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Sales Engineer

CleanControlling®

Online Basic Seminar
TECHNICAL CLEANLINESS
VDA 19.1 and VDA 19.2



Microscopic Analysis in VDA 19.1

Dr. Nils Rotthowe, CleanControlling GmbH

MICROSCOPIC ANALYSIS IN VDA 19.1

- Gravimetric analysis
- Microscopy according to VDA 19.1 – general
- Technical fundamentals of microscopy
- Standard analysis
- Free analysis
- What light-optical particle analysis can do…
- … and what it can’t

MICROSCOPIC ANALYSIS IN VDA 19.1

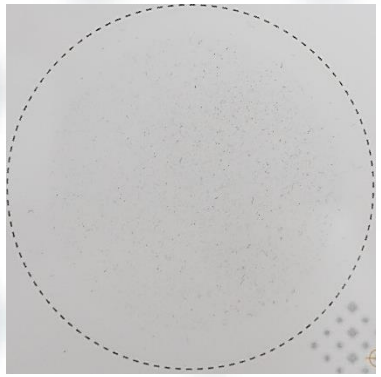
Gravimetric analysis

- Gravimetry determines the total mass of all extracted particles
- Integral measure of the contamination level



- Well suited for heavily contaminated components
  - Where microscopic analysis may be impossible
- Poorly suited for low contamination levels
  - Reproducible results for < 1 mg only possible with high technical effort





- **No information on particle size or material!**

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MICROSCOPIC ANALYSIS IN VDA 19.1

Microscopy – general

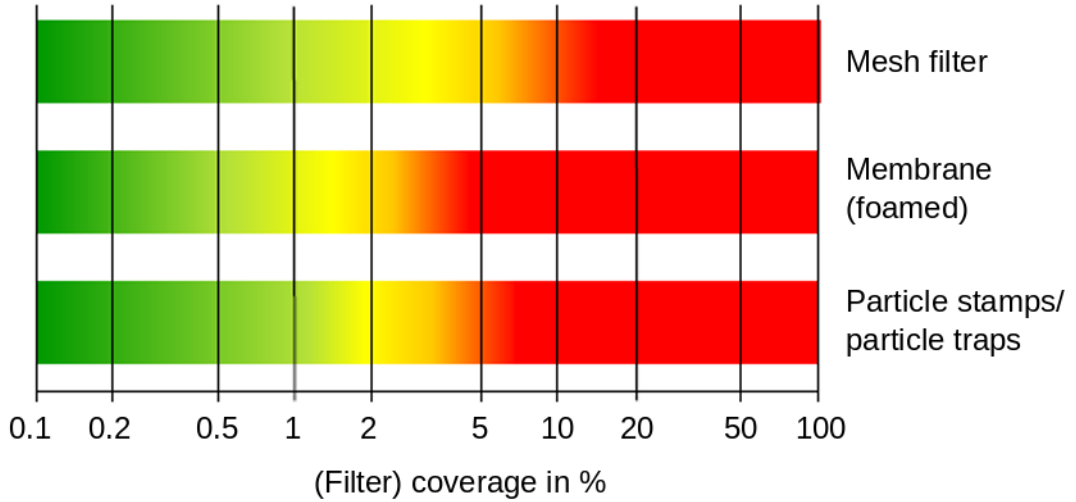
- Microscopy is the core of VDA 19.1 cleanliness analysis
- Standard analysis → verification of cleanliness specifications
  - Use of automated and standardized measurement methods
- Free analysis → special questions (size, color, etc.)
- Distinction between three particle classes
  - metallic-shiny particles
  - not-metallic-shiny particles
  - Fibers

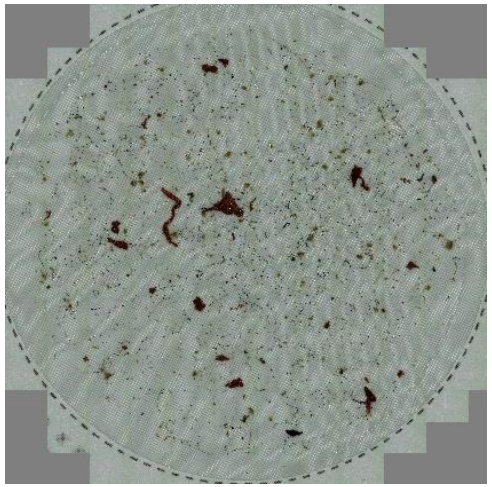


MICROSCOPIC ANALYSIS IN VDA 19.1

Microscopy – general

- Prerequisites
  - Qualified extraction → successful declining test (from 50 µm)
  - Analyzable filter → little/no particle overlap
  - Qualified personnel
  - Post-analysis review

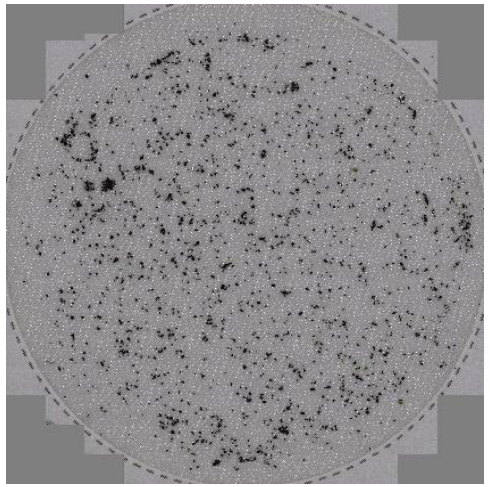




4 % filter coverage

← Not analyzable

Analyzable →



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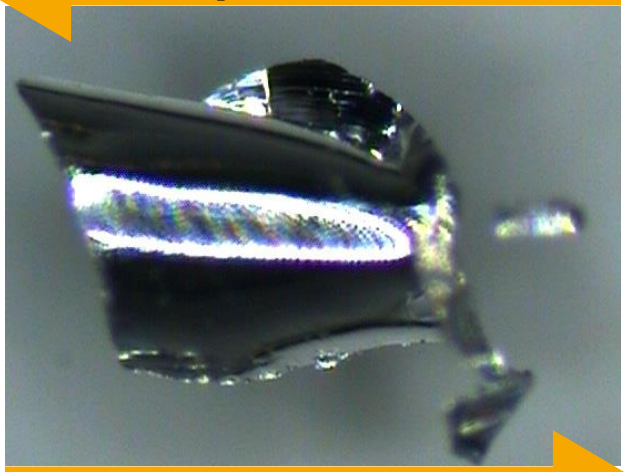
MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – The microscopes

Stereo microscope



Depth of field



Magnification

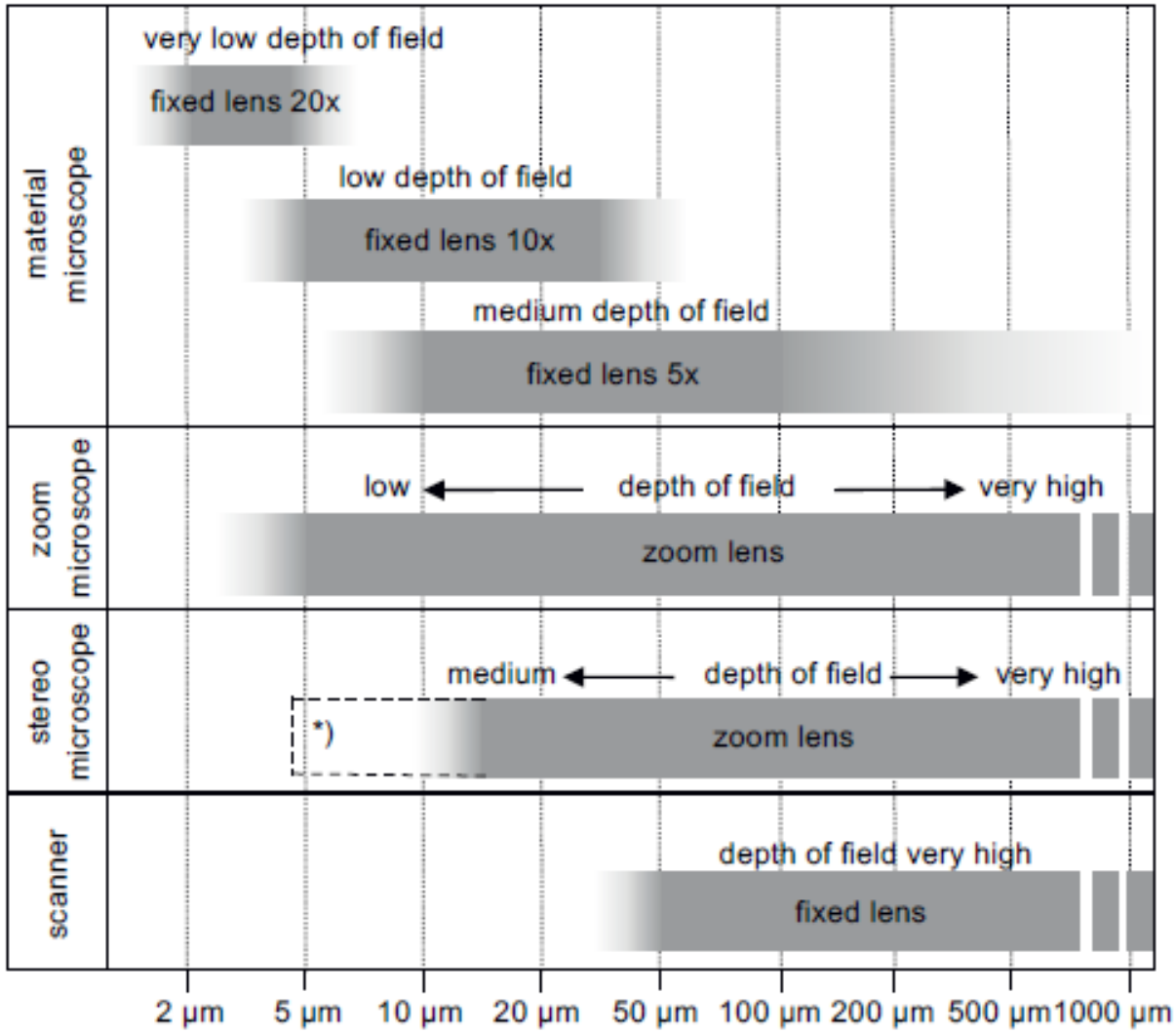


Material microscope

Source: Jomesa

MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – The microscopes



recommended suitability for particle size range

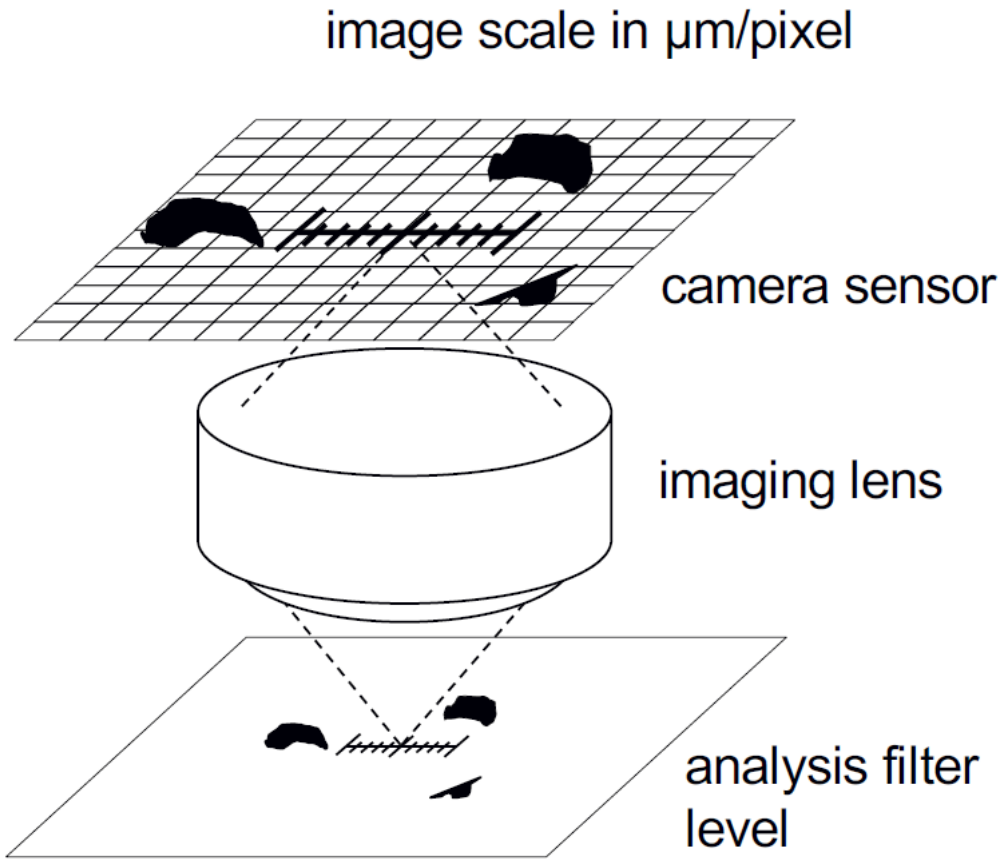
depth of field:		
	very low	1 – 10 µm
	low	10 – 50 µm
	medium	50 – 100 µm
	high	100 – 1000 µm
	very high	> 1000 µm

Source: VDA 19.1

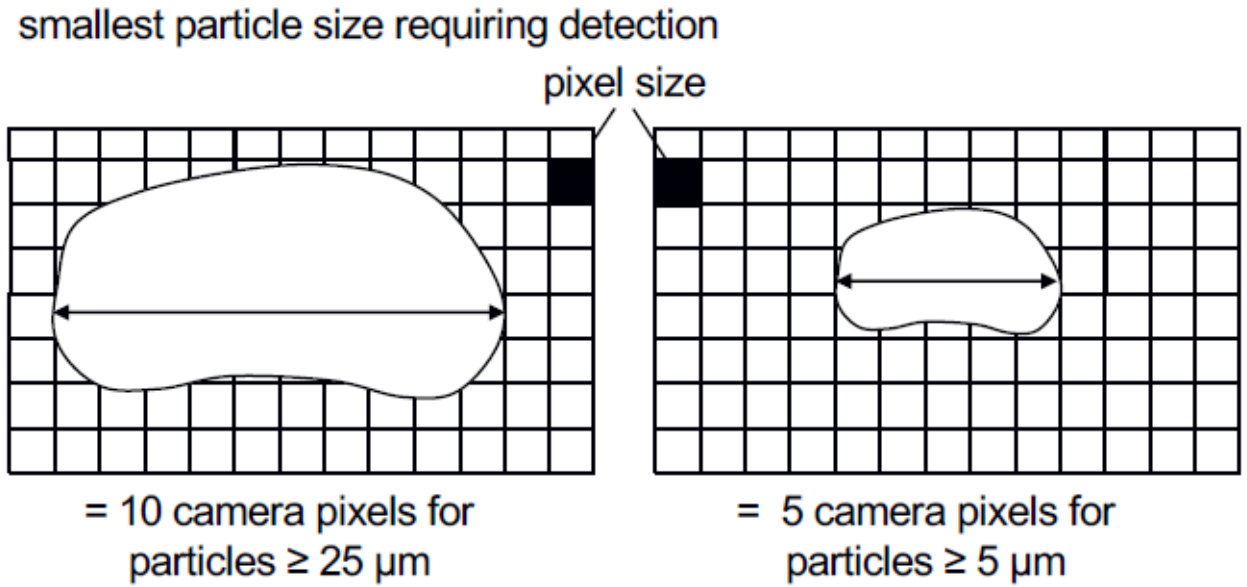
MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Image acquisition, resolution and magnification

- Particles are digitized by the microscope and stored as a pixel image



Source: VDA 19.1

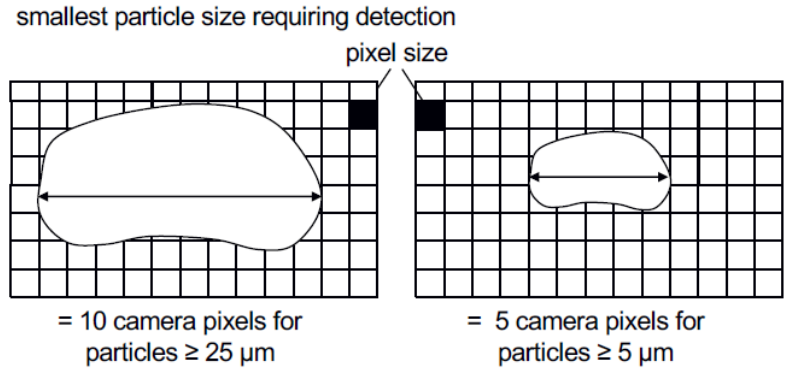


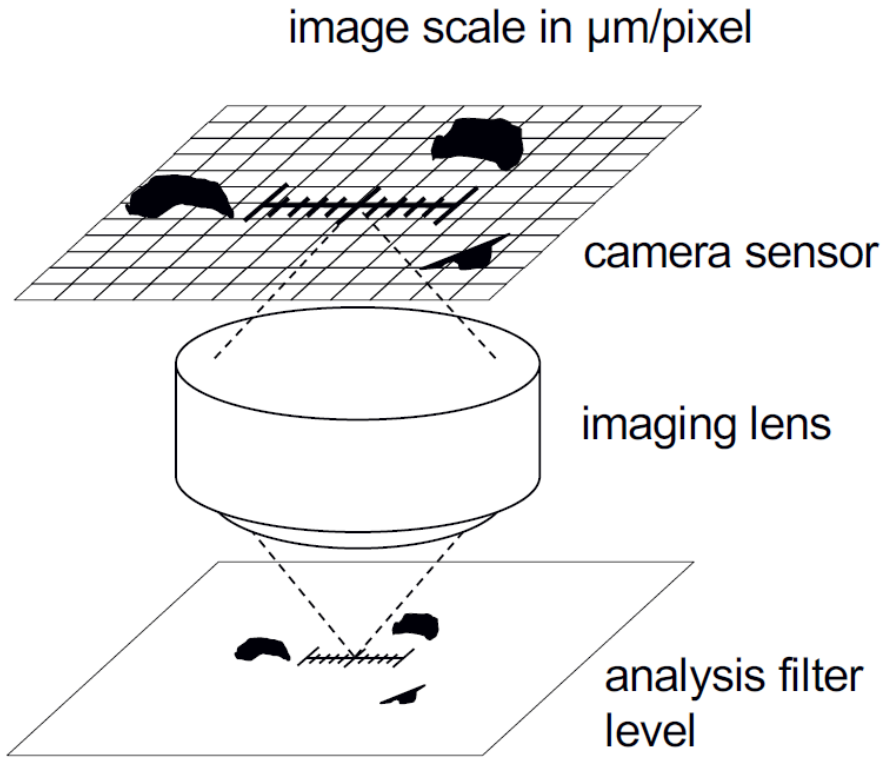
Source: VDA 19.1

MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Image acquisition, resolution and magnification

- Particles are digitized by the microscope and stored as a pixel image
- A correlation with the magnification of a standard light microscope is not possible

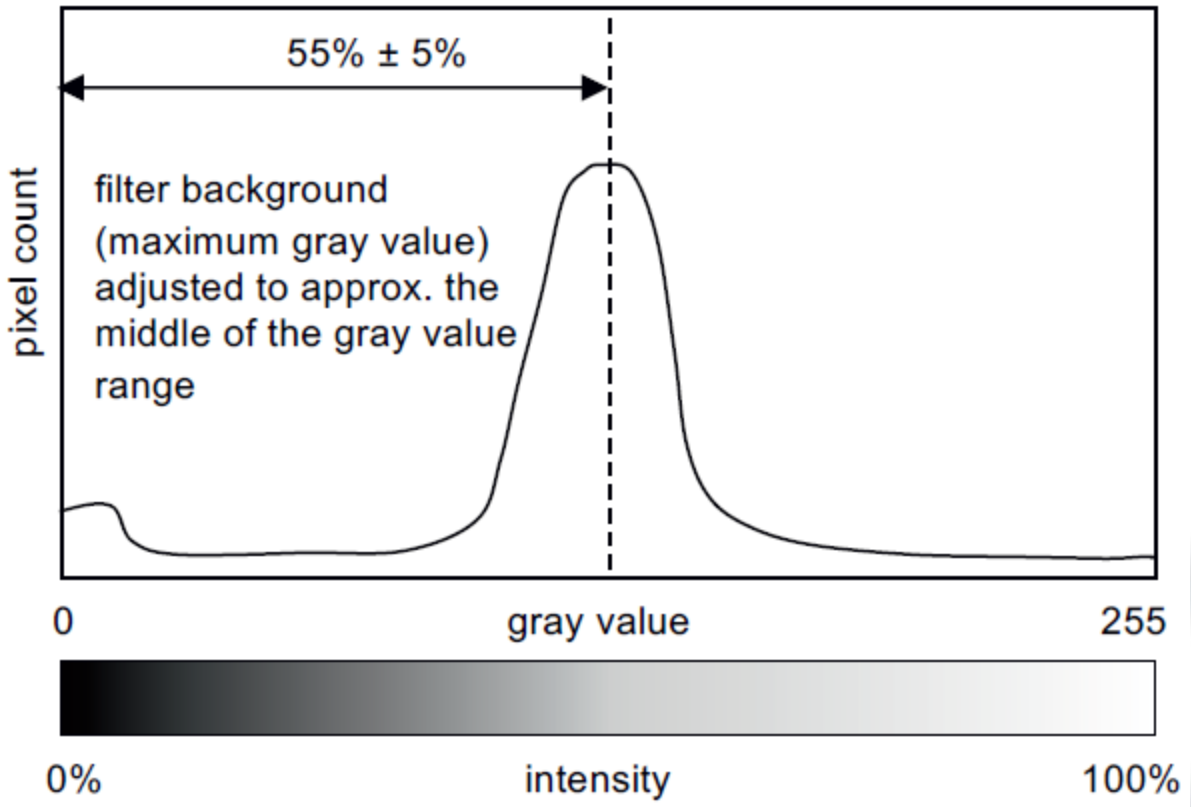




MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle detection

- When is a particle detected as a particle?
  - Division of the filter image into gray levels
  - Filter background defined at 55 % ± 5 %

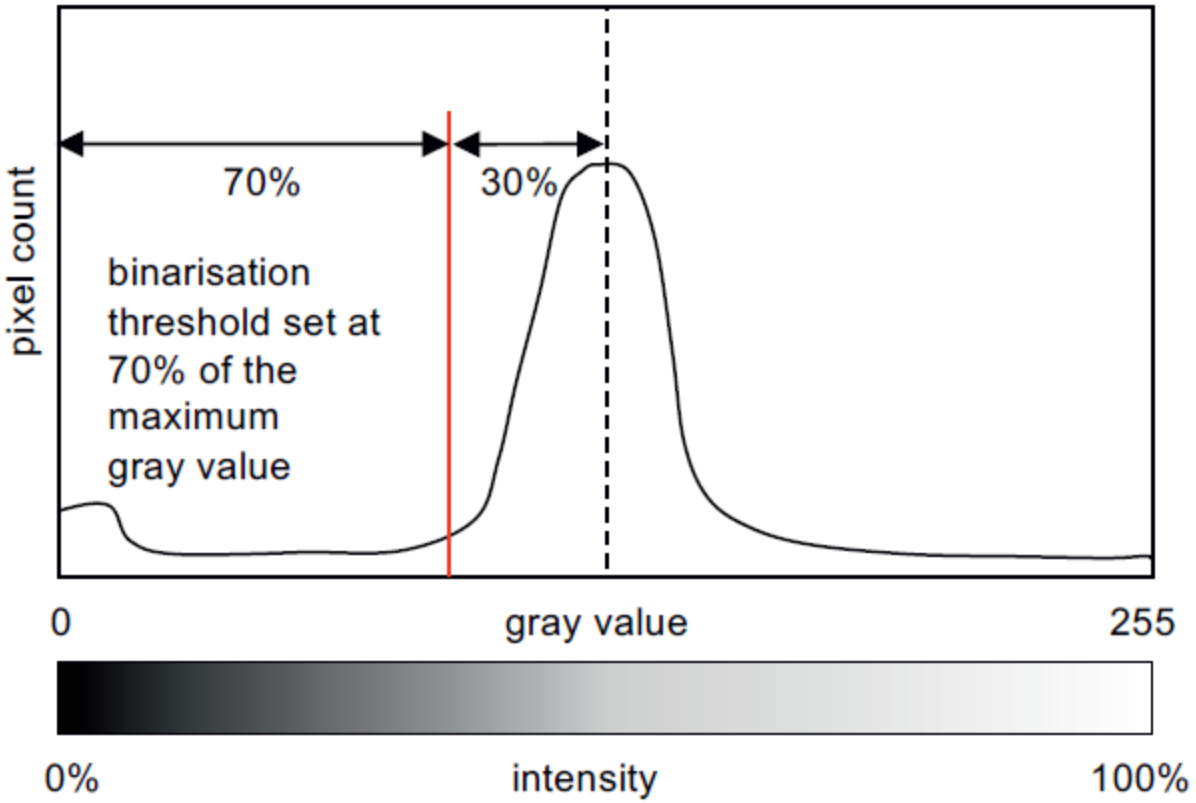


Source: VDA 19.1

MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle detection

- When is a particle detected as a particle?
  - Division of the filter image into gray levels
  - Filter background defined at 55 % ± 5 %
  - Binarization threshold at 70 % of the filter background



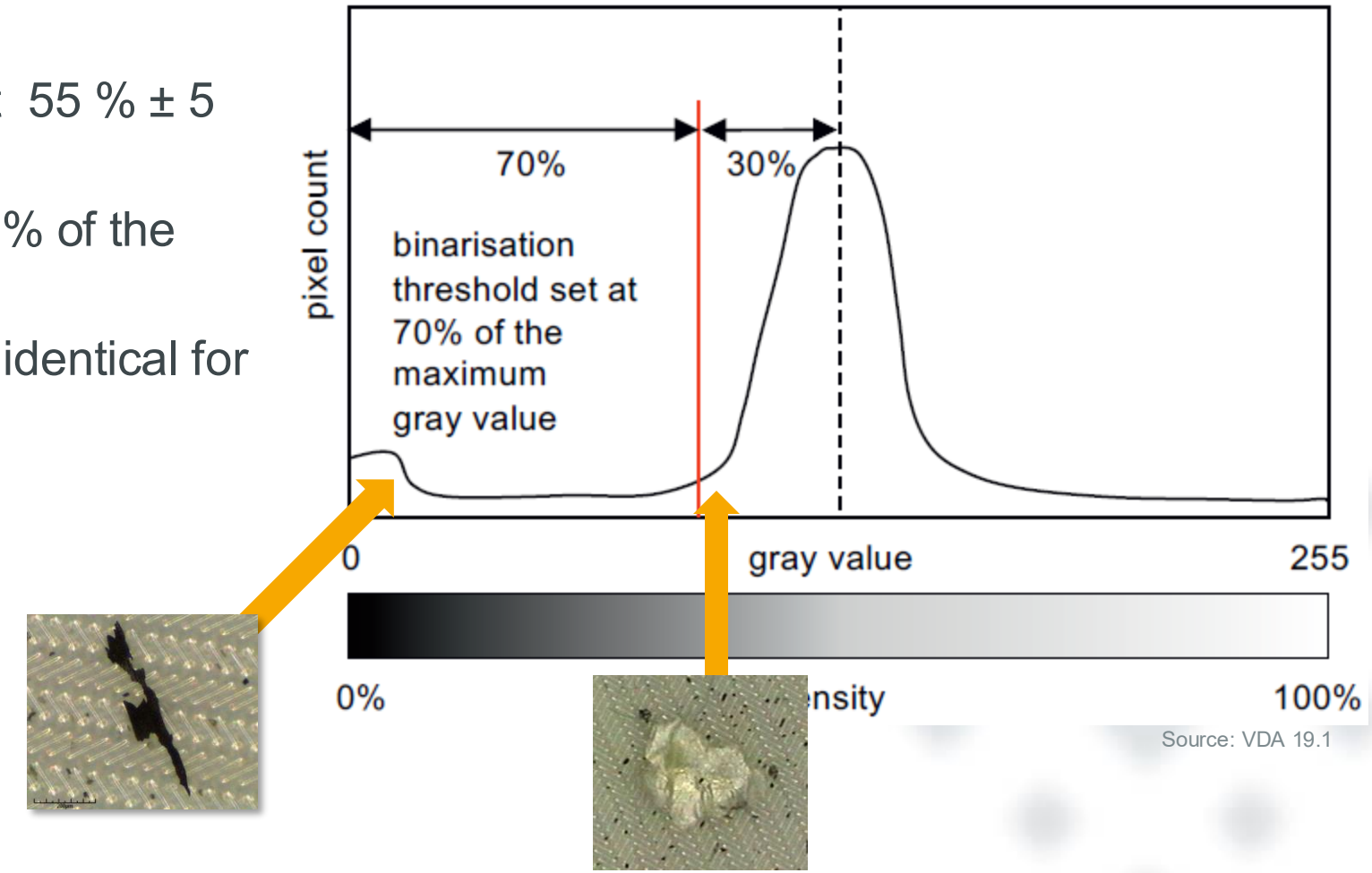
Source: VDA 19.1

MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle detection

- When is a particle detected as a particle?
  - Division of the filter image into gray levels
  - Filter background defined at 55 % ± 5 %
  - Binarization threshold at 70 % of the filter background
  - These parameters **must** be identical for every analysis

→ reproducibility of results

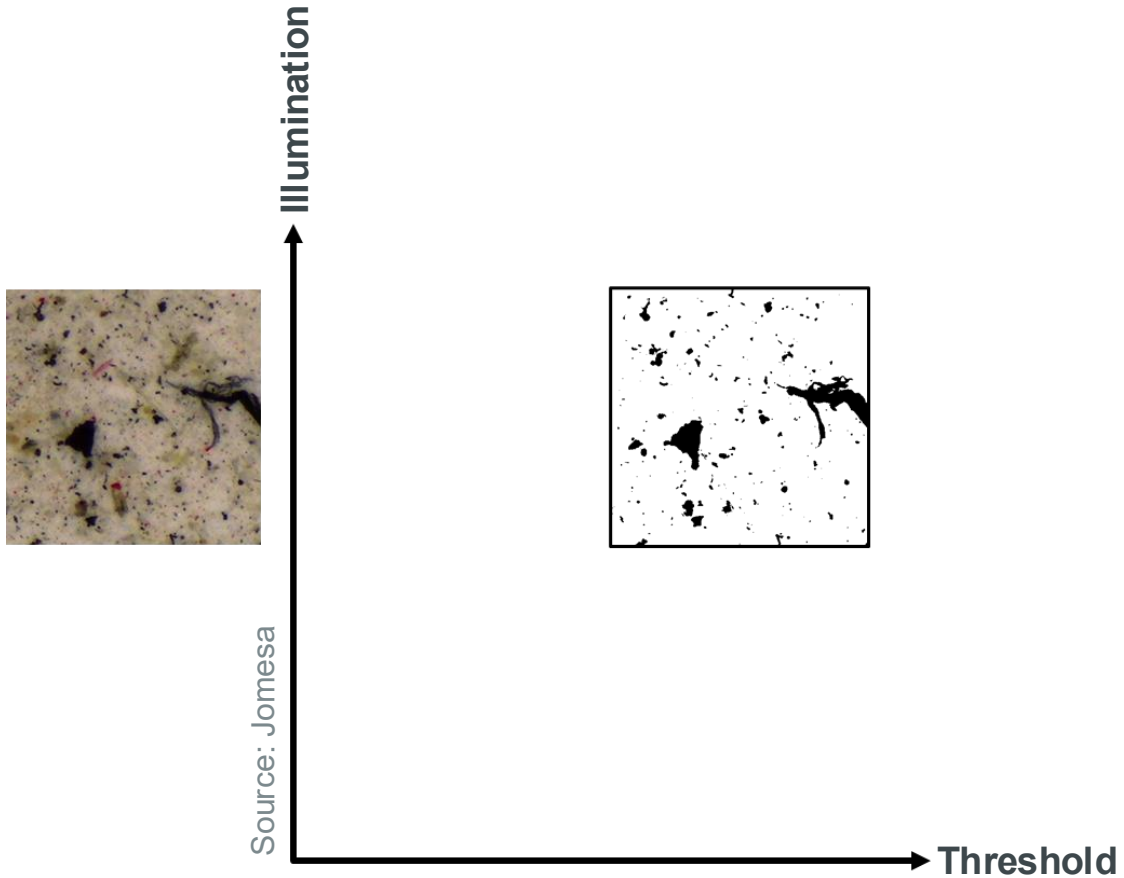


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MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle detection

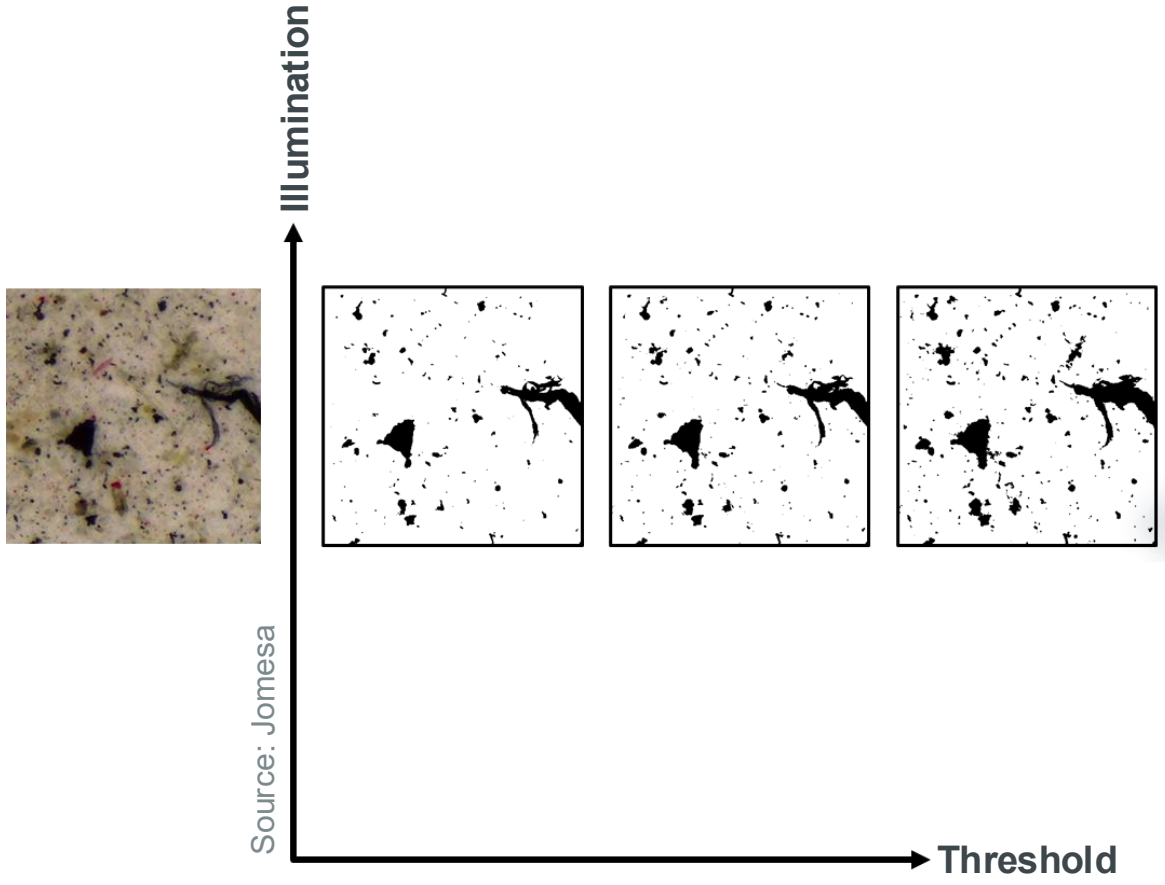
Particle detection / illumination and threshold



MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle detection

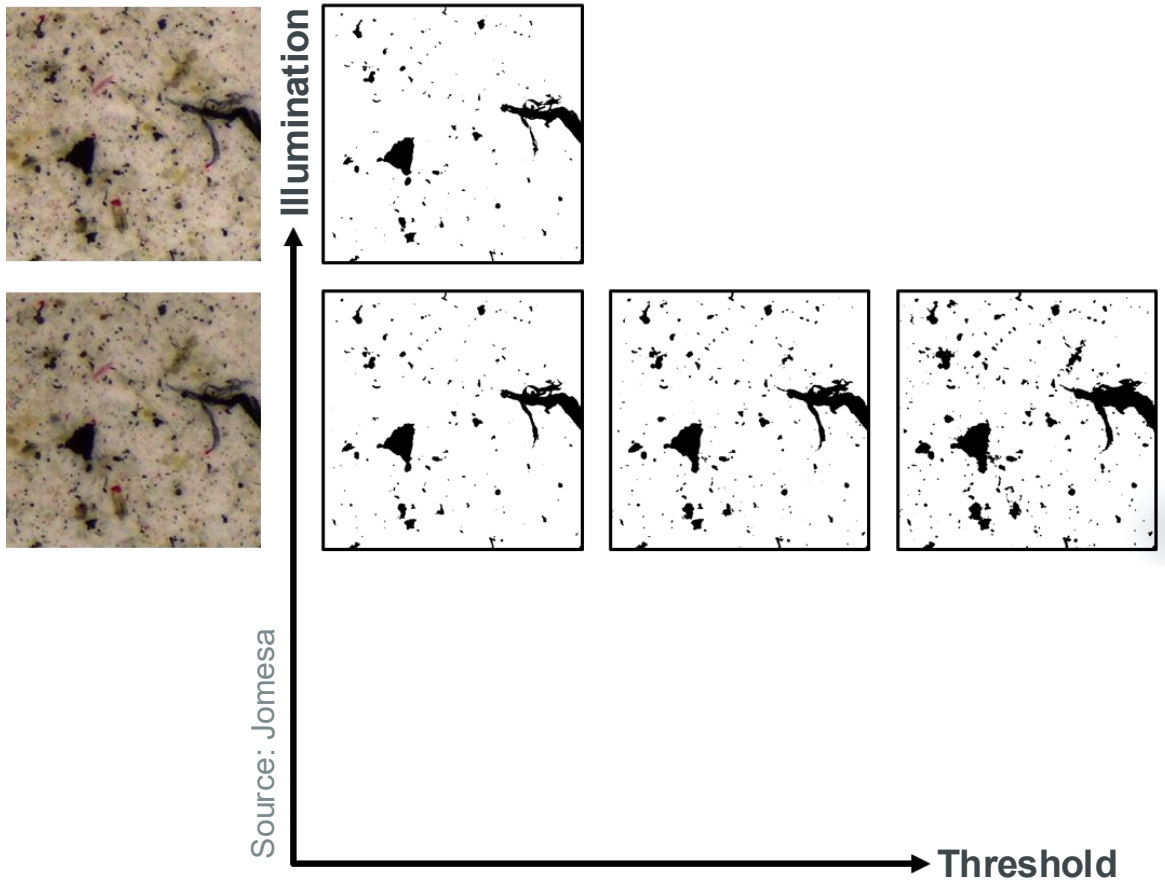
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Technical fundamentals – Particle detection

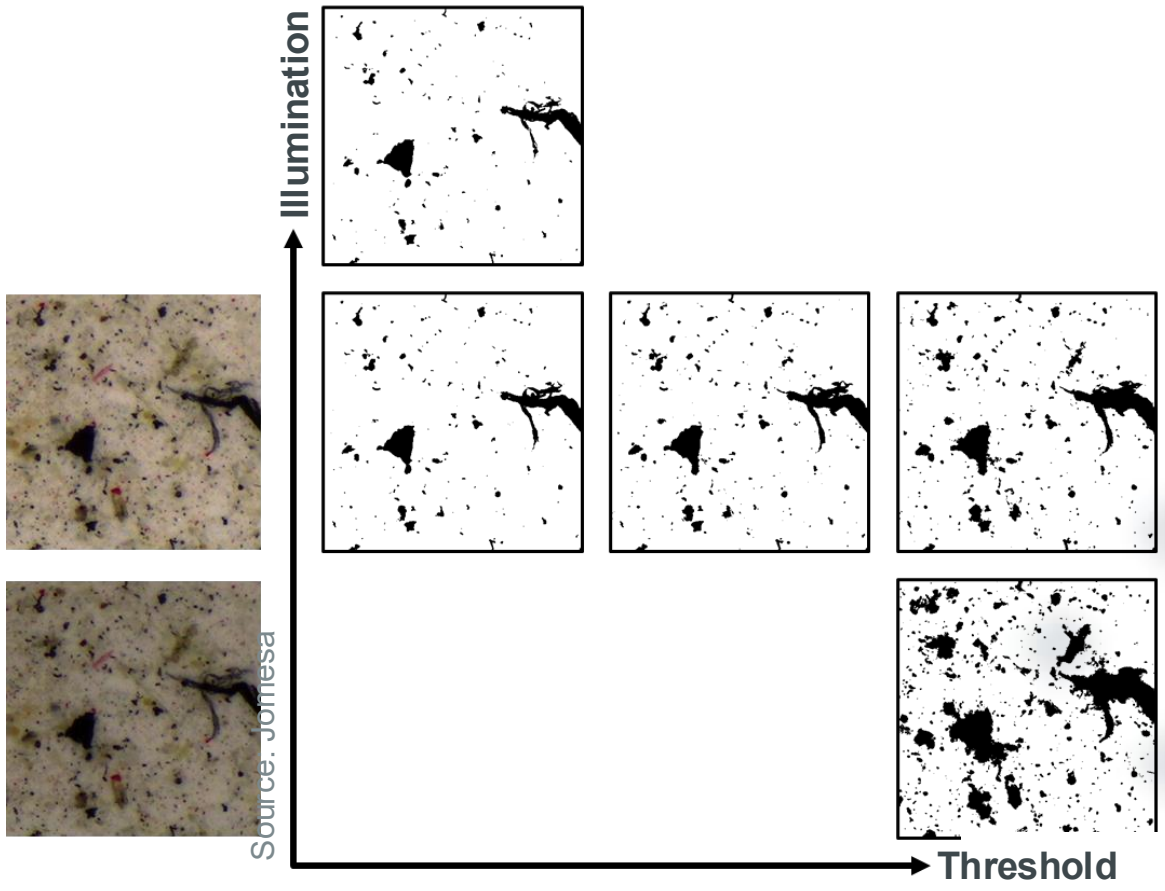
Particle detection / illumination and threshold



MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle detection

Particle detection / illumination and threshold



MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle measurement

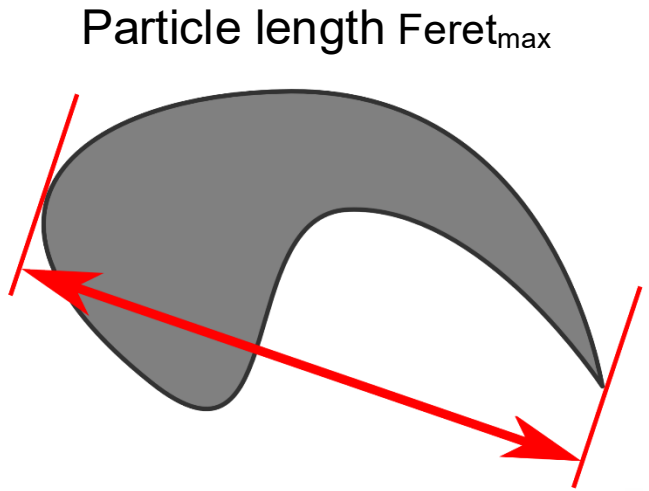
- Particles have no defined geometry
- Length and width must be defined



MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle measurement

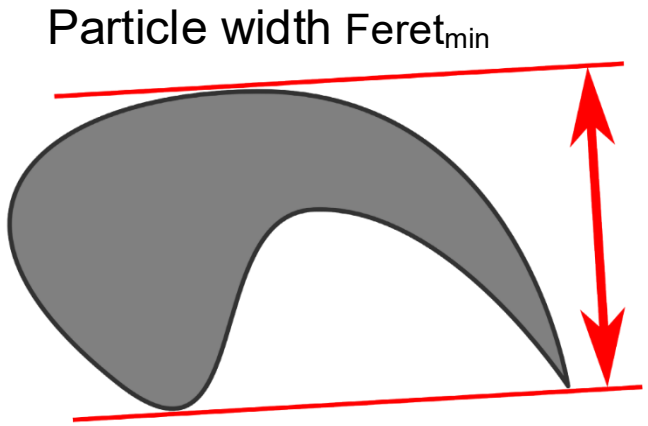
- Particles have no defined geometry
- Length and width must be defined
  - Length is defined by $Feret_{max}$ → max. risk potential



MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle measurement

- Particles have no defined geometry
- Length and width must be defined
  - Length is defined by $Feret_{max}$
  - Width is defined by $Feret_{min}$:

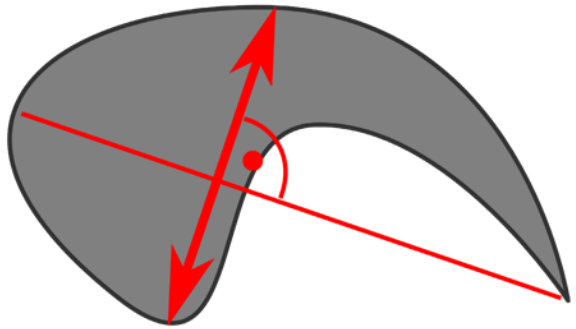


MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle measurement

- Particles have no defined geometry
- Length and width must be defined
  - Length is defined by $Feret_{max}$
  - Width is defined by $Feret_{min}$:
  - Alternatively:
    - Orthogonal cut

Particle width perpendicular section

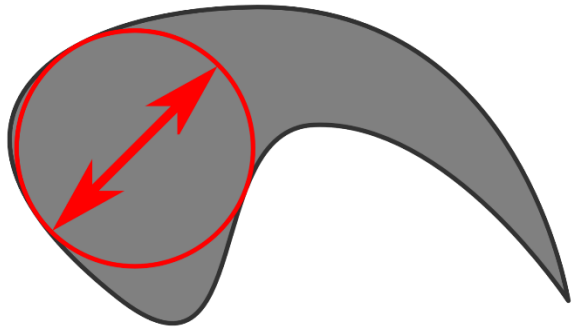


MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle measurement

- Particles have no defined geometry
- Length and width must be defined
  - Length is defined by $Feret_{max}$
  - Width is defined by $Feret_{min}$:
  - Alternatively:
    - Perpendicular section
    - Maximum inscribed circle

Particle width maximum inscribed circle



MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle detection

Particle detection

- For particles with $Feret_{max} \geq 50$ µm
- A particle must consist of at least 10 pixels
- Illumination and brightness threshold are strictly defined values



Comparability
of
anaylsis results

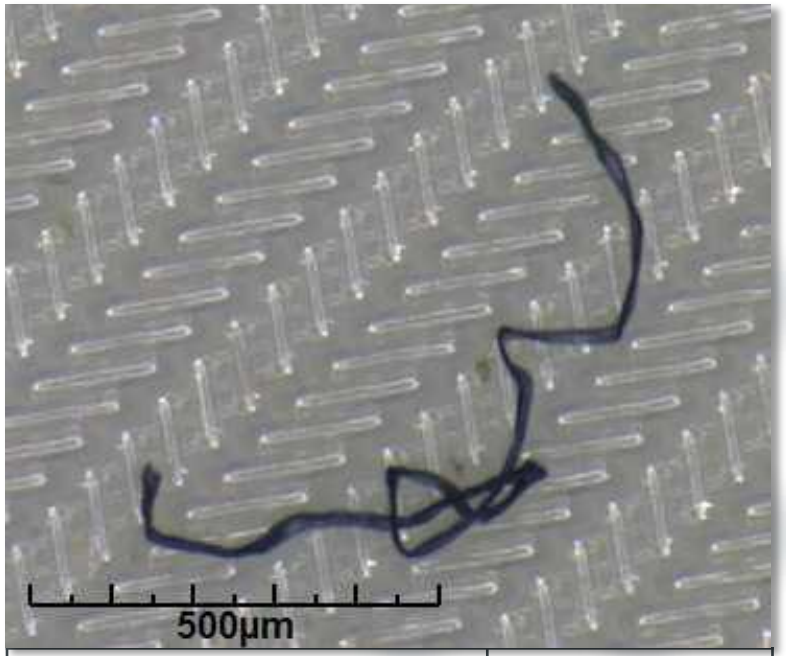
MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle measurement: fibers

- A human hair is 60 µm – 120 µm wide and therefore counts as a particle according to VDA 19.1, **not** as a fiber

- $Feret_{max}$ unsuitable as length measure due to flexibility → random orientation
- Stretched length is better suited

- When is a particle a fiber?
  - non-metallic-shiny
  - geometric criterion



$Feret_{max}$	756 µm
Stretched length	1500 µm

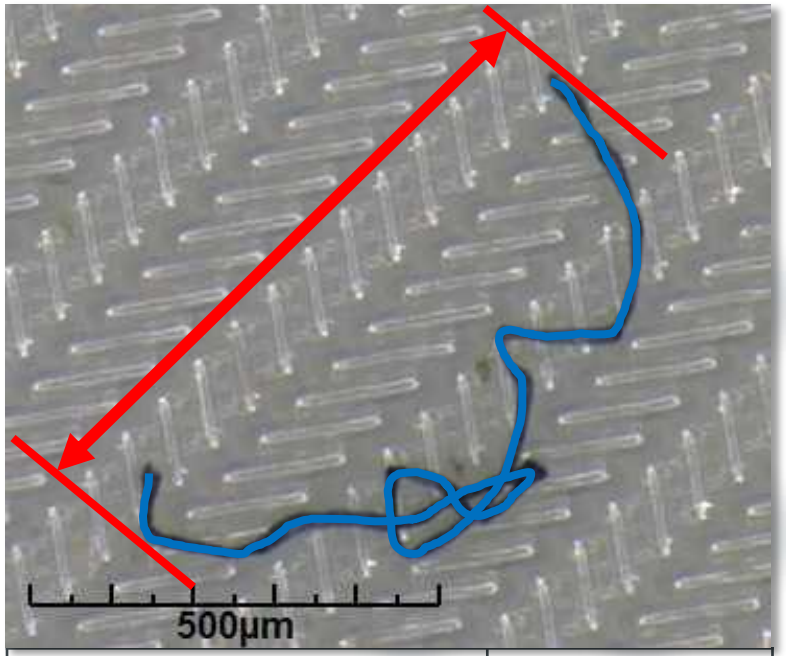
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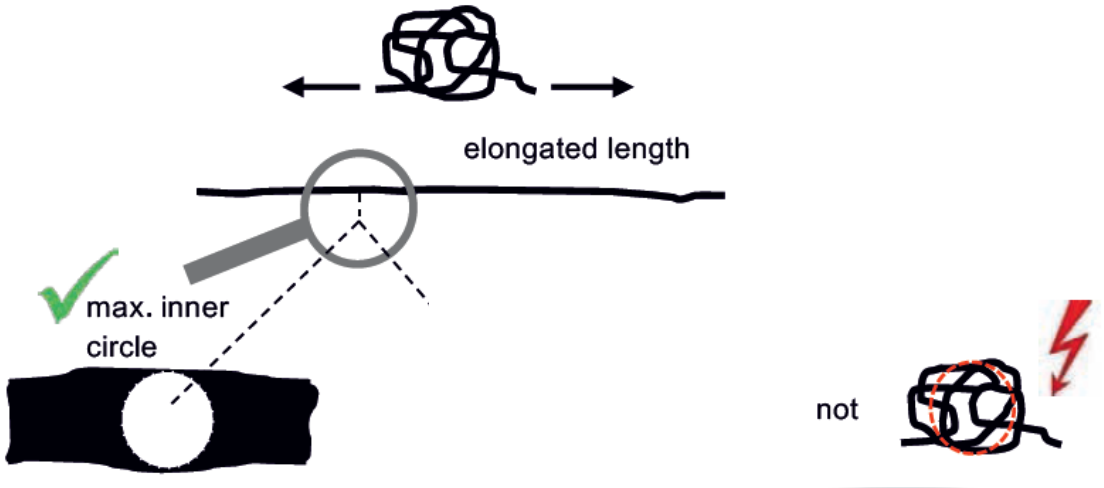


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MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle measurement: fibers

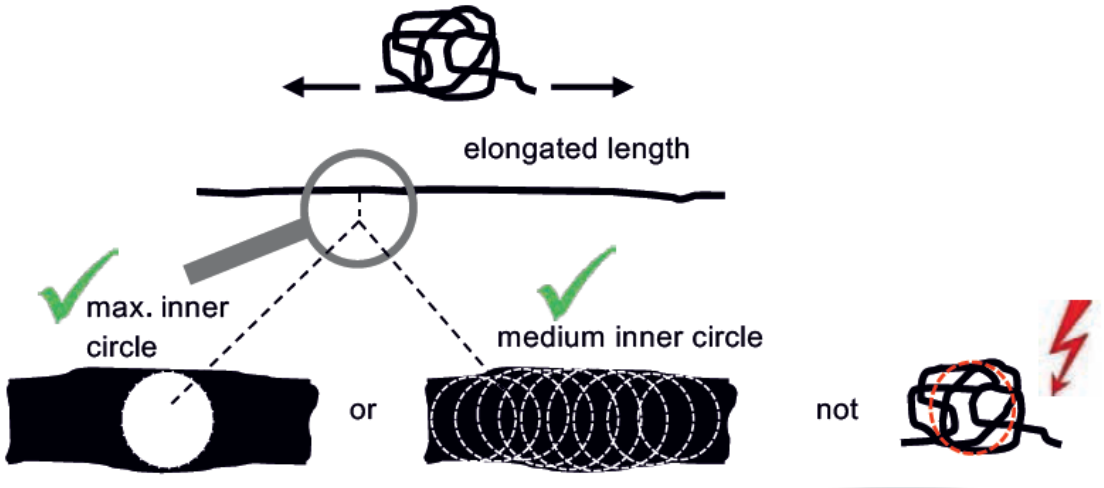
- Option 1 (max. inscribed circle):
  - not-metallic-shiny
  - Stretched length / maximum inscribed circle > 20
  - Width: maximum inscribed circle ≤ 50 µm



MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle measurement: fibers

- Option 1 (max. inscribed circle):
  - non-metallic-shiny
  - Stretched length / maximum inscribed circle > 20
  - Width: maximum inscribed circle ≤ 50 µm



- Option 2 (mean inscribed circle):
  - non-metallic-shiny
  - Stretched length / mean inscribed circle > 20
  - Width: mean inscribed circle ≤ 50 µm

- Option 2 “smooths” fibers with “bulges” or adhering particles

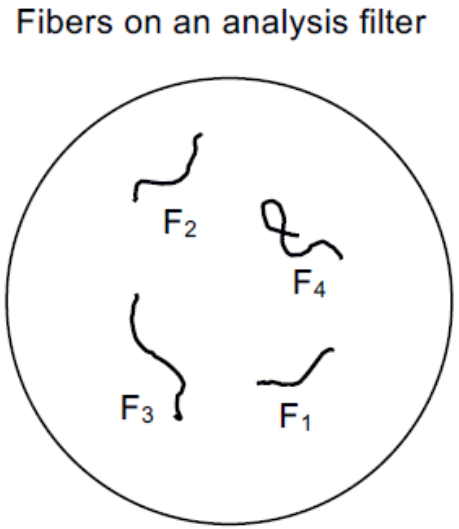
MICROSCOPIC ANALYSIS IN VDA 19.1

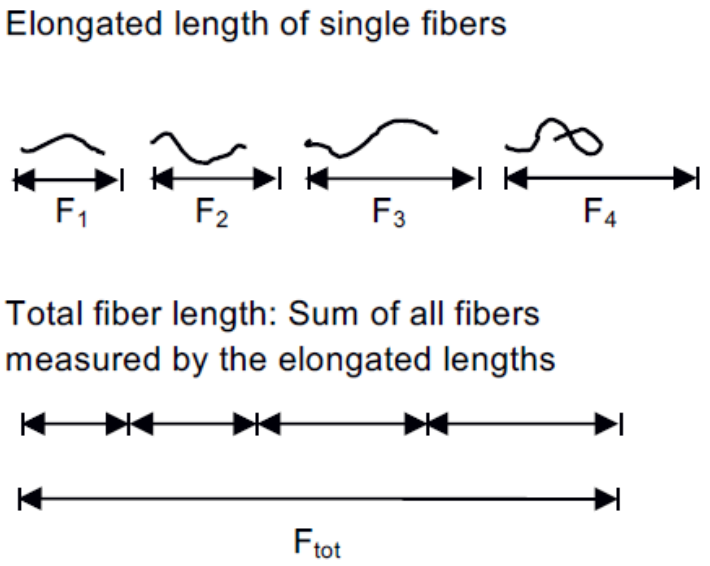
Technical fundamentals – Particle measurement: fibers

- Fiber criteria in cleanliness specs quickly lead to high effort (production, handling, packaging etc.)

- Total length (F_{Tot}) of all fibers as an integral measure







MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle measurement: fibers

- Particle length is always $Feret_{max}$
- Particle width is $Feret_{min}$
- Length ≥ width ≥ height applies
- Fibers:
  - $Feret_{max}$ *vs.* stretched length
  - Definition via *max. vs. medium inscribed circle*

MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle typing

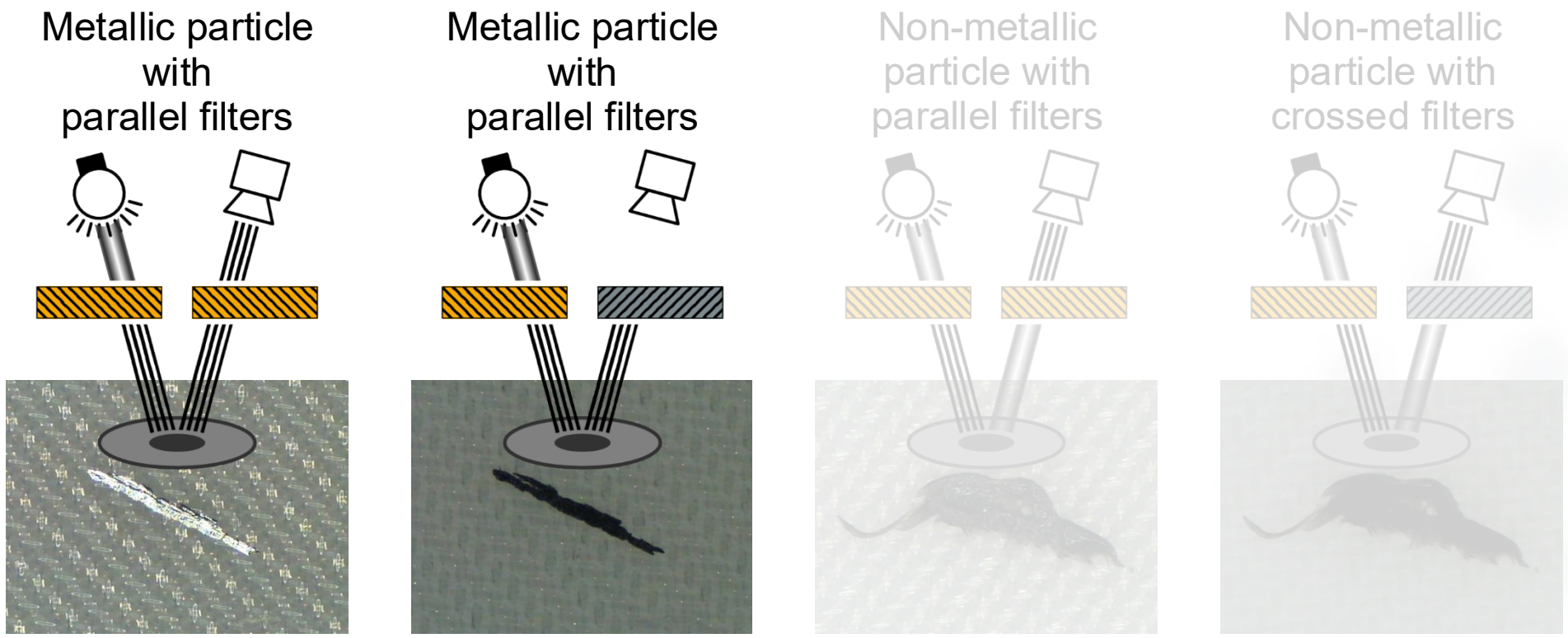
- The three “classes” of particles
  - metallic-shiny particles
  - not-metallic-shiny particles
  - Fibers



MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle typing

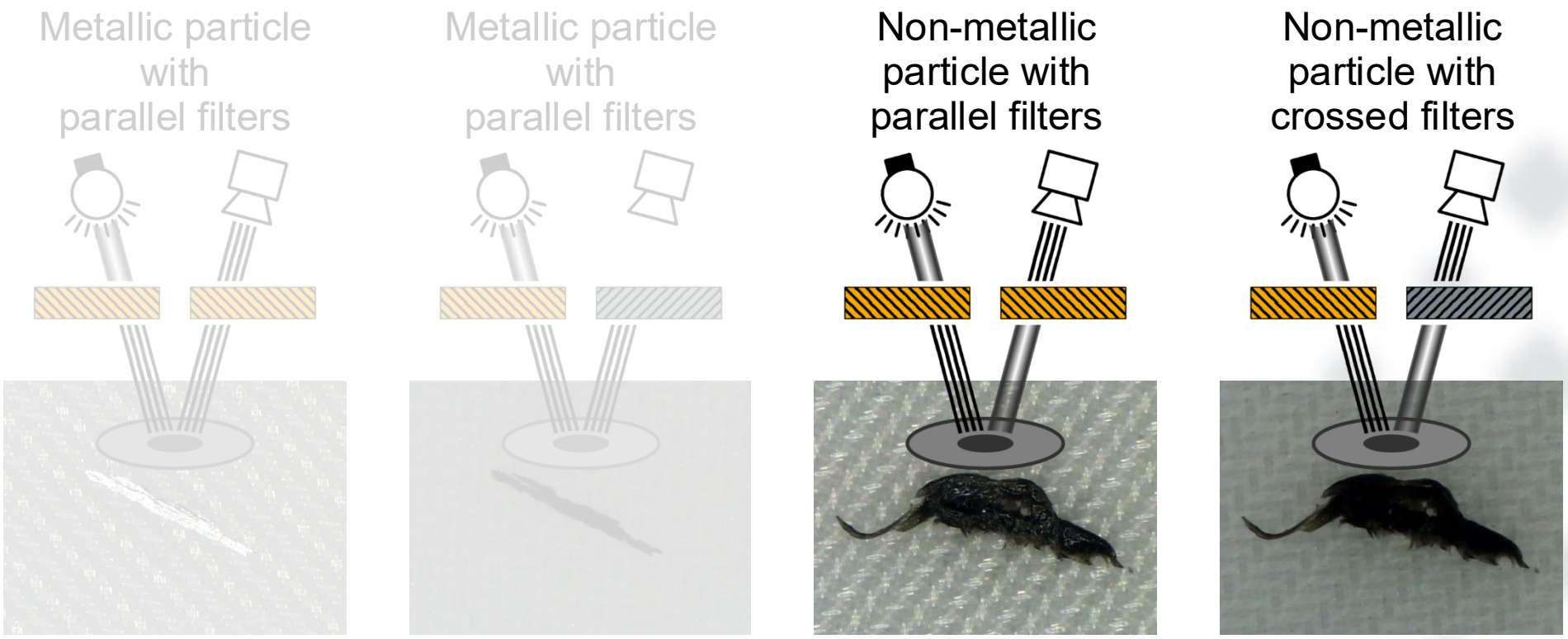
- How are these particle types distinguished?



MICROSCOPIC ANALYSIS IN VDA 19.1

Technical fundamentals – Particle typing

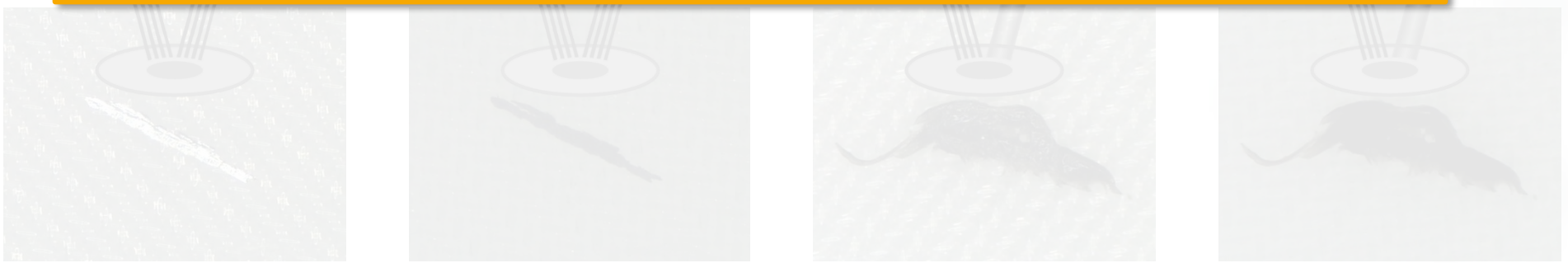
- How are these particle types distinguished?



Technical fundamentals – Particle typing

How are these particle types distinguished?

- Material classification is based on reflection behavior (shine) under polarized light
- No material characterization in a chemical sense → SEM/EDX required



MICROSCOPIC ANALYSIS IN VDA 19.1

Microscopy output & documentation

No	X-Pos	Y-Pos	Max. Diameter	Min. Diameter	Area	Circumference	Width (Torus)	Width (ortho.Cut)	Bord. Circ	Width (feret min)	Area. Circ	Stretched length	Assoc. Radius	Type
	[mm]	[mm]	[µm]	[µm]	[µm²]	[µm]	[µm]	[µm]	[µm]	[µm]	[µm]	[µm]	[µm]	
1	-7,46	20,29	817	11	13272	1954	244	33	15	236	13	977	65	FIB
2	16,35	-13,36	659	9	8773	1499	137	24	15	137	13	750	53	FIB
3	8,24	19,53	611	24	21419	1600	181	62	34	153	34	800	83	FIB
4	-11,43	-17,40	478	5	4790	975	47	15	9	47	9	488	39	FIB
5	3,50	21,51	389	9	5595	802	45	21	20	38	19	401	42	FIB
6	-7,37	1,92	374	196	52461	1094	218	218	190	218	170	578	129	NONMET
7	-1,39	16,07	353	199	59802	1372	328	290	167	294	138	723	138	NONMET
8	20,38	6,83	351	11	5349	742	41	27	15	33	13	371	41	FIB
9	-8,59	7,16	348	114	38383	857	144	143	125	142	123	433	111	NONMET
10	15,32	-8,53	332	129	34422	907	161	147	130	147	126	476	105	NONMET
11	4,29	21,38	321	20	9982	823	63	53	33	63	30	411	56	FIB
12	5,01	7,56	312	57	15980	750	114	106	65	102	63	375	71	MET
13	15,28	9,64	295	147	35093	778	180	180	147	175	131	391	106	NONMET
14	8,14	3,83	294	198	47873	881	219	206	189	218	189	441	123	NONMET
15	-12,27	17,76	289	9	3670	590	27	17	11	24	9	295	34	FIB
16	-15,89	0,76	289	137	33661	859	185	178	132	175	131	430	104	NONMET
17	7,39	17,32	282	144	31826	779	191	167	148	161	85	397	101	NONMET
18	3,12	21,48	269	9	4767	646	91	28	15	85	9	323	39	FIB
19	-6,74	20,15	261	91	25179	786	157	152	103	151	102	393	90	NONMET
20	16,36	-3,99	255	7	4163	531	33	30	24	33	21	266	36	NONMET
21	4,95	-14,84	251	21	6692	520	48	40	38	43	38	260	46	MET
22	5,01	18,46	245	33	14771	758	114	115	55	114	48	379	69	NONMET
23	11,07	15,45	243	33	11549	734	109	95	54	90	47	385	61	NONMET
24	18,85	-4,08	240	66	20859	693	138	115	85	118	85	346	81	NONMET
25	-12,20	12,34	240	100	20300	617	133	120	107	121	106	309	80	NONMET
26	17,58	6,53	237	102	19001	641	139	124	110	121	107	321	78	NONMET
27	15,83	2,64	236	11	3738	486	28	17	12	21	9	243	34	FIB
28	14,59	8,16	229	152	29968	672	181	174	152	175	151	336	98	MET

MICROSCOPIC ANALYSIS IN VDA 19.1

Microscopy output & documentation

Metallic shiny particles				
Particle size [µm]	Size class	Number of particles, all components	Number of particles per component	Total particles per 1000 cm²
15-25	C	5	5.0	5.0
25-50	D	6	6.0	6.0
50-100	E	3	3.0	3.0
100-150	F	1	1.0	1.0
150-200	G	0	0.0	0.0
200-400	H	1	1.0	1.0
400-600	I	0	0.0	0.0
600-1000	J	0	0.0	0.0
1000-1500	K	0	0.0	0.0
1500-2000	L	0	0.0	0.0
2000-3000	M	0	0.0	0.0
>3000	N	0	0.0	0.0

Non-shiny particles without fibers				
Particle size [µm]	Size class	Number of particles, all components	Number of particles per component	Total particles per 1000 cm²
15-25	C	221	221.0	221.0
25-50	D	113	113.0	113.0
50-100	E	31	31.0	31.0
100-150	F	6	6.0	6.0
150-200	G	1	1.0	1.0
200-400	H	0	0.0	0.0
400-600	I	0	0.0	0.0
600-1000	J	1	1.0	1.0
1000-1500	K	0	0.0	0.0
1500-2000	L	0	0.0	0.0
2000-3000	M	0	0.0	0.0
>3000	N	0	0.0	0.0

Fibers				
Particle size [µm]	Size class	Number of fibers, all components	Number of fibers per component	Total fibers per 1000 cm²
100-150	F	0	0.0	0.0
150-200	G	0	0.0	0.0
200-400	H	1	1.0	1.0
400-600	I	2	2.0	2.0
600-1000	J	2	2.0	2.0
1000-1500	K	2	2.0	2.0
1500-2000	L	0	0.0	0.0
2000-3000	M	0	0.0	0.0
>3000	N	0	0.0	0.0

Note: The stretched length is used as the criterion for typing fibers. These typed fibers are then assigned to the size classes according to the $Feret_{max}$ criterion.

Fibrous content		
Total of all stretched fiber lengths	7.30	mm
Longest fiber by stretched length	1670	µm

MICROSCOPIC ANALYSIS IN VDA 19.1

Microscopy output & documentation

Metallic shiny particles				
Reference: number of particles per 1000 cm²				
Particle size	**Limit value**	**Result**	**Assessment**
50-100 µm	8000.00	131.5	passed
100-150 µm	4000.00	111.0	passed
150-200 µm	2000.00	28.8	passed
200-400 µm	1000.00	45.2	passed
400-600 µm	8.00	4.1	passed
600-1000 µm	1.00	4.1	failed
1000-1500 µm	0	0.0	passed
1500-2000 µm	0	0.0	passed
2000-3000 µm	0	0.0	passed
>3000 µm	0	0.0	passed

All particles without fibers			
Reference: number of particles per 1000 cm²			
Particle size	**Limit value**	**Result**	**Assessment**
50-100 µm	64000.0	542.5	passed
100-150 µm	16000.0	143.9	passed
150-200 µm	8000.0	28.8	passed
200-400 µm	4000.0	45.2	passed
400-600 µm	500.0	4.1	passed
600-1000 µm	32.0	4.1	passed
1000-1500 µm	10.0	0.0	passed
1500-2000 µm	6.0	0.0	passed
2000-3000 µm	0.0	0.0	passed
>3000 µm	0.0	0.0	passed

MICROSCOPIC ANALYSIS IN VDA 19.1

- Gravimetric analysis
- Microscopy according to VDA 19.1 – general
- Technical fundamentals of microscopy
- Standard analysis
- **Free analysis**
- What light-optical particle analysis can do…
- … and what it can’t

MICROSCOPIC ANALYSIS IN VDA 19.1

Free analysis

- For particles with $Feret_{max} \leq 50$ µm
- Special questions
  → Particles with low contrast, morphology, height, **monitoring** etc.
- Illumination, brightness threshold and imaging method are **freely selectable**

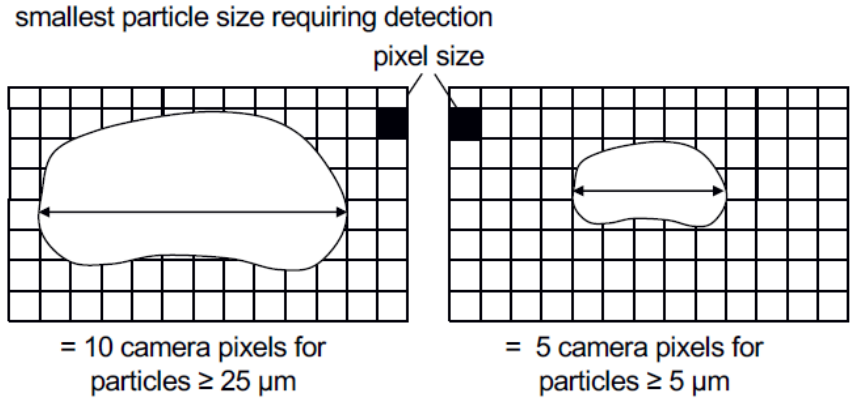


Freedom to choose testing methodology

MICROSCOPIC ANALYSIS IN VDA 19.1

Free analysis

- Higher demands towards the extraction procedure
  - Declining test, filtration, blank value
- Min. 5 pixels for the smallest particle size must be maintained
- Generally higher requirements on the microscopy setup

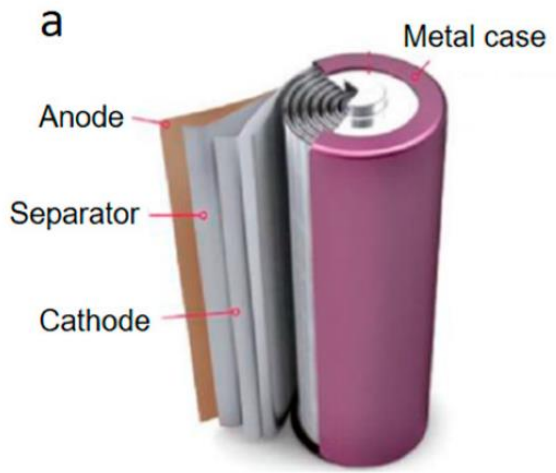


Particle Size	Detection at
≥ 50 µm	≤ 5.0 µm/pxl
≥ 25 µm	≤ 2.5 µm/pxl
≥ 15 µm	≤ 2.5 µm/pxl
≥ 5 µm	≤ 1.0 µm/pxl

MICROSCOPIC ANALYSIS IN VDA 19.1

Free analysis – an example: particle height

- Particle height is a critical dimension for contamination on battery separator foils
  - Requirements < 50 µm
  - Measurement with material microscope (shallow depth of field allows measurement along the z-axis)





MICROSCOPIC ANALYSIS IN VDA 19.1

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MICROSCOPIC ANALYSIS IN VDA 19.1

What light-optical particle analysis can do … and what it can't

Max. particle size (metallic shiny):

600 µm

additionally 5 particles up to 1000 µm permitted if length-to-width ratio > 2:1

additionally 2 particles up to 1500 µm permitted if length-to-width ratio > 3:1

additionally 1 particle up to 3000 µm permitted if length-to-width ratio > 5:1

Max. particle size (non-metallic shiny):

1000 µm

additionally 150 particles up to 3000 µm permitted if length-to-width ratio > 2:1

additionally 5 particles > 3000 µm permitted if length-to-width ratio > 2:1

```
Permitted residual dirt:
Max. dirt quantity:      0.4 mg/component
Max. particle size       hard particles:   max. 90µm x 500µm x  500µm
                         soft particles:   max. 90µm x  90µm x 2000µm
Additionally permitted: 10 soft particles max. 90µm x  90µm x 3000µm
```

Hard particles(*1) ≤ 600 µm x 90 µm Solf particles(*2) ≤ 2000 µm x 90 µm The maximum dirt quantity <0.05 mg	Hard particles ≤ 600 µm x 90 µm Solf particles ≤ 2000 µm x 90 µm The maximum dirt quantity <0.05 mg		1 times / Year		Cleanliness Test Laboratory

*1 Hard particles are e.g. ferrous and non-ferrous materials, hard elastomers (>90 Shore A),polymers, plastics, fibre-reinforced Teflon, polyamides.
*2 Soft particles are e.g. elastomers (<90 Shore A), paper, brush hairs, textiles.Abrasive particles such as quartz, sand, corundum, qlass etc. are not permitt

MICROSCOPIC ANALYSIS IN VDA 19.1

What light-optical particle analysis can do … and what it can't

Max. particle size (metallic shiny):

600 µm

additionally 5 particles up to 1000 µm permitted if length-to-width ratio > 2:1

additionally 2 particles up to 1500 µm permitted if length-to-width ratio > 3:1

additionally 1 particle up to 3000 µm permitted if length-to-width ratio > 5:1

Max. particle size (non-metallic shiny):

1000 µm

additionally 150 particles up to 3000 µm permitted if length-to-width ratio > 2:1

additionally 5 particles > 3000 µm permitted if length-to-width ratio > 2:1

```
Permitted residual dirt:
Max. dirt quantity:      0.4 mg/component
Max. particle size       hard particles:   max. 90µm x 500µm x  500µm
                         soft particles:   max. 90µm x  90µm x 2000µm
Additionally permitted: 10 soft particles max. 90µm x  90µm x 3000µm
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Hard particles(*1) ≤ 600 µm x 90 µm Solf particles(*2) ≤ 2000 µm x 90 µm The maximum dirt quantity <0.05 mg	Hard particles ≤ 600 µm x 90 µm Solf particles ≤ 2000 µm x 90 µm The maximum dirt quantity <0.05 mg		1 times / Year		Cleanliness Test Laboratory

*1 Hard particles are e.g. ferrous and non-ferrous materials, hard elastomers (>90 Shore A),polymers, plastics, fibre-reinforced Teflon, polyamides.
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MICROSCOPIC ANALYSIS IN VDA 19.1

What light-optical particle analysis can do … and what it can't

	#1 (Mica particles)	#2 (SiC particles)
Bright image		
Image with crossed polarizer		
Comment	The non-metallic particles above appear somewhat shiny in the bright image and are jet black in the image with crossed polarizers. They are therefore very likely to be identified as metallic-shiny in automatic analysis, but they are of mineral origin.	

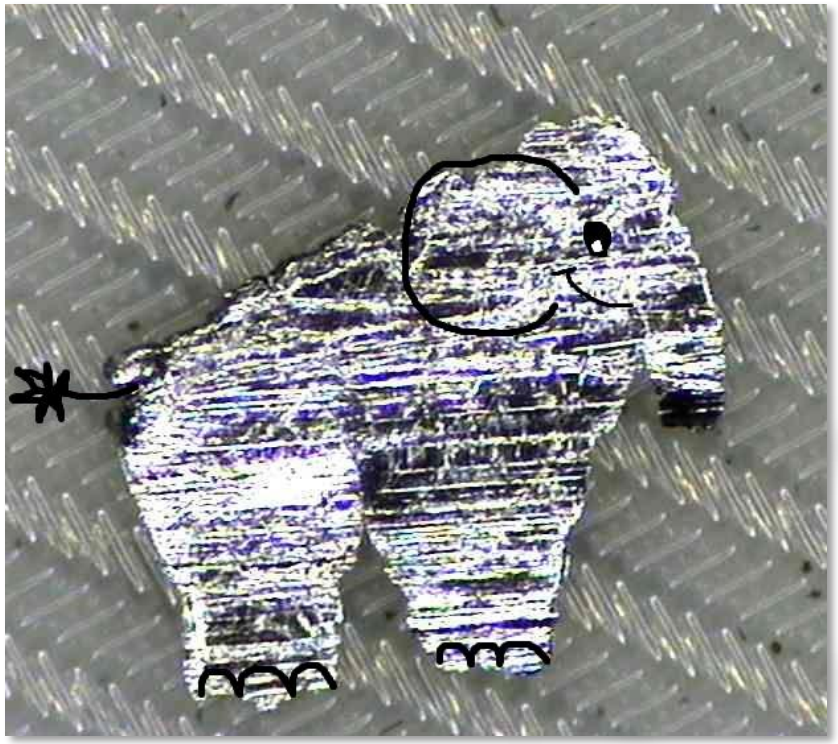


To be continued tomorrow …

MICROSCOPIC ANALYSIS IN VDA 19.1

Despite all standardization: ***the microscopist makes the difference!***





QUESTIONS

Any questions about the talk?

Chat

Raise hand

E-Mail

MICROSCOPIC ANALYSIS IN VDA 19.1

Thank you for your attention



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