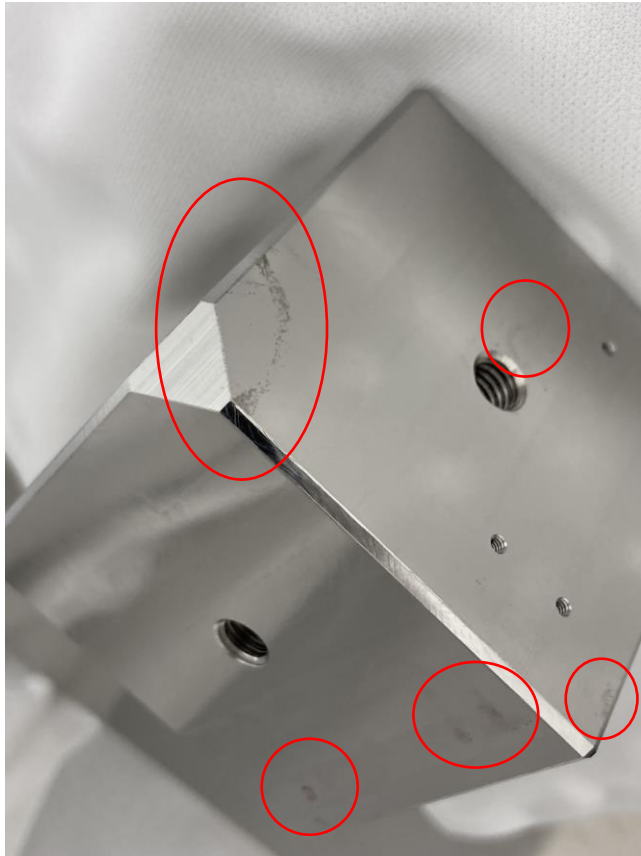


Stainless steel version



Oxygen Free High
Conductivity Copper
version

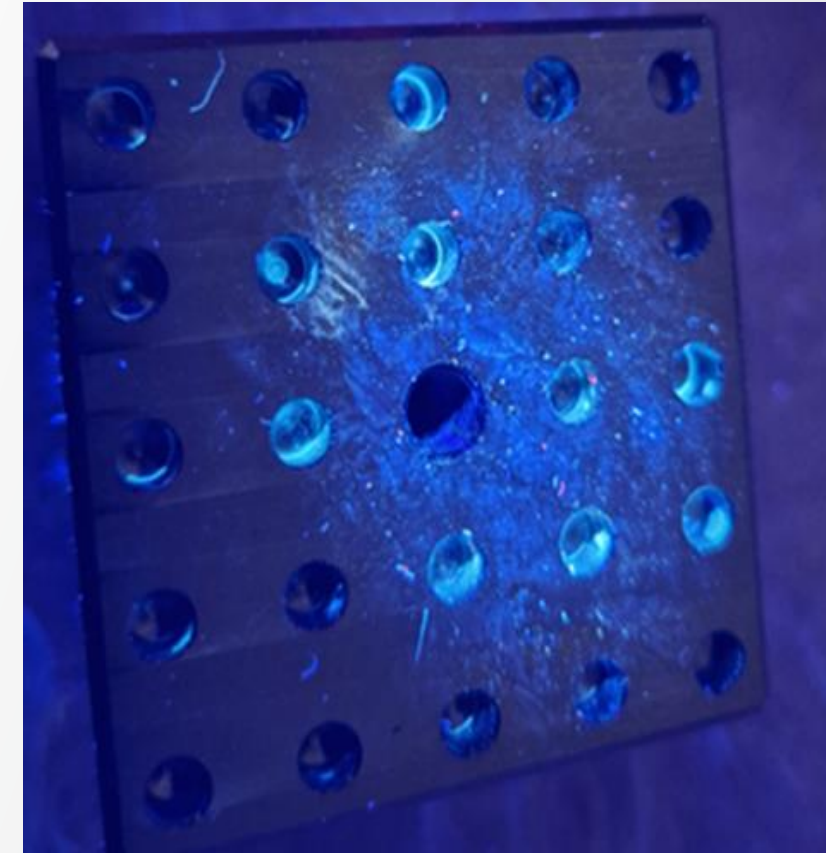
Important: work clean, use gloves.
Keep in mind: prevent cross contamination.



Stainless steel version
Stains

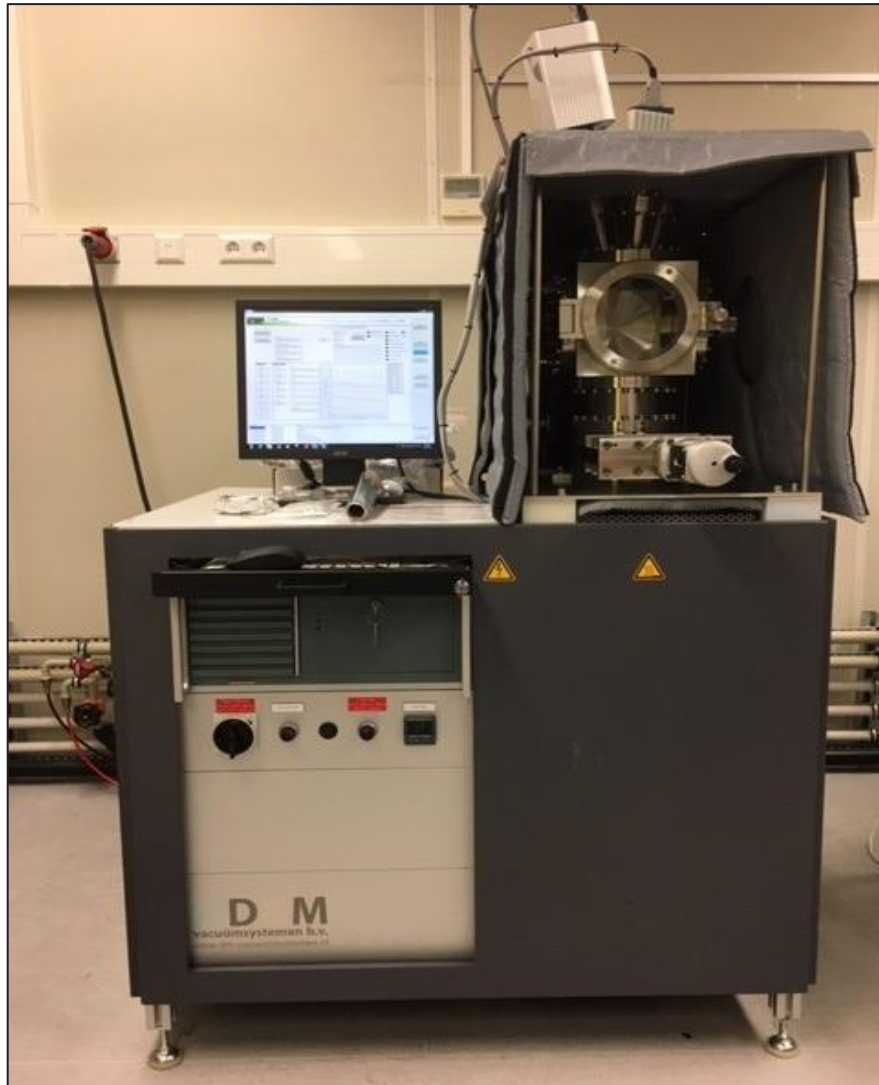


Oxygen Free High
Conductivity Copper
version
Stains

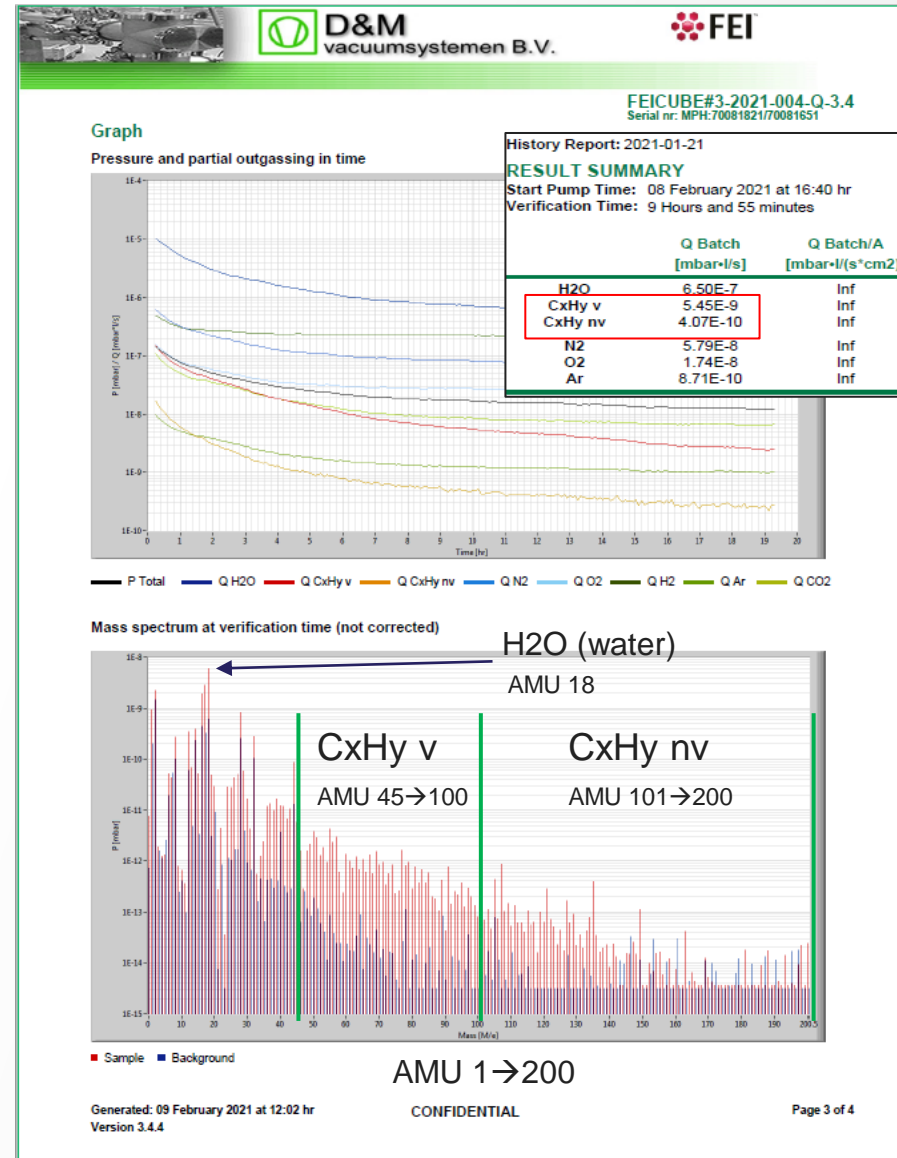


Stainless steel version
**Stains and (organic)
particles and fibers**

Molecular (chemical) contamination: D&M Cube-150 Vacuum Qualification System @



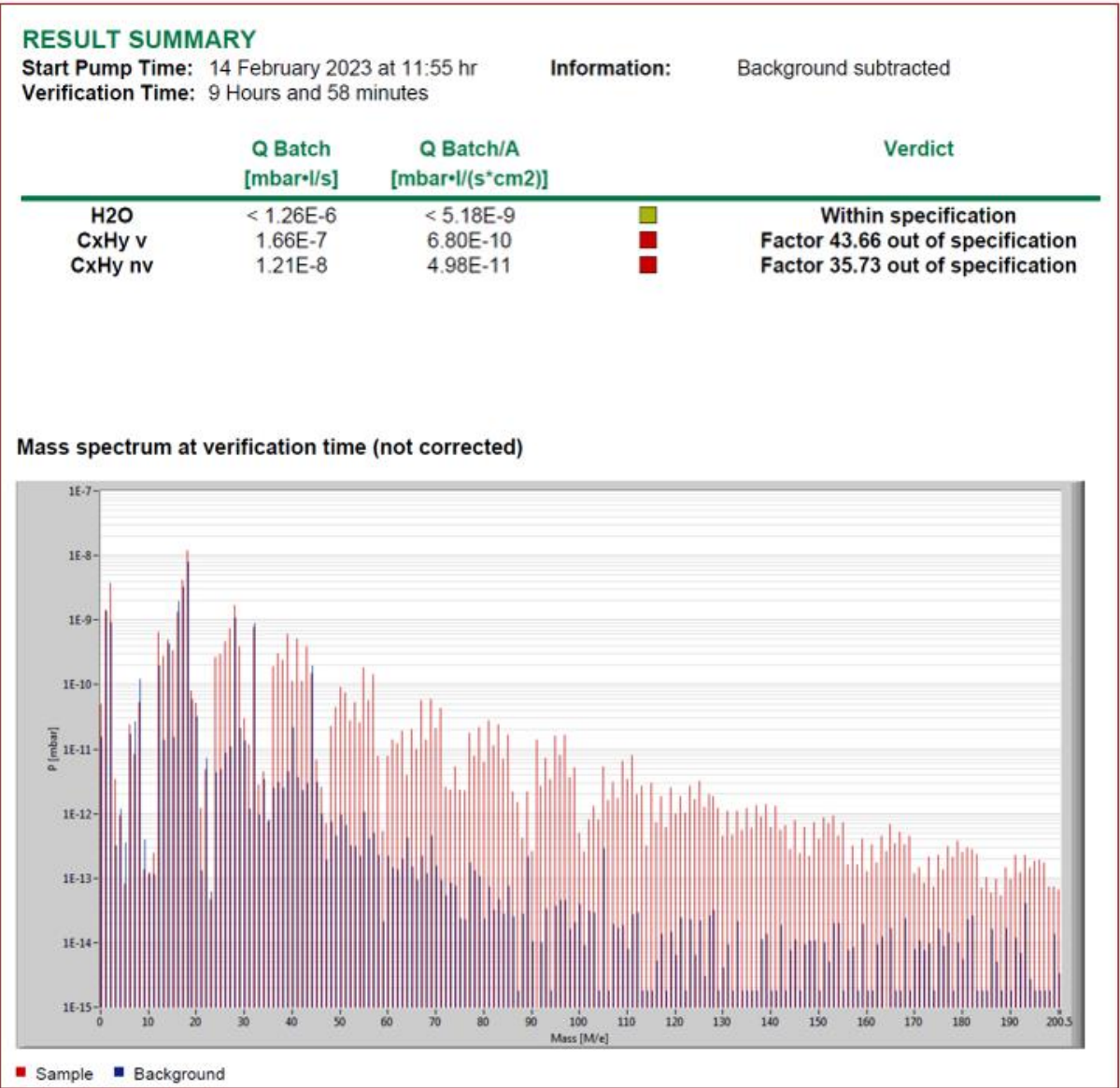
D&M Cube-150 Vacuum Qualification System



Example Test Cube chemical contamination measurement @ Thermo Fisher Scientific



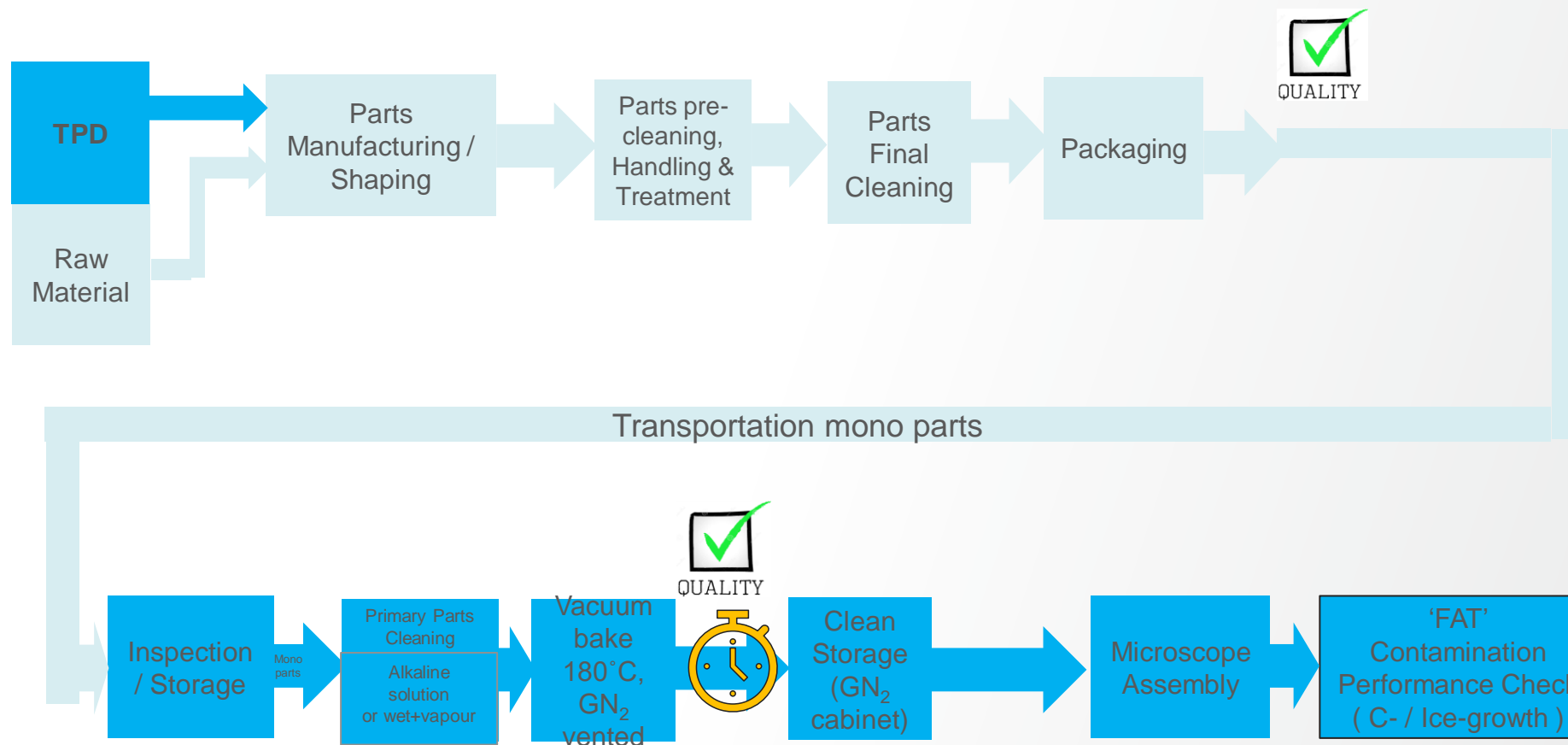
Example Test Cube chemical contamination measurement @ Thermo Fisher Scientific



40 times
out of specification

Cleanliness in the supply chain of EM Vacuum Mono Parts manufacturing

Mono



Supplier

Thermo Fisher Scientific

Max 24 h

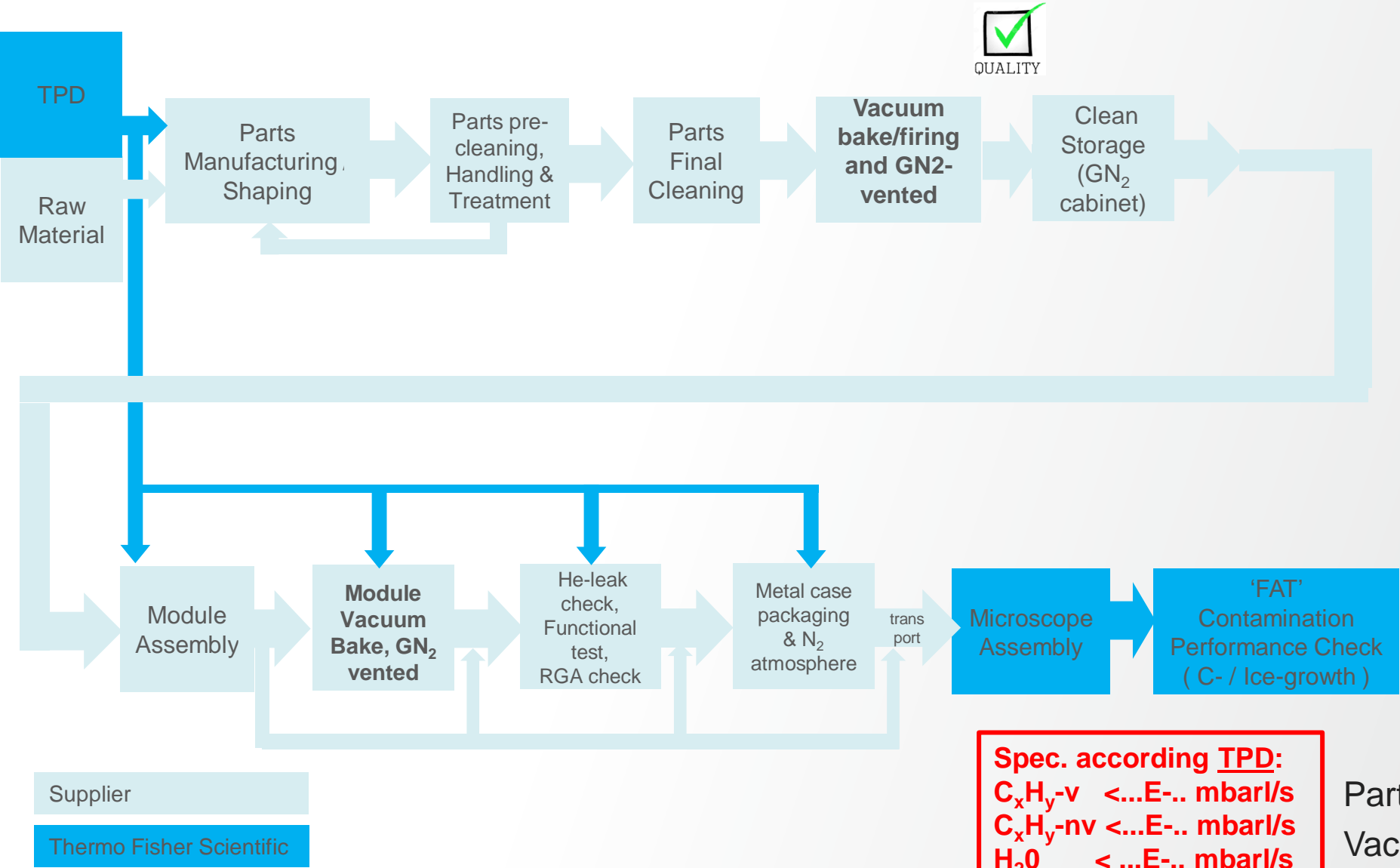
Note: UHV/UHV requires
different approach. Vacuum Firing

Spec. according TPD:
 $C_xH_y-v < \dots E\text{-}.. \text{ mbar/l/s}$
 $C_xH_y-nv < \dots E\text{-}.. \text{ mbar/l/s}$
 $H_2O < \dots E\text{-}.. \text{ mbar/l/s}$
 $H_2 < \dots E\text{-}.. \text{ mbar/l/s}$
Spectra with limits

Cleanliness in the supply chain of EM Vacuum Module/Assy manufacturing



Module/Assy



Spec. according TPD:
 $C_xH_y-v < \dots E \dots \text{ mbarl/s}$
 $C_xH_y-nv < \dots E \dots \text{ mbarl/s}$
 $H_2O < \dots E \dots \text{ mbarl/s}$
 $H_2 < \dots E \dots \text{ mbarl/s}$
Spectra with limits

Part of
Vacuum Budget
of Microscope

Preview New Cleanliness Specification for EM

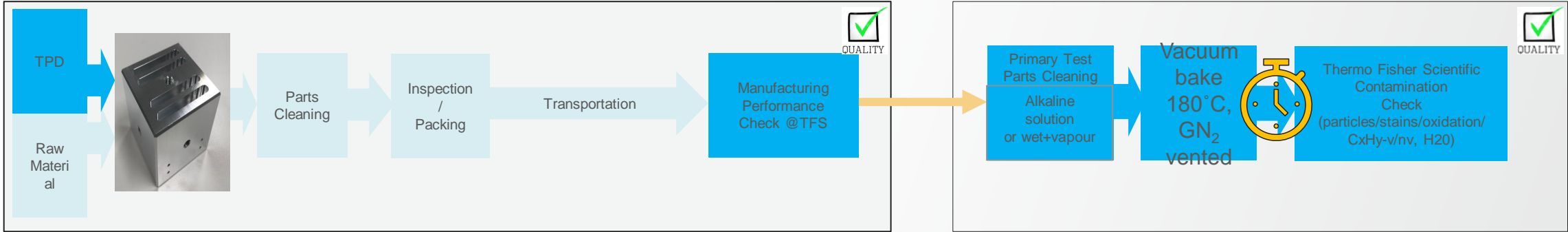
QEO-11-061

Audit: Evaluation procedure for EM parts manufacturing according QEO-11-070 or 061



Evaluation procedure:

- Part (Test Cube or part) manufacturing & cleaning process at the supplier
- Supplier cleaning evaluation is based on so-called “**WET Processing**”, meaning no bake-out step!
- Evaluation by Thermo Fisher Scientific (or authorized body) on specification
- Application cleaning evaluation based on final cleaning and vacuum bake/N₂ vented
- Application clearing evaluation for UHV/XHV that requires Vacuum Firing (removing H₂ from bulk matrix)



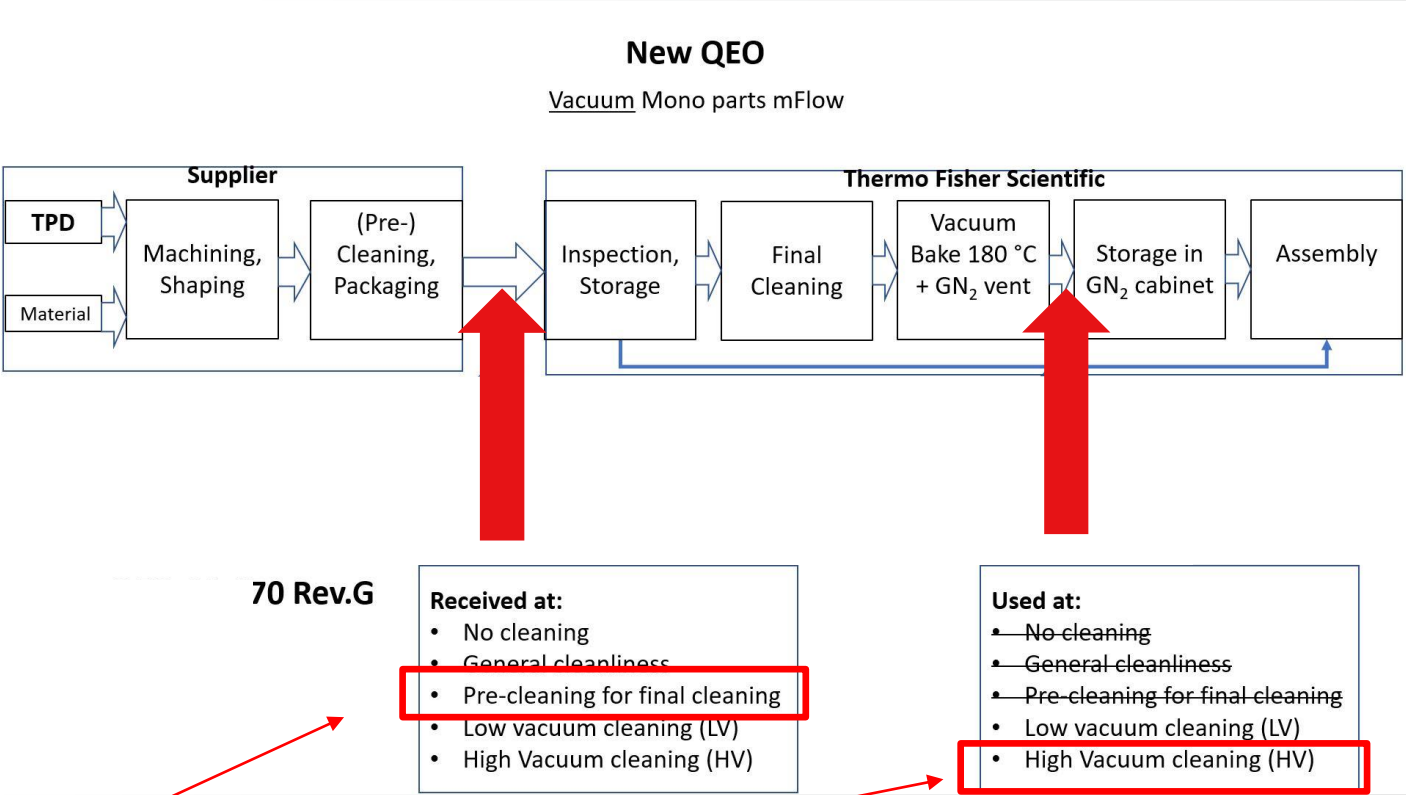
	QEO-11-070 used at HV	QEO-11-061 used at HV
Particles	> 3 µm	PCL5, PCL4, PCL3, or PCL2
Visual appearance	No stains, no decolorization	No stains, no decolorization
RGA C _x H _y -v [mbar*I/s*cm ²]	Audit: <2E-9	Audit: CCL3; HV/UHV: CCL2
RGA C _x H _y -nv [mbar*I/s*cm ²]	Audit: <1.2E-10	Audit: CCL3; HV/UHV: CCL2
RGA spectrum	-	> AMU44 : peaks 3 decades lower than H ₂ O peak
RGA H ₂ O [mbar*I/s*cm ²]	-	HV/UHV: CCL2, CCL1
RGA H ₂ [mbar*I/s*cm ²]	-	UHV: CCL1

Example Cleanliness on sheet 110 (QEO-11-070 applicable)

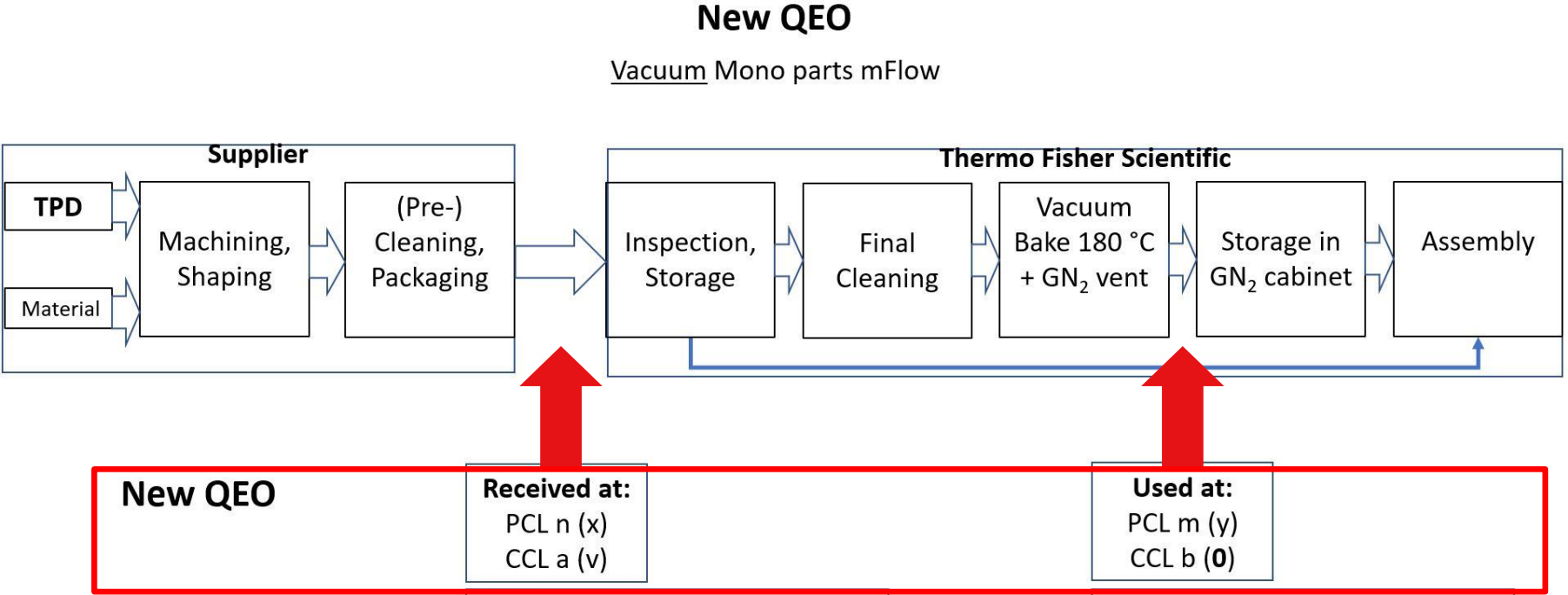


REV	DATE	ECR	DESCRIPTION OF CHANGE	NAME
1				
2				
3				
4				

THIRD ANGLE PROJECTION		ThermoFisher SCIENTIFIC		Checked and Packaged P2 acc. QEO-11-074	Welded acc. ISO 2553 and ISO 4063
				PREDECESSOR:	REVISION: B
GENERAL TOLERANCES: √ No in µm		MATERIAL: See partslist		STATUS:	
SCALE: 2:1		TREATMENT: See sheet 160-01		NAME:	
SIZE: A4		CLEANING LEVEL according to QEO-11-070: Used at: High Vac/ Received at: Pre-cleaning		SHEET BIRTH DATE:	
UNIT: mm				REVISION: C	
DRAWN ACCORDING TO: QEO-11-010		FILE TYPE: UGPART	LOCATION: ACHT	SHEET NAME: Physical data	SHEET 110-01 OF 01



New QEO-11-061 on cleanliness of EM parts and mFlow: Levels!



PCL= Particle Cleanliness Level
CCL= Chemical Cleanliness Level

Example QEO-11-070 to new QEO-11-061 on cleanlines of EM parts

REV	1	2	3	4
DATE	ECR	DESCRIPTION OF CHANGE	NAME	
a				
b				
c				

THIRD ANGLE PROJECTION	Checked and Packaged P2 acc. QEO-11-074	Welded acc. ISO 2553 and ISO 4063
GENERAL ROUGHNESS: ✓ Ra in µm	MATERIAL:	PREDECESSOR:
SCALE: 2:1	TREATMENT:	REVISION: B
SIZE: A4	CLEANING LEVEL according to QEO-11-070: Used at: High Vac/ Received at: Pre-cleaning	NAME:
UNIT: mm	TITLE:	SHEET BIRTH DATE:
FILE TYPE: UGPART	LOCATION: ACHT	SHEET NAME: Physical data
SHEET 110-01		OF 01

Used at: HV

QEO-11-070 (Rev.G)

Received at: Pre-cleaning

REV	1	2	3	4
DATE	ECR	DESCRIPTION OF CHANGE	NAME	
a				
b				
c				

THIRD ANGLE PROJECTION	Checked and Packaged P2 acc. QEO-11-074	Welded acc. ISO 2553 and ISO 4063
GENERAL ROUGHNESS: ✓ Ra in µm	MATERIAL:	PREDECESSOR:
SCALE: 2:1	TREATMENT:	REVISION: B
SIZE: A4	CLEANING LEVEL according to QEO-11-070: Used at: PCL 4 (1) / CCL 2 (0) / Received at: PCL 8 (100) / CCL 6 (0)	NAME:
UNIT: mm	TITLE:	SHEET BIRTH DATE:
FILE TYPE: UGPART	LOCATION: ACHT	SHEET NAME: Physical data
SHEET 110-01		OF 01

Used at: PCL 4 (1)
CCL 2 (0)

New QEO-11-061

Received at: PCL 8 (100)
CCL 6 (0)

Preview New Cleanliness Specification for EM

Particle Cleanliness Levels (PCL) for EM

based on
ISO 14644-9

Description	Verification Method	Measurement Targeted Particle Size	PCL						
			8	7 (ISO SCP7)	6 (ISO SCP6)	5 (ISO SCP5)	4 (ISO SCP4)	3 (ISO SCP3)	2 (ISO SCP2)
Particles/fibers	Otoscope or Endoscope (in drilling holes)	≥ 1 µm ≥ 5 µm ≥ 10 µm ≥ 50 µm ≥ 100 µm	Removable particles allowed **)	A	A	A	A (*)	A (*)	A (*)
	Bare Eye	≥ 100 µm	Removable particles allowed **)	A	A	A	A	A	A
	UV-A	≥ 50 µm	Removable particles allowed **)	A	A	A	A	A	A
	Bright Light	≥ 50 µm	Removable particles allowed **)	A	A	A	A	A	A
	Optical Stereo Microscope (magn. 40x)	≥ 10 µm	N/A	N/A	A	A	A	A	A
	Surface Particle Measur. Counter,	≥ 5 µm	N/A	N/A	N/A	A	A	A	A
	Surface Particle Measur. Counter, or Digital Microscopy w. particle measur. count software, or SEM w. particle measur. count software	≥ 1,0 µm	N/A	N/A	N/A	N/A	A	A	A
	SEM with particle measurement count software	≥ 0,1 µm	N/A	N/A	N/A	N/A	N/A	A	A
General items related to the level									
Approval manufacturing flow by Thermo Fisher Scientific	According to measurement procedures							Yes, but only on assembly level	Yes, but only on assembly level

A=Applicable, N/A=Not applicable

← Levels!
In Sync with ISO,
except Level 8
(Removable particles allowed)

Preview New Cleanliness Specification for EM

Chemical Cleanliness Level (CCL) for EM

(Not according ISO 😞)

Description	Verification Method	Targeted size	CCL					
			6	5	4 (audit only)	3 (audit only)	2	1
Stains/Discolorations (coated and uncoated materials)	UV-A (Measurement reference 2)	Small as identifiable	Allowed if removable	0	0	0	0	0
	Bright light (Measurement reference 3)	Small as identifiable	0	0	0	0	0	0
liquids on surfaces	UV-A (Measurement reference 2) Bright light (Measurement reference 3)	Small as identifiable	0	0	0	0	0	0
liquids on <u>surfaces</u> (drilling and thread holes)	Endoscope Otoscope Light microscopy (magn. X) (Measurement reference 4)	Small as identifiable	0	0	0	0	0	0
Chemical contamination (outgassing) For MONOpart	RGA (Measurement reference 7)	C _x H _y (volatile)	N/A	N/A	1,0·10 ⁻⁹	2,0·10 ⁻⁹	2,0·10 ⁻¹⁰	2,0·10 ⁻¹¹
		C _x H _y (non-volatile)	N/A	N/A	1,0·10 ⁻⁹	1,2·10 ⁻¹⁰	1,5·10 ⁻¹¹	1,5·10 ⁻¹¹
		H ₂ O	N/A	N/A	[mbar·l/s·cm ² ·10h] Low Vac No Bakeout	[mbar·l/s·cm ² ·10h] High Vac No Bakeout	[mbar·l/s·cm ² ·10h] with Bakeout see ref.5	[mbar·l/s·cm ² ·10h] with Bakeout see ref.5
Chemical contamination (outgassing) For Assembly	RGA (Measurement reference 7)	C _x H _y (volatile)	N/A	N/A	N/A	N/A	N/A	x
		C _x H _y (non-volatile)	N/A	N/A				y
		H ₂ O	N/A	N/A				z [mbar·l/s @ time] with Bakeout see TPD-160
Chemical contamination (<u>disturbing</u> elements)	XPS on samples (not on product) from process qualification by TF (Measurement reference 8)	Elements, atomic level	N/A	Avoid elements Zn, Si and Cu on surface verification not obligated	Avoid elements Zn, Si and Cu on surface verification not obligated	Avoid elements Zn, Si and Cu on surface verification not obligated	Avoid elements Zn, Si and Cu on surface verification not obligated	Elements and maximum levels described in TPD 160
Chemical contamination (hydrogen content)	Measured on samples at the process qualification by TF (Measurement reference 9)	H ₂ -Gas content	N/A	N/A	N/A	N/A	N/A	Maximum Value [H ₂ gr/m ²] in TPD 160

← Level 6 1

Verification:

Visual inspection with bare eye, microscope, Bright light, UV-A, Otoscope light in holes.

- 0 means not visible,
- Exception Level 6 – UV-A light - stains and discolouration allowed if removable

Verification: **Outgassing measurement.**

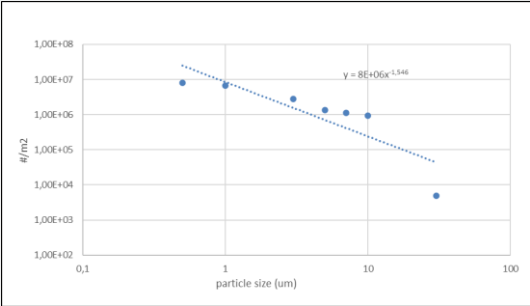
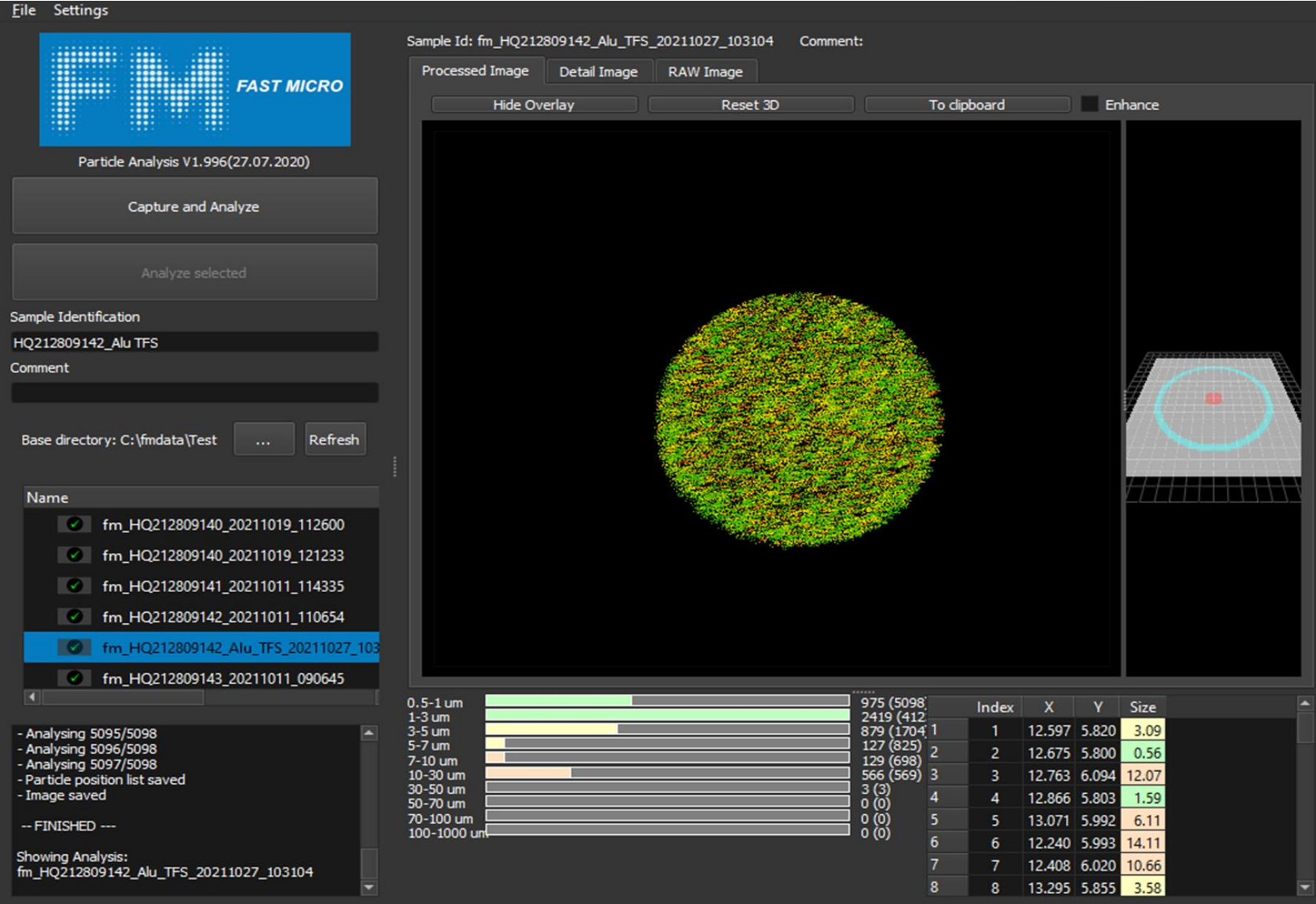
- Level 3 and 4: audit, wet processing
- C_xH_y [mbar·l/s *cm²] and ISO 14644-10 [ng/m²] do not match
Measurement methods for EM cleanliness in ISO 14644-10 not sensitive enough

Verification on unwanted elements, e.g. Zn,Si, via SEM-EDS, XPS,.. (future)

Verification outgassing UHV/XHV after vacuum firing, H₂ outgassing mbar·l/s *cm²

Examples particle measurement methods

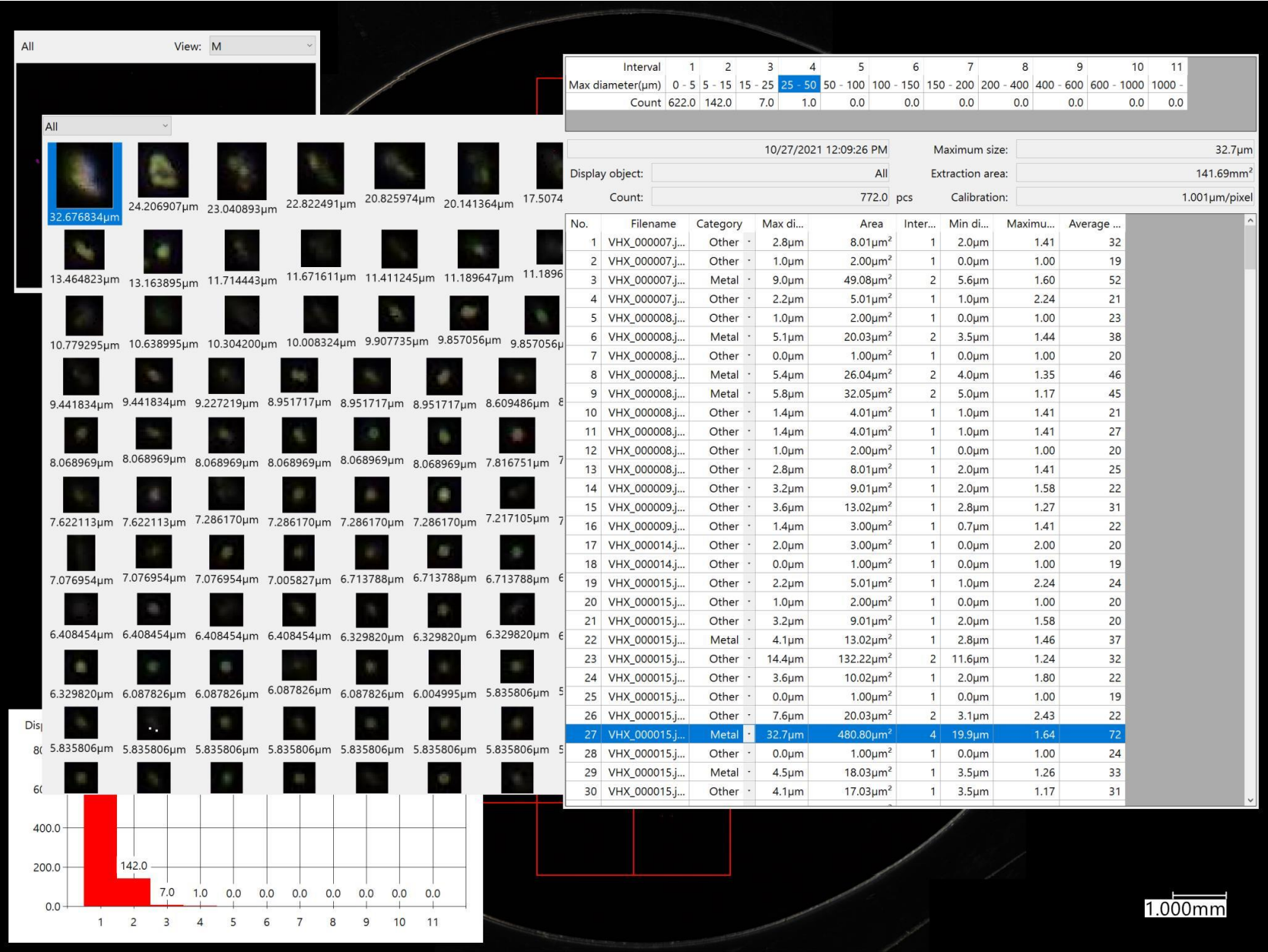
Example particle contamination measurement Fastmicro



Particle measurement on PMC 2.0
Test object cube Aluminium

Note: Quick

Example particle contamination measurement Keyence VHX-7000



Particle measurement on PMC 2.0
Test object cube Aluminium

Note:

- Contactless
- Particles, fibers & metal particles individual visible
- Less quick than Fastmicro

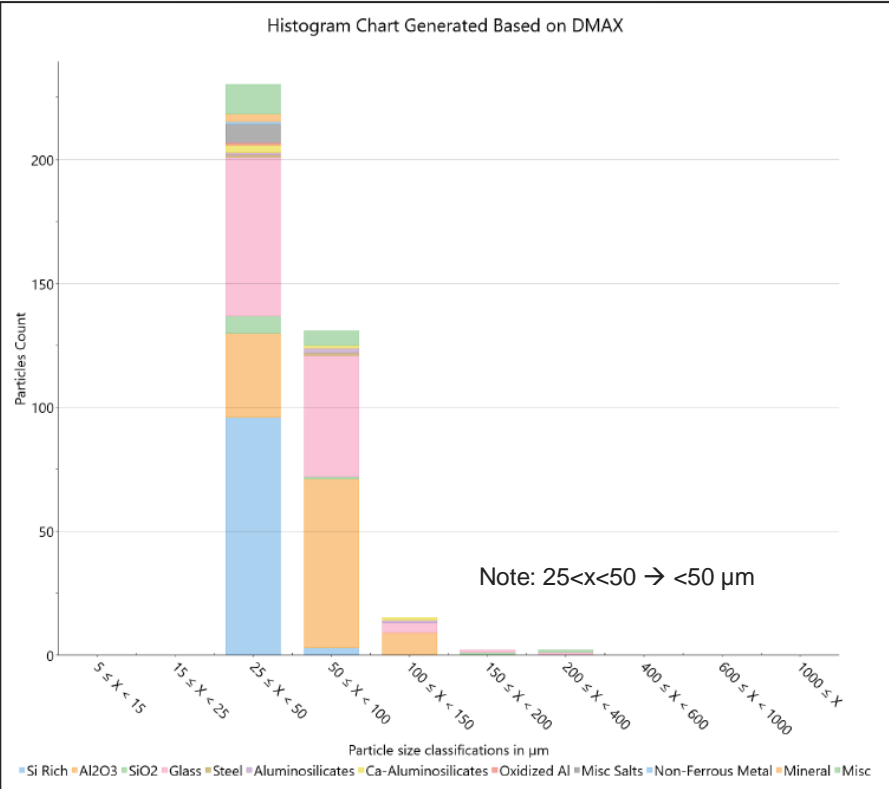
Example particle contamination measurement Phenom SEM



Individual Particle Parameters

Stage Map													
Rank	ID	Size (µm)	Width (µm)	Class	X	Y	Aspect	Area	Roundness	Form Factor	EdgeRoughness	ECD	O
1	2	25.432	21.773	Misc	-9.171	41.684	1.177	333.983	0.657	0.590	0.330	20.621	64.600
2	3	72.569	49.169	Glass	-8.222	42.006	1.476	2194.584	0.525	0.478	0.049	52.740	67.000
3	4	89.001	39.243	Al2O3	-7.737	41.864	2.268	2615.990	0.420	0.536	0.612	87.708	73.600
4	5	94.022	56.146	Al2O3	-8.276	41.772	1.675	3623.126	0.522	0.628	0.870	67.920	67.100
5	6	33.184	14.047	Al2O3	-7.994	41.770	2.450	231.818	0.268	0.323	0.661	17.180	68.800
6	7	47.773	8.771	Al2O3	-8.249	41.615	5.711	255.463	0.143	0.244	2.103	18.035	68.200
7	8	95.939	67.034	Al2O3	-8.103	41.559	1.431	4743.889	0.656	0.719	0.378	77.718	65.200

Table with hardness groups and histogram											
Sample Information			Sample Prep Information			Run Information					
Company:			Component Surface By:		Volume	Magnification:		220x			
Operator Name:			Volume of Extraction (cm³)		100	Number of Stage Fields:		61			
Part # / Sample ID:			Projected Volume (cm³)		100	Area Scanned (mm²):		36.42			
Analysis Date:		3/29/2021 1:30:40 PM				Run ID's Present:		Run_1			
Particle Results											
Size Class Based On: DMAX		B	C	D	E	F	G	H	I	J	K
Size Range (µm)	Total	5 ≤ X < 15	15 ≤ X < 25	25 ≤ X < 50	50 ≤ X < 100	100 ≤ X < 150	150 ≤ X < 200	200 ≤ X < 400	400 ≤ X < 600	600 ≤ X < 1000	1000 ≤ X
Si Rich	99			96	3						
Al2O3	111			34	68	9					
SiO2	9			7	1		1				
Glass	119			64	49	4	1	1			
Steel	2			1	1						
Aluminosilicates	4			1	2	1					
Ca-Aluminosilicates	5			3	1	1					
Oxidized Al	1			1							
Misc Salts	7			7							
Non-Ferrous Metal	1			1							
Mineral	3			3							
Misc	19			12	6			1			
Total Counts	380	0	0	230	131	15	2	2	0	0	0
Notes: This chart lists particles based on hardness value. To create it, the range of hardness (in HV) was added as a property to the classes and the range of hardness was defined for the hard, medium and soft particles.											
Class Name	Color	Sub Total									
Hard		338									
Medium		11									
Soft		31									

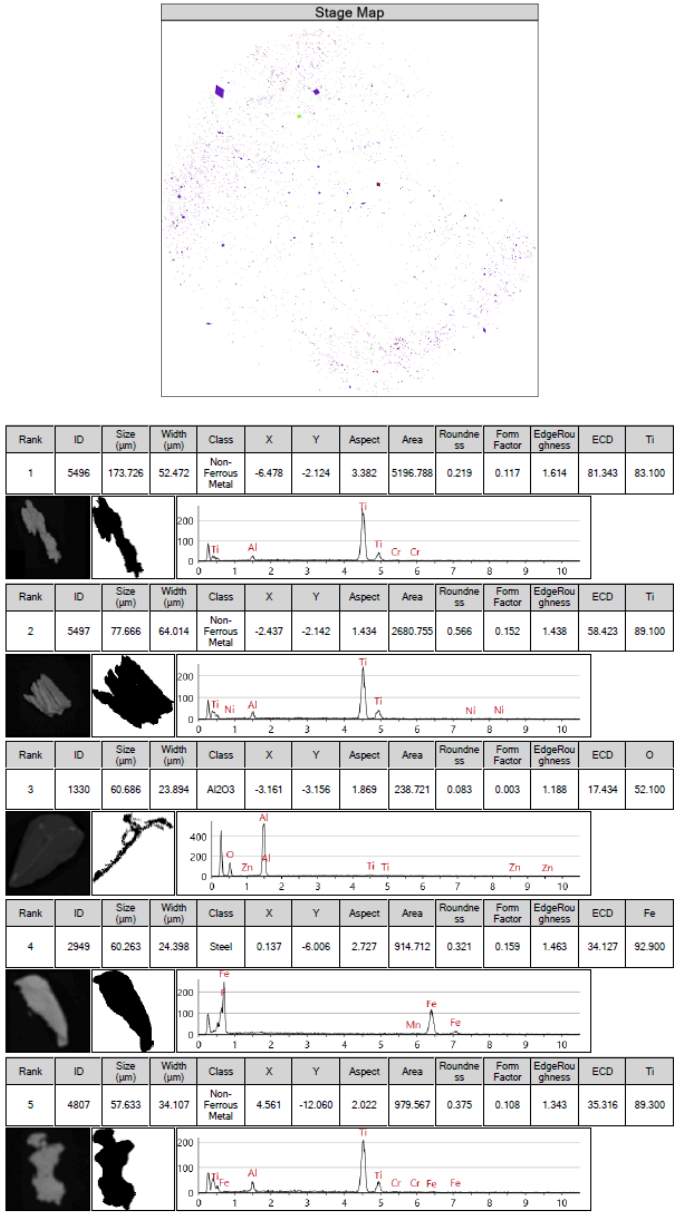


Particle measurement on PMC 2.0
Test object: Test on table surface
Measurement: Phenom ParticleX TC Desktop SEM

- Note:
- Vacuum
 - Particles, fibers & metal particles individual visible
 - Particle size & chemical composition discovered
 - Less quick than Fastmicro

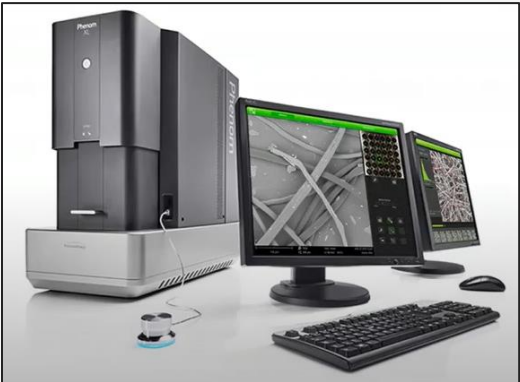
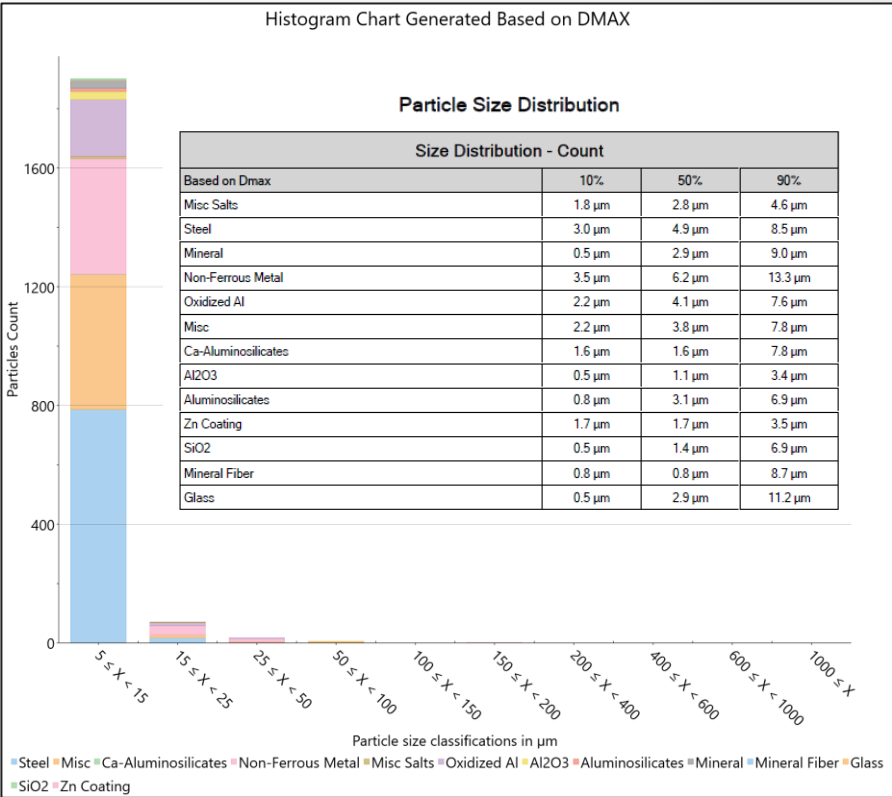


Example particle contamination measurement Phenom SEM



Sample Information		Sample Prep Information		Run Information	
Company:		Component Surface By:	Volume	Magnification: 1000x	
Operator Name:		Volume of Extraction(cm³)	100	Number of Stage Fields: 6789	
Part # / Sample ID:		Projected Volume(cm³)	100	Area Scanned(mm²): 196.18	
Analysis Date: 3/6/2023 4:26:31 PM		Filter Size (mm):	300	RunID's Present: Run_1	

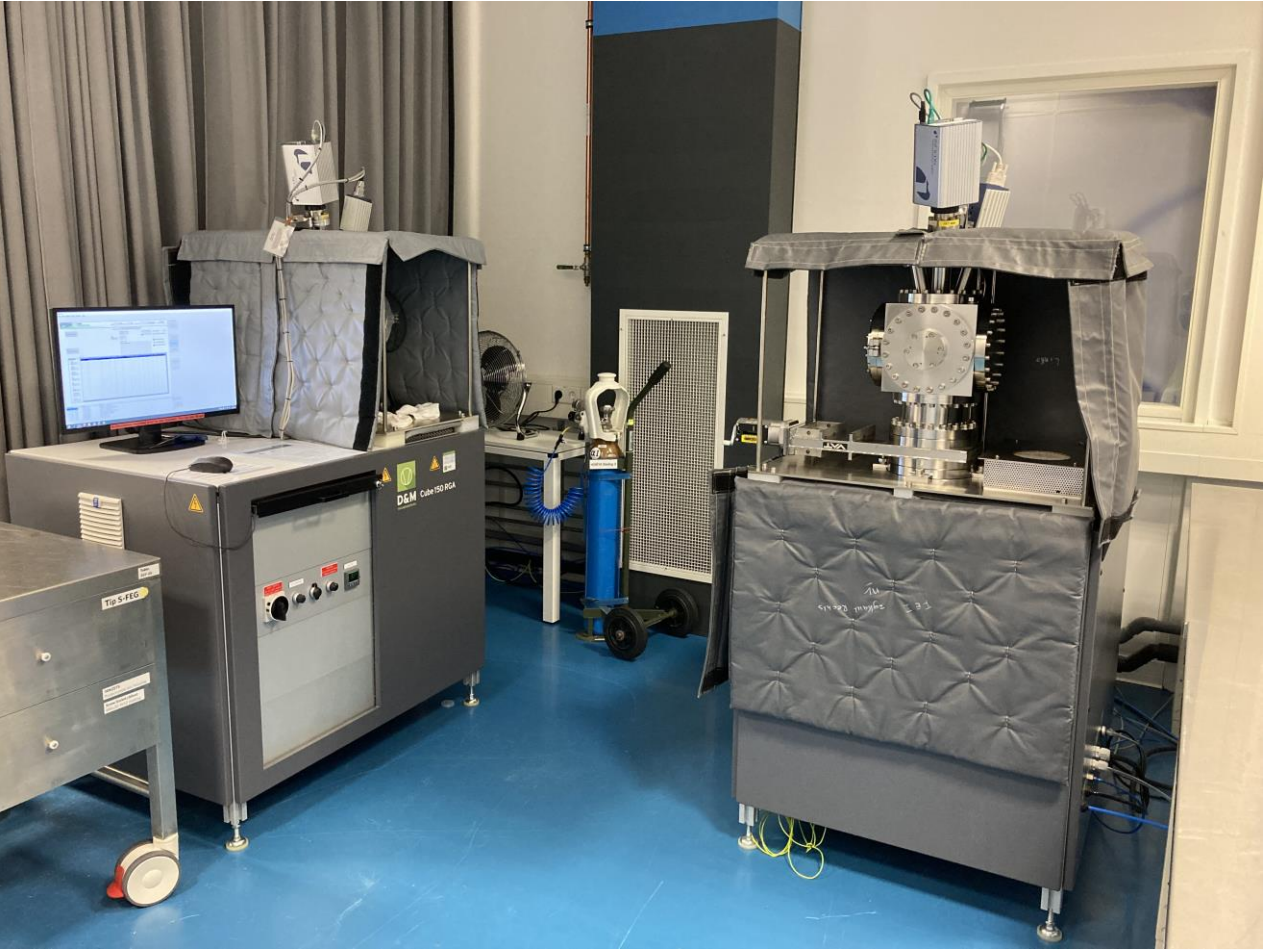
Particle Results											
Size Class		B	C	D	E	F	G	H	I	J	K
Size Range (µm)	Total	5 ≤ X < 15	15 ≤ X < 25	25 ≤ X < 50	50 ≤ X < 100	100 ≤ X < 150	150 ≤ X < 200	200 ≤ X < 400	400 ≤ X < 600	600 ≤ X < 1000	1000 ≤ X
Steel	807	787	18	1	1						
Misc	468	454	9	3	2						
Ca-Aluminosilicates	1	1									
Non-Ferrous Metal	435	390	31	11	2		1				
Misc Salts	8	8									
Oxidized Al	203	192	9	2							
Al2O3	28	26	1		1						
Aluminosilicates	12	11	1								
Mineral	26	25	1								
Mineral Fiber	1	1									
Glass	2	2									
SiO2	4	4									
Zn Coating	0										
Total Counts	1995	1901	70	17	6	0	1	0	0	0	0
Notes:											



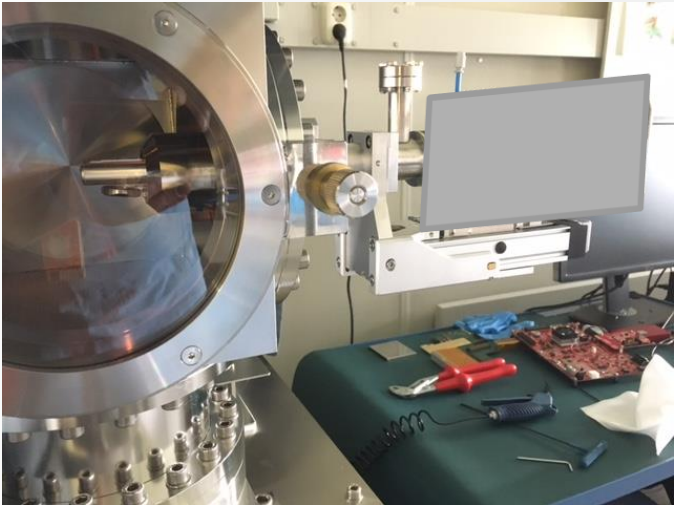
Particle measurement on PMC 2.0
Test object: Test cube (PMC_1sample_Rev2.rafa
3/6/2023)
Measurement: Phenom ParticleX TC Desktop SEM

- Note:
- Vacuum
 - Particles, fibers & metal particles individual visible
 - Particle size & chemical composition discovered
 - Less quick than Fastmicro

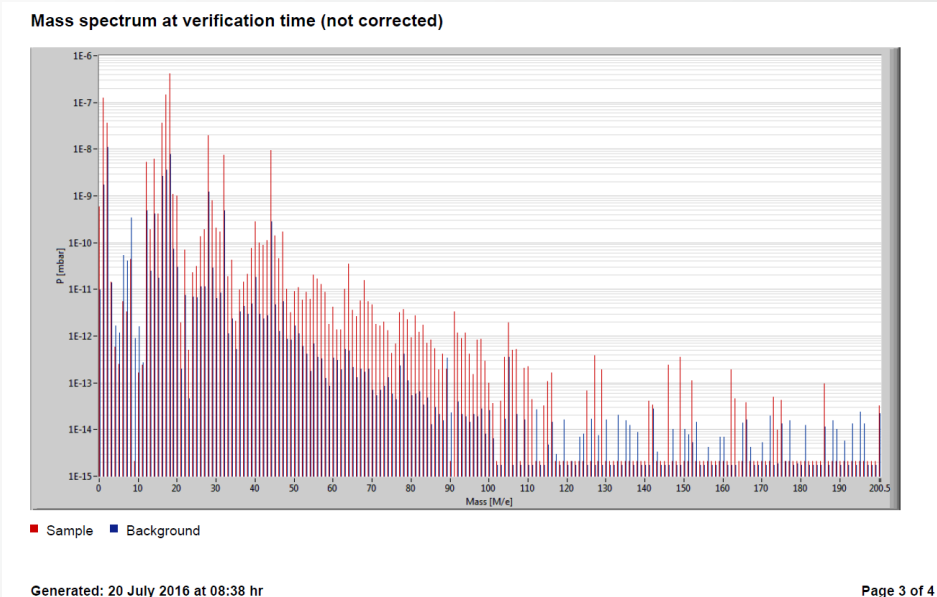
Example RGA measurement @ Thermo Fisher Scientific



Thermo Fisher Clean Lab with 2 D&M RGA-tools



Example Module measurement at D&M RGA-tool



Example RGA- spectrum measurement result: not clean.

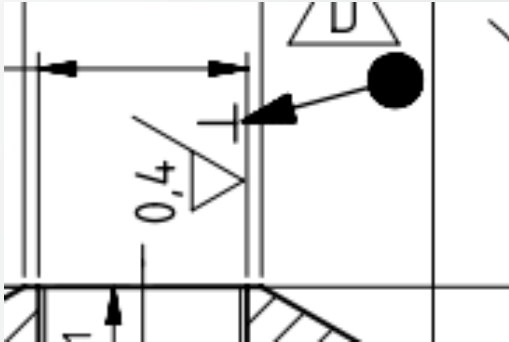
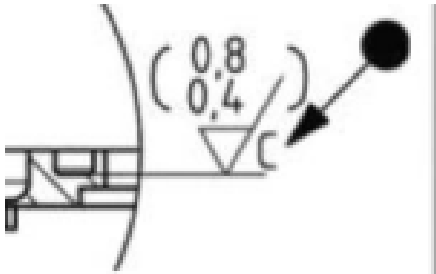
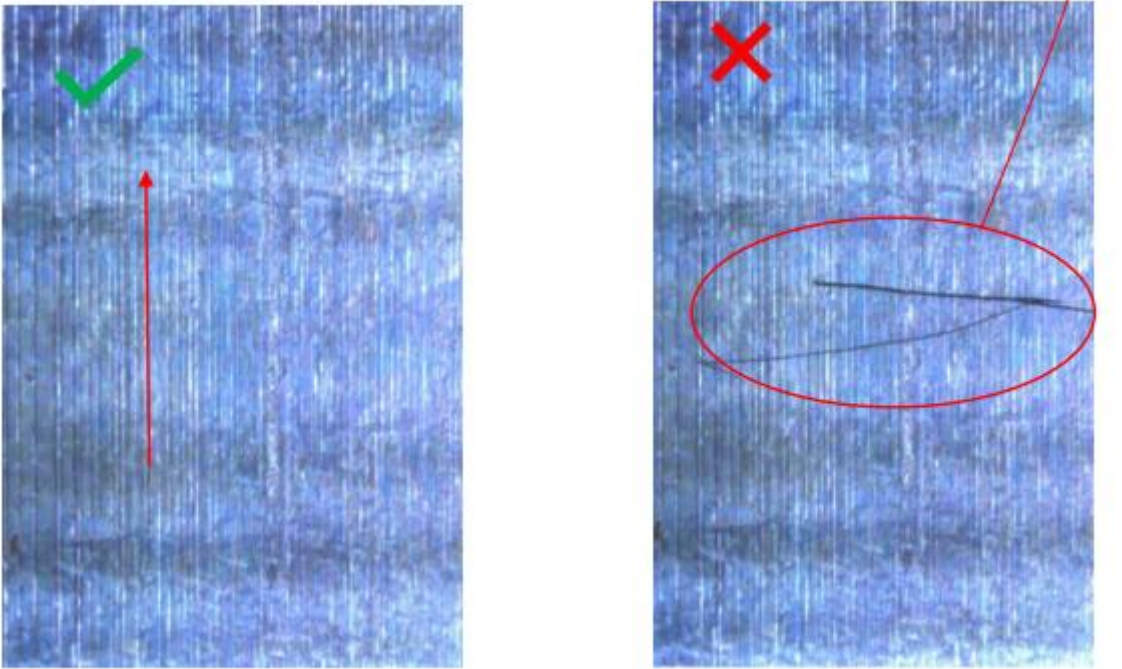
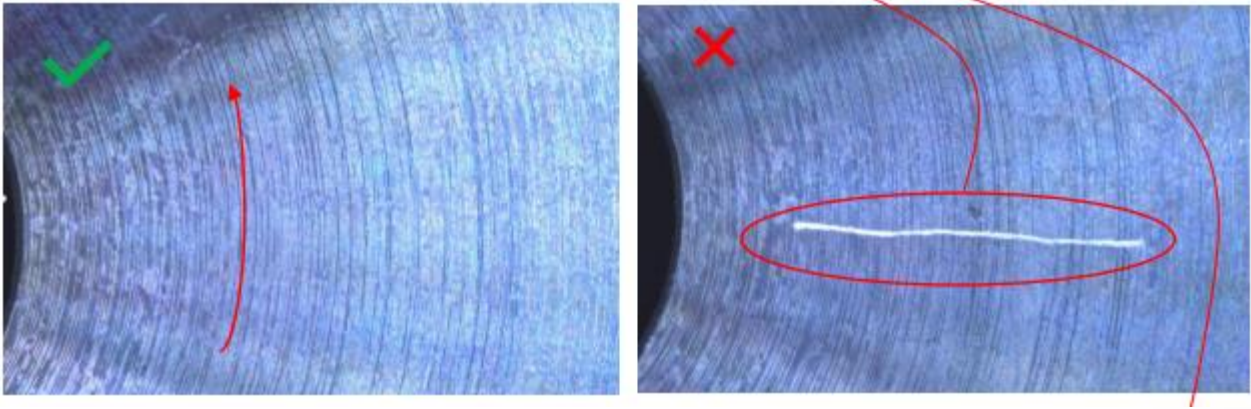


Outgassing:
D&M Cube150 RGA-tools

Particle size/analysis:
Phenom XL G2 Desktop
SEM

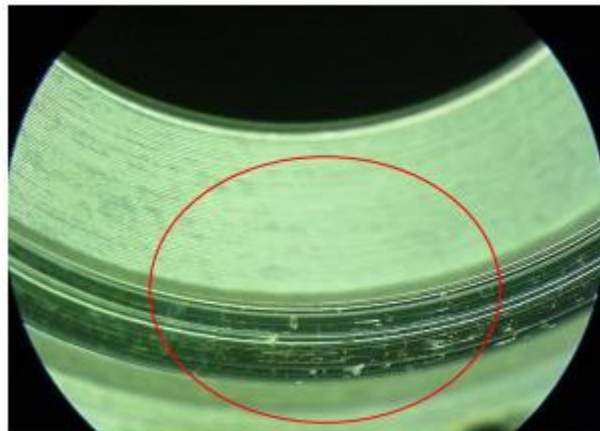


Scratches on vacuum surfaces

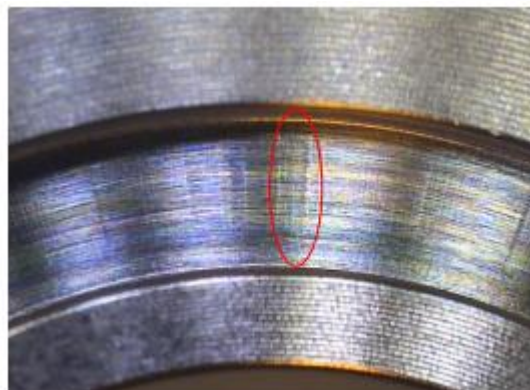


Other problems with vacuum surfaces

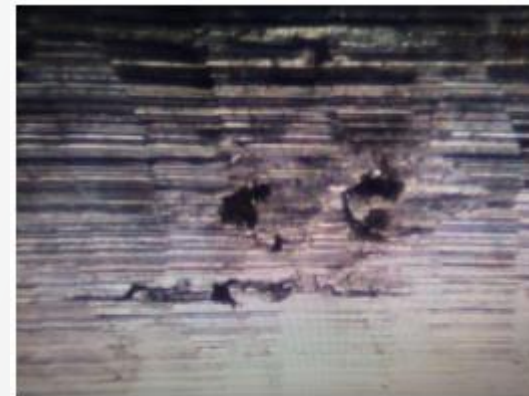
Roughness, flatness



Vibration of tooling



Material imperfections



Example contamination

Incomming inspection of
Stainless Steel part.

Problem: not HV clean,
not ready for final cleaning

Particles and dirt from holes,
removed with cleaning stick

Visual inspection by: bare eye



Example contamination

Incomming inspection of
Stainless Steel part.

Problem: not HV clean,
not ready for final cleaning

Particles from thread holes,
removed with cleaning stick

Visual inspection by: bare eye



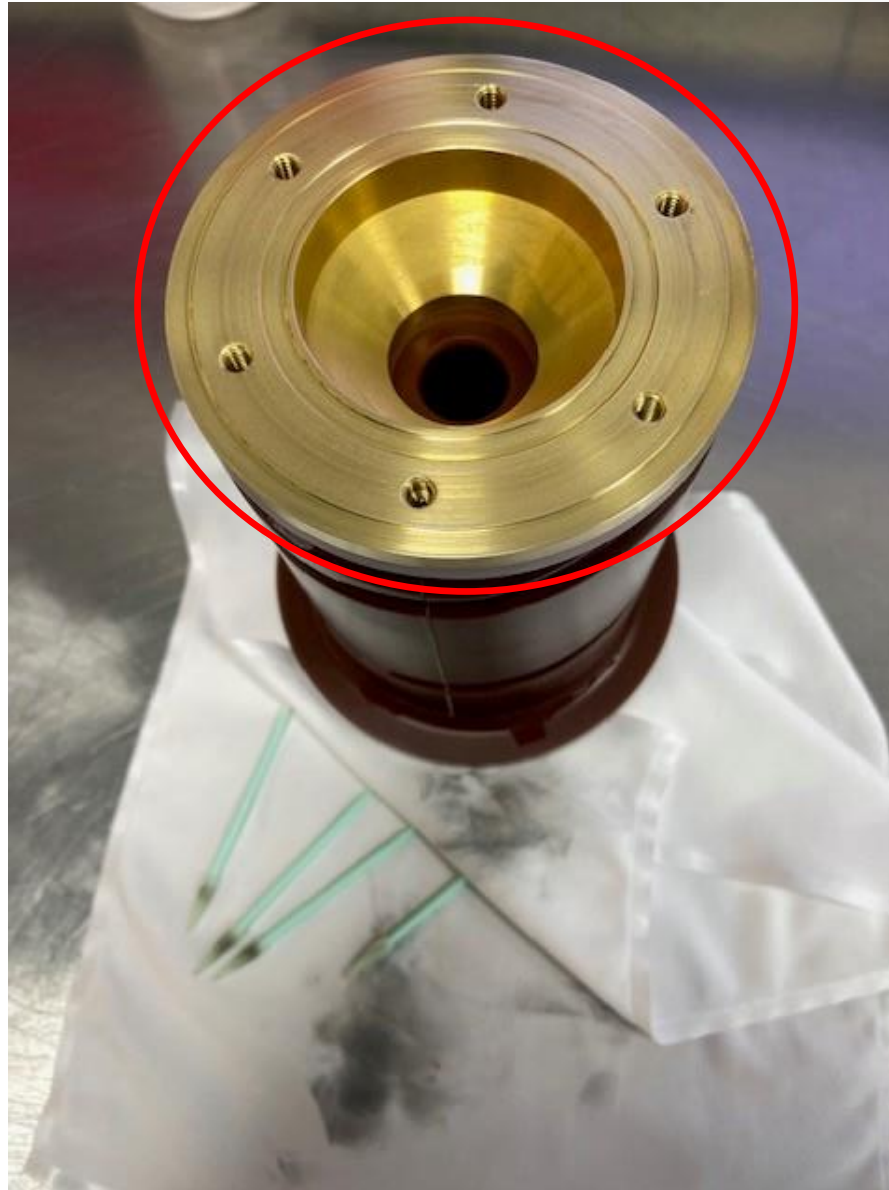
Example contamination

Incomming inspection of
part.

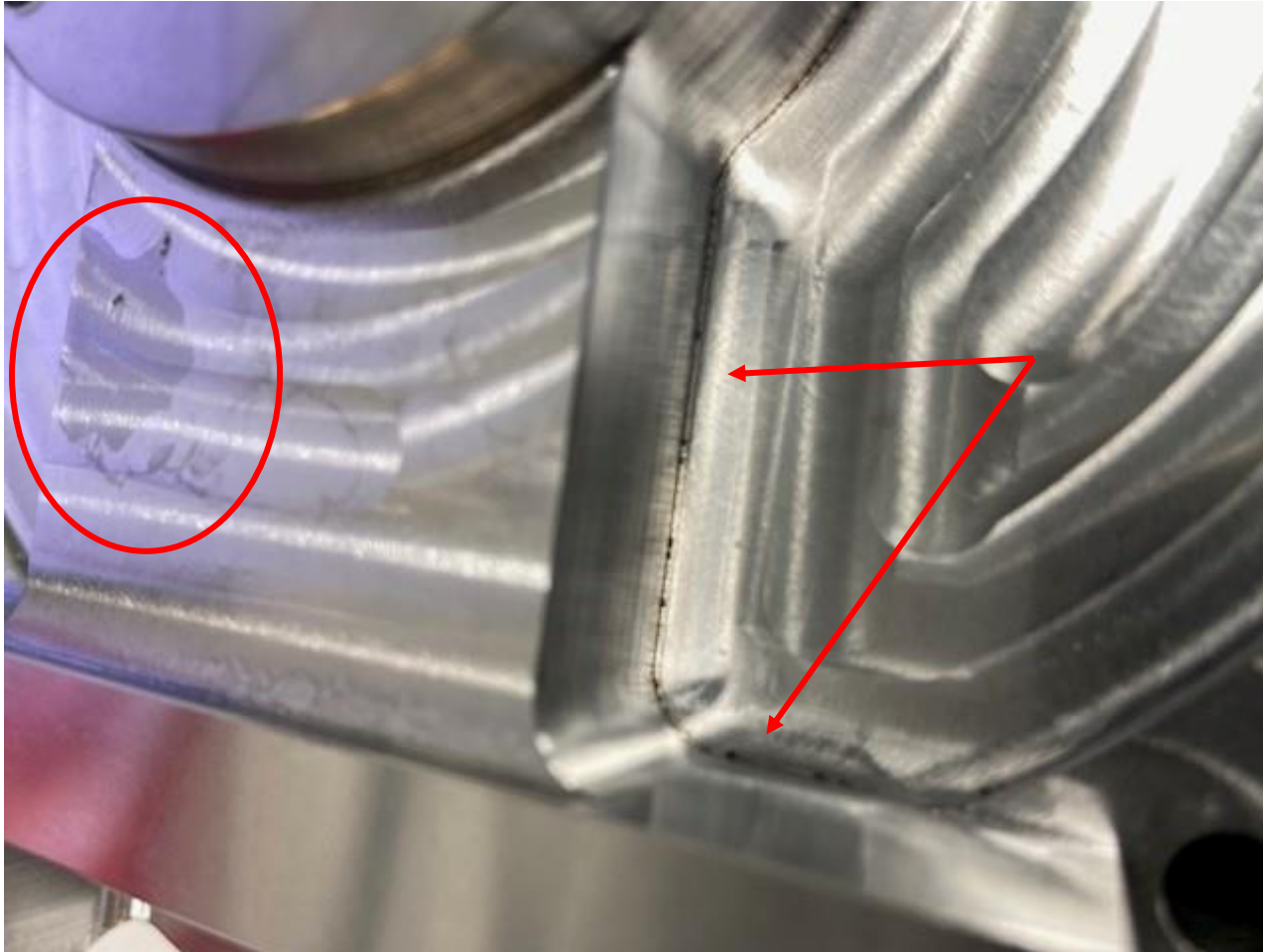
Problem: not HV clean,
not ready for final cleaning

Wipe and cleaning stick
cleaning

Visual inspection by: bare eye

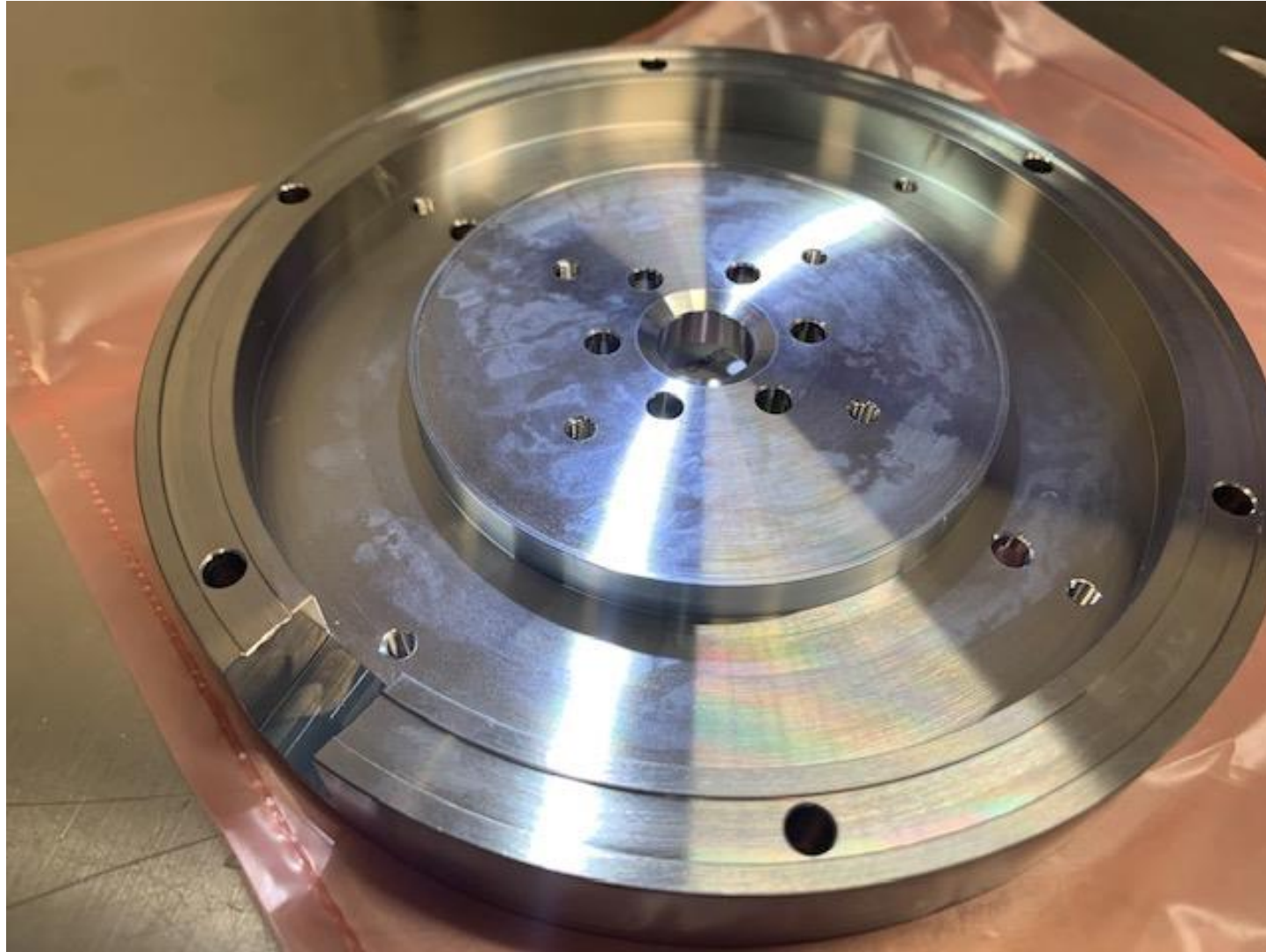


Example contamination



Incomming inspection of
Stainless Steel part.
Local corrosion, particles and stains
Visual inspection by:
bare eye, UVA-light

Example stains on part for UHV application



Remainings from foam blocker added to cutting fluid, e.g. by drying in between processing steps.

White stains
Bare eye observation
Made visible with Bright light

Example Discolourization, oxidation and stains

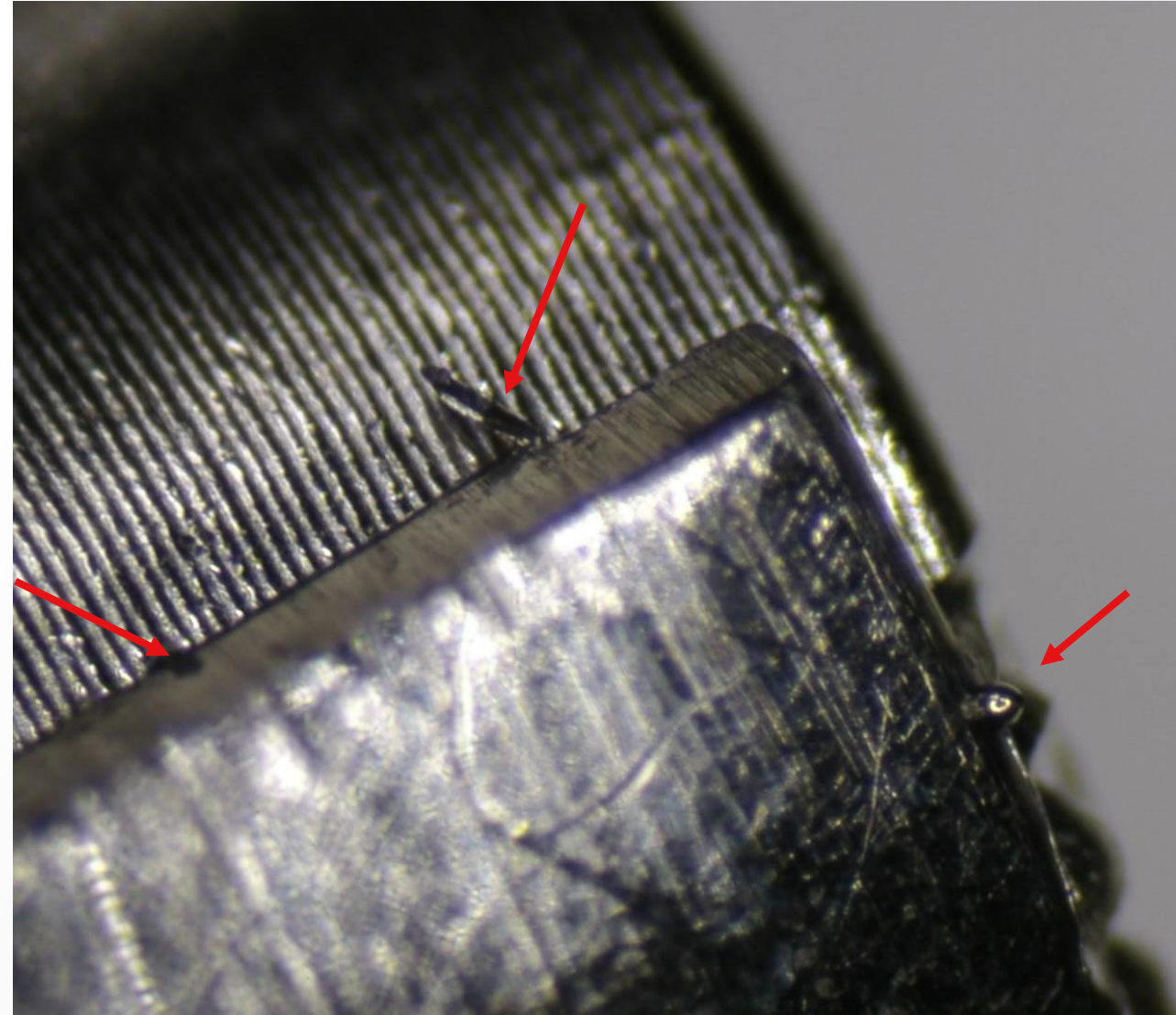


Made visible with bare eye, Optical Microscope, UV-A and Bright light

Example Assy (laser welded):

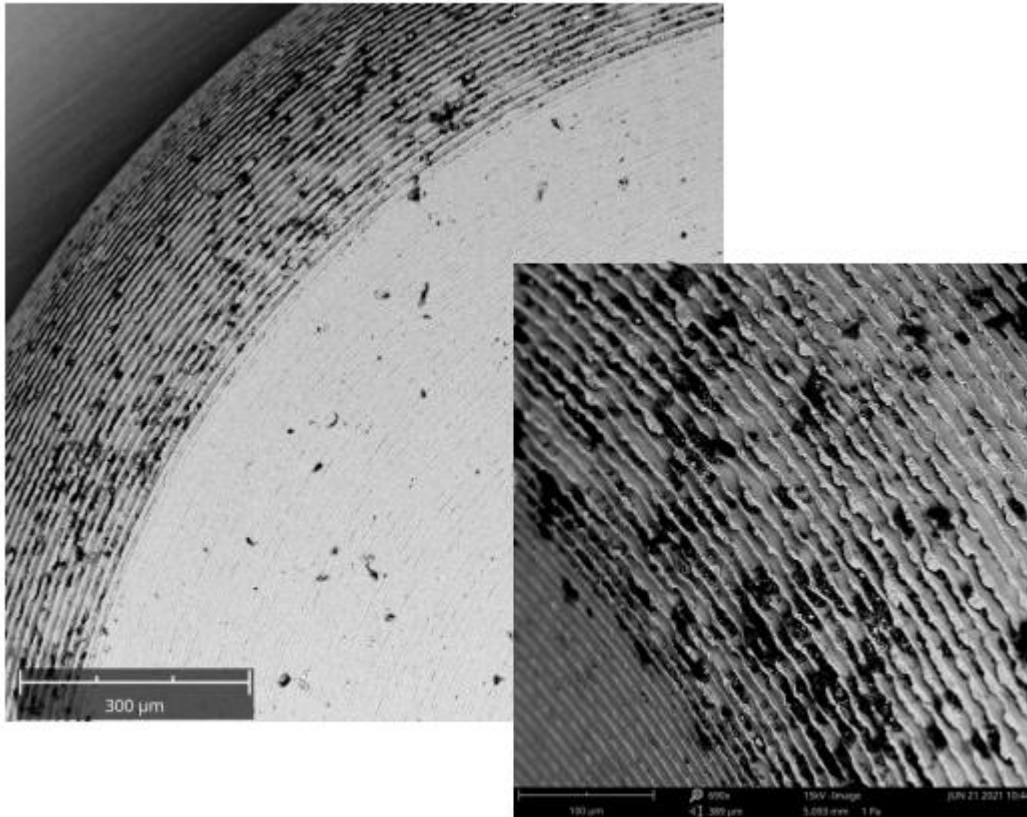


Assy cleaned too long
Galvanic corrosion Ni-Cu wire



Metal burr, laser welding droplet, organic dirt particle, discolorisation:
Made visible via Stereo Microscope 9x-40x

Example organic contamination



Imaged/analyzed in backscattered electron mode at 15kV

Current process

Titanium Turned Surface

$Ra < 0.6 \mu m$

Ploughing tracks, plastic deformed top surface and contamination

Problem: electrical charging in application

Analysis with: Phenom SEM

→ Tool for manufacturing dependency

Example organic contamination

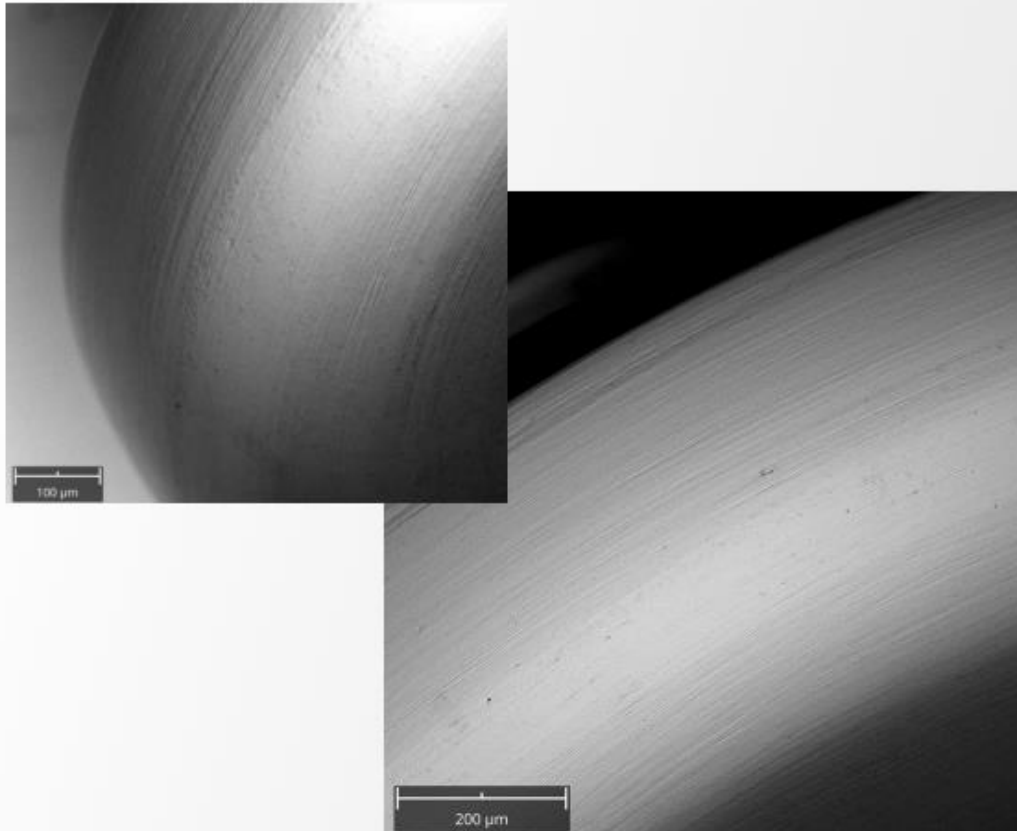
Titanium Turned Surface

$Ra < 0.6 \mu m$

→ New process

→ Tool for manufacturing changed

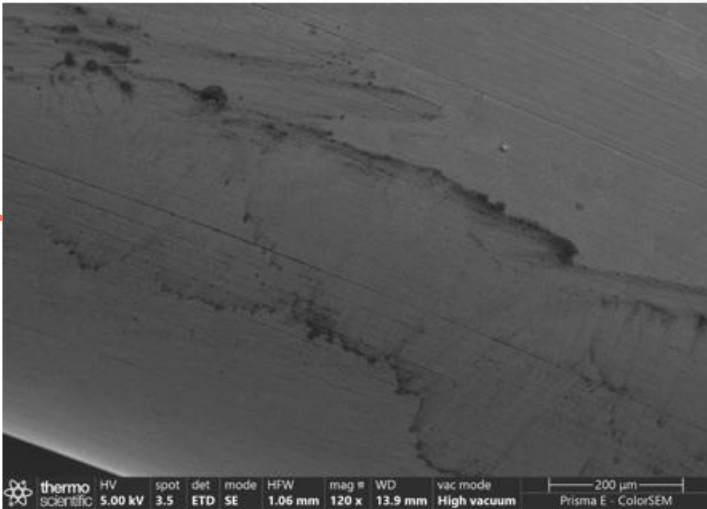
Analysis with: Phenom SEM



Imaged/analyzed in backscattered electron mode at 15kV

New process

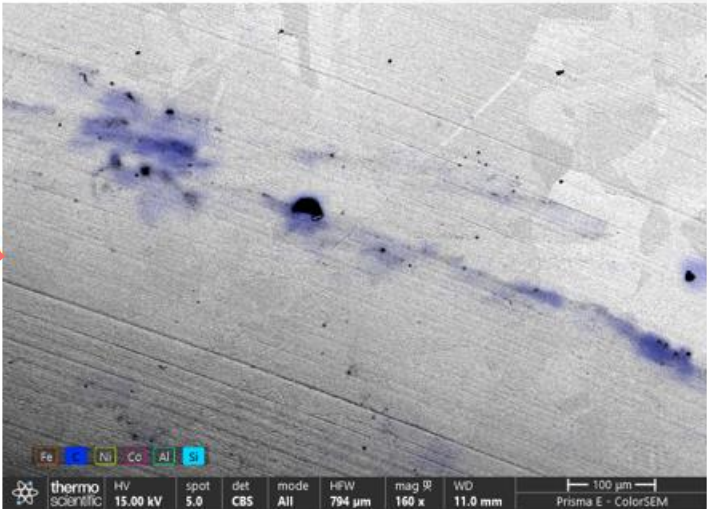
Example molecular contamination



Problem: Carbon growth in application.

Shielding bush UHV/XHV application
Surface roughnes <0.2 µm Ra

Carbon molecular contamination
(blue)

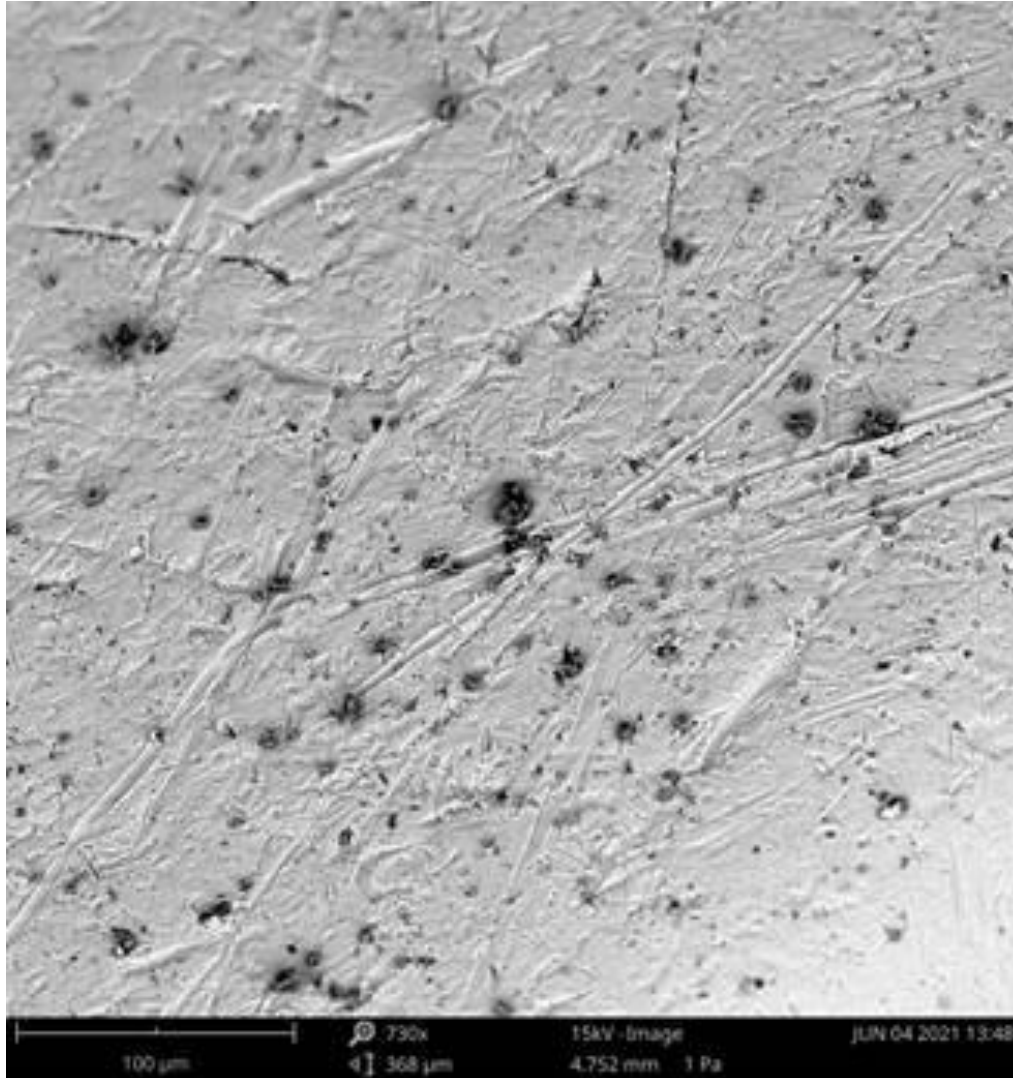


Analysis with Phenom SEM
with EDS

Element	Atomic %	Atomic % Error	Weight %	Weight % Error
Fe	48.8 %	0.1 %	52.4 %	0.1 %
C	11.5 %	0.1 %	2.7 %	0.0 %
Ni	24.5 %	0.1 %	27.6 %	0.1 %
Co	15.2 %	0.1 %	17.3 %	0.1 %

→ Mechanical polishing not wanted for UHV/XHV
Electrolitic polishing, mirror turning, plasma polishing: yes!

Example particle contamination



Molybdenum surface

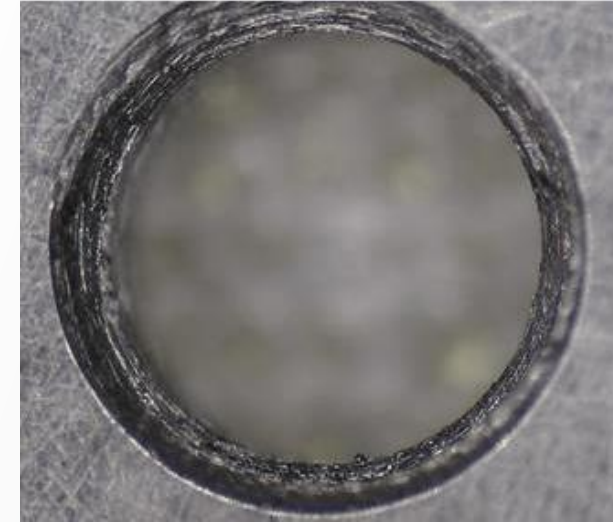
Al₂O₃ particles, size 10 μm

Half embedded in surface

Analysis with: Phenom SEM

Powder blasting not wanted!

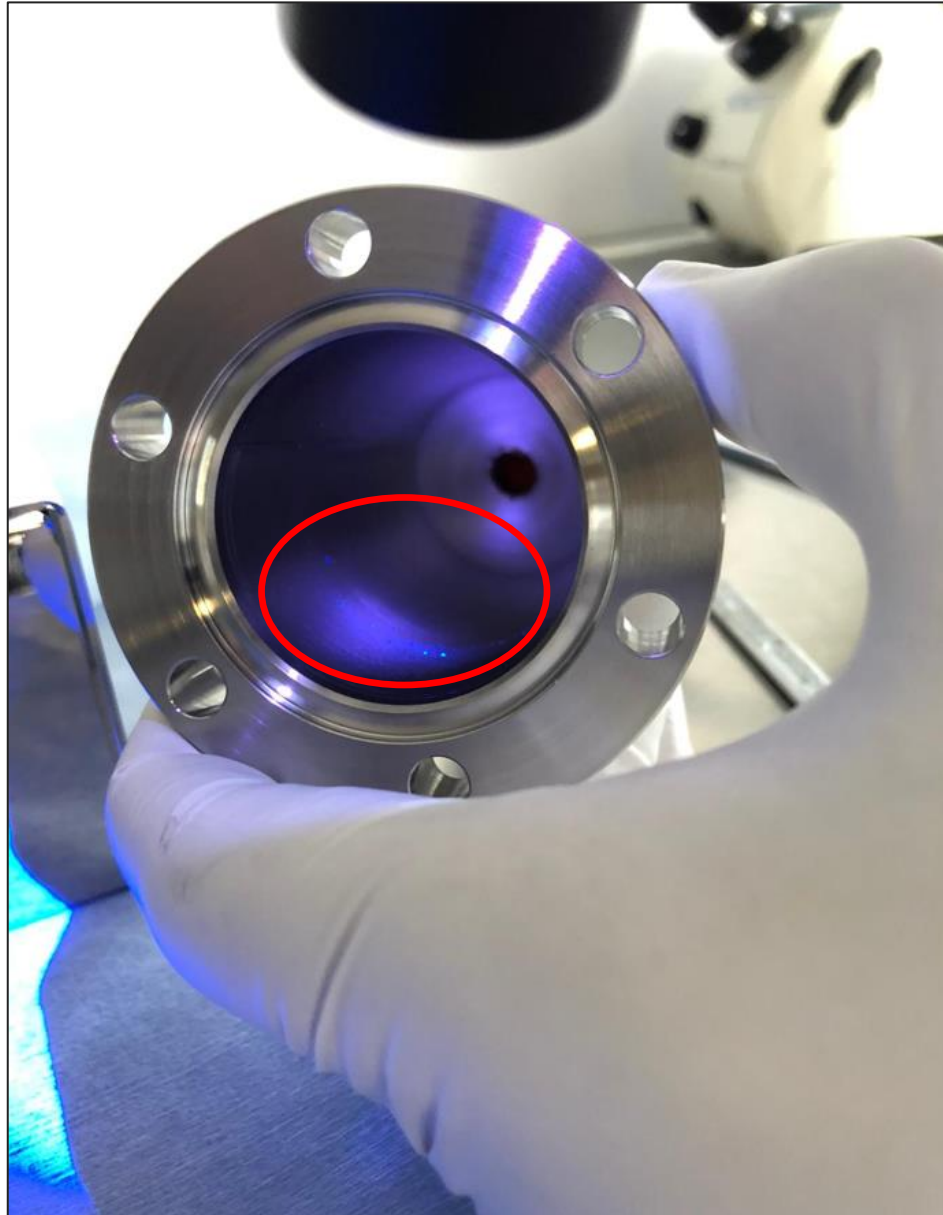
Example particle contamination



Screw thread in Molybdenum part
burrs can come loose
Visible with Optical Microscope

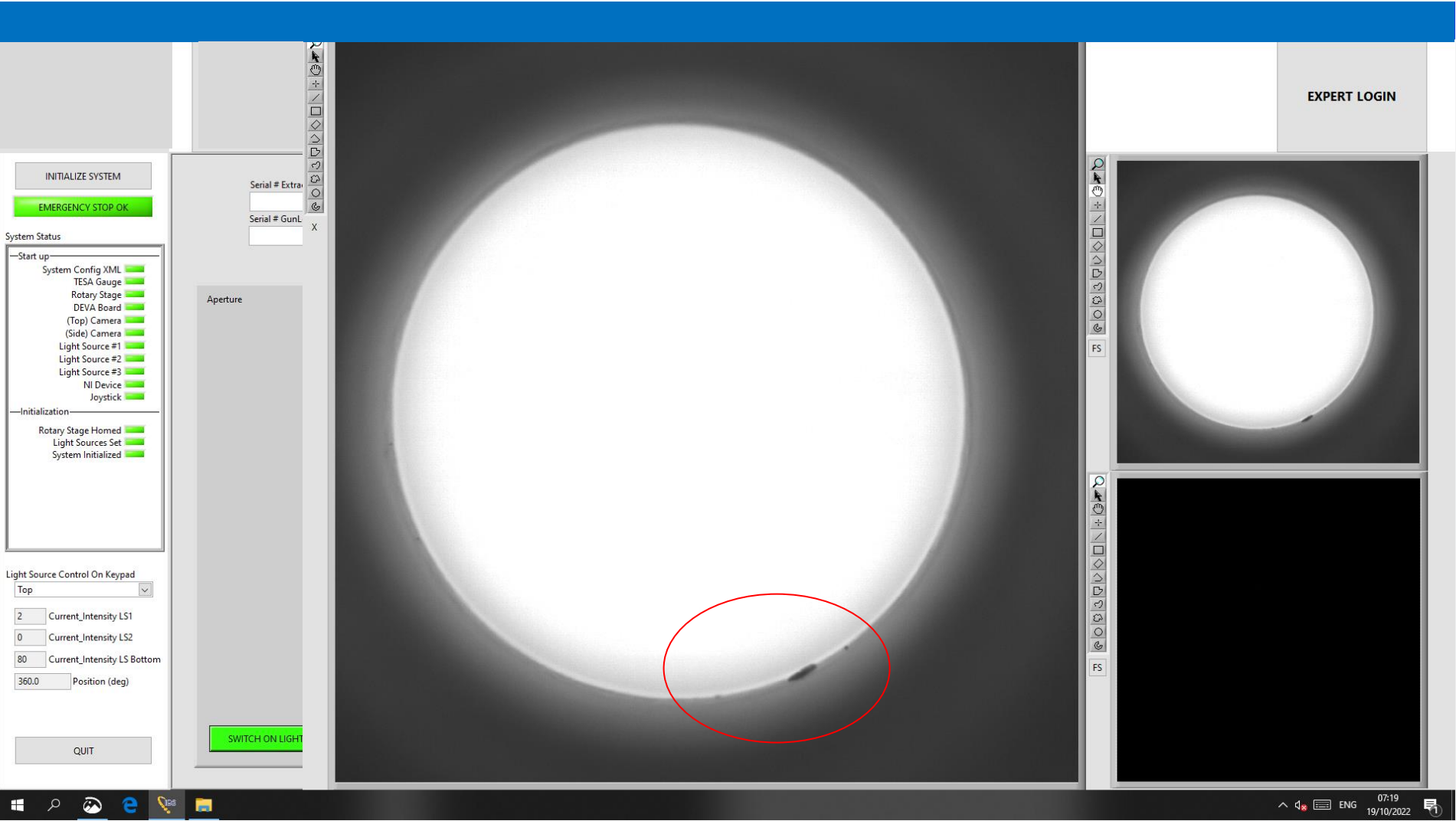
Analysis with: Phenom SEM

Example (organic) particle contamination



Particles in a vacuum transport container
Bare eye observation
Made visible with: UV-A light

Example particle contamination



Particles inside aperture

Problem electrical charging in application

Made visible with:
optical microscope

Example particle contamination

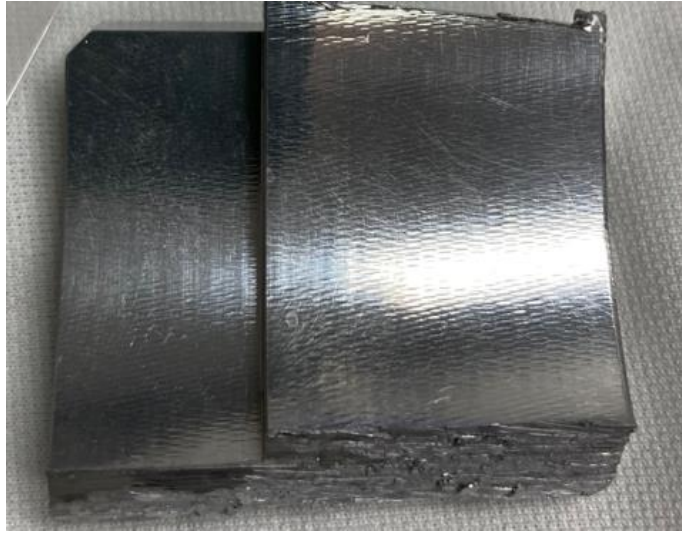


Beam blocking issue

Aperture hole with metal droplet
from a spark welding process

Visible with: Optical Stereo Microscope
Analysis with: Phenom SEM

Contamination on lead parts.



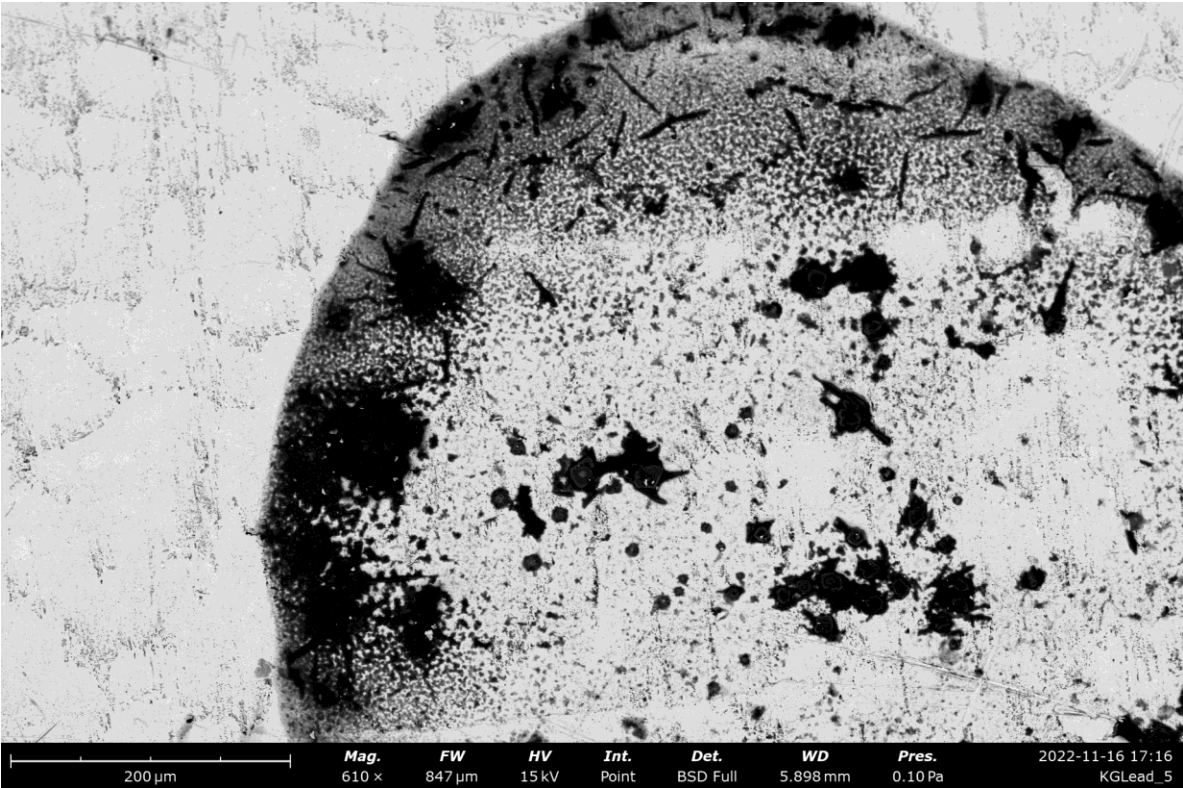
lead



Cloth with Ipa wiped over the lead

Contamination on lead parts.

Samples cleaned and bake-out. (Surface without stains).



2. Region

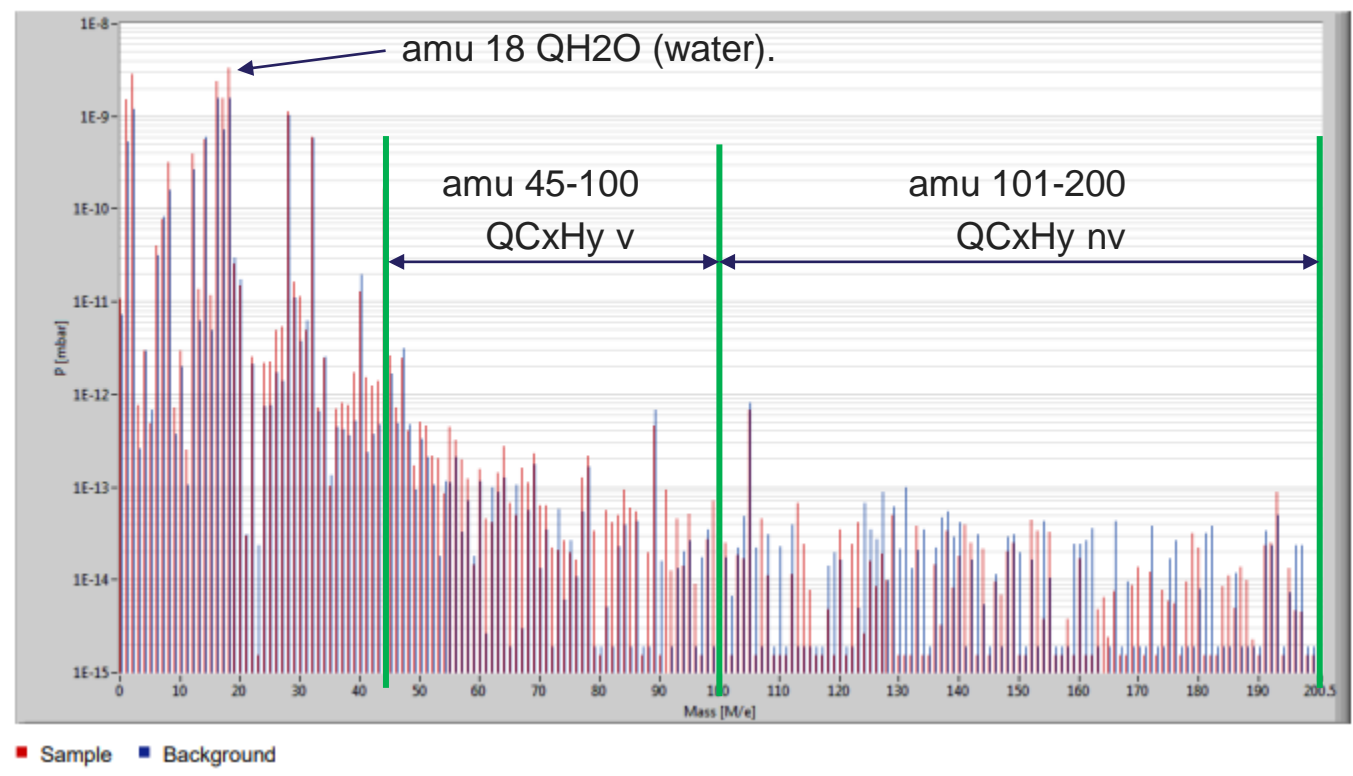
Scanning electron micrograph (SEM) of a lead part surface. A blue rectangular box highlights a region of interest in the upper right quadrant. The image shows a dark, irregularly shaped region on the left side, which is heavily contaminated with small, dark, needle-like or fibrous structures. The rest of the surface is lighter and appears relatively clean. A scale bar at the bottom left indicates 200 μm. Technical data at the bottom: Mag. 610 x, FW 847 μm, HV 15 kV, Int. Point, Det. BSD Full, WD 5.898 mm, Pres. 0.10 Pa, 2022-11-16 17:16, KGLoad_5.

Photograph of a lead part. A red arrow points to a specific region on the surface, which is the same region highlighted in the SEM image above.

FW: 450 um. Mode: 15 kV - Point, Detector: BSD Full, Time: 11/16/22 5:16 PM

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	53.386	14.400
11	Na	Sodium	11.864	4.600
17	Cl	Chlorine	7.862	4.700
19	K	Potassium	6.220	4.100
82	Pb	Lead	20.669	72.200

Contamination on Lead parts



Outgas Values				
	Qmeasured [mbar•l/s]	Qbackground [mbar•l/s]	Qbatch [mbar•l/s]	Qspec [mbar•l/s]
H2O	5.33E-7	2.51E-7	2.82E-7	5.00E-6
CxHy v	1.59E-9	1.22E-9	< 1.22E-9	5.00E-9
CxHy nv	2.55E-10	3.34E-10	< 3.34E-10	5.00E-10
N2	1.48E-7	1.34E-7	< 1.34E-7	5.00E-7
O2	7.90E-8	7.79E-8	< 7.79E-8	5.00E-8
Ar	1.72E-9	2.56E-9	< 2.56E-9	1.00E+0

