

OPTIMIZING FRACTIONATION: QUALITY CONTROL AND ANALYSIS OF BLACK MASS

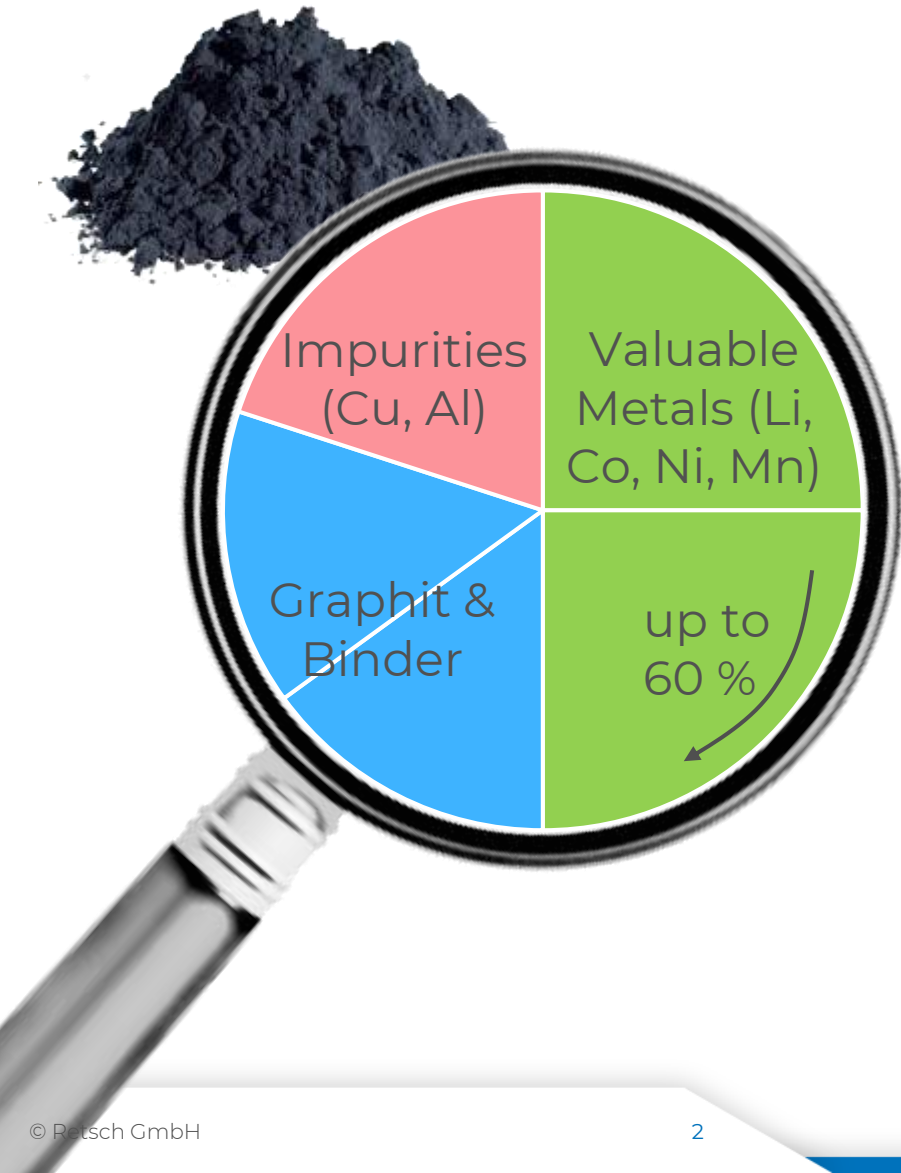


Dr. Markus Kirschner



Dr. Lena Weigold





- **significant variation in composition**
- **inhomogenities** -> **reduces value**
 -> **difficult to analyze**

WHY DOES QUALITY VARY?

¹ Wilke, C et al. *Batteries* **2023**, 9, 514

- different battery chemistries
- non-selective crushing & incomplete separation



Particle size-dependent composition¹

Coarse

... more cathode material



> 500 µm

Fine

... more graphite



< 300 µm

Ultra fine

... few impurities



< 64 µm



Sieving machine
AS 200 control



Sieve Shaker

> 9 Laboratory Sieving Machines



Cutting Mills



Rotor Mills



Disk Mills



Ball Mills

> 35 Laboratory Mills

CRUSHING OF BATTERIES



Cutting Mill
SM 300

**find this machine
at booth E45**

CRUSHING OF BATTERIES



Cutting Mill
SM 300

**find this machine
at booth E45**

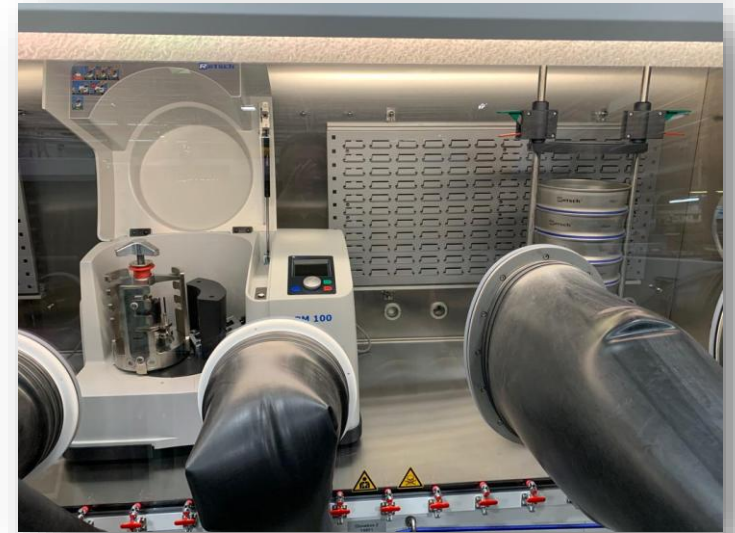




SAFETY MEASURES



- risk of explosion
- health risk



VIBRATORY SIEVING



Sieving machine
AS 200 control
**find this machine
at booth E45**



> 50 mm

housing parts (e.g. long
sheet pieces)

50 - 0.5 mm

mixtures of metal and
plastic particles

0.2–0.5 mm

metal particles

< 0.2 mm

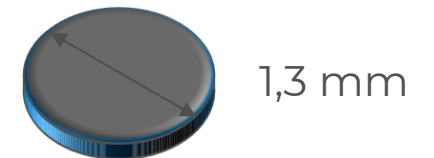
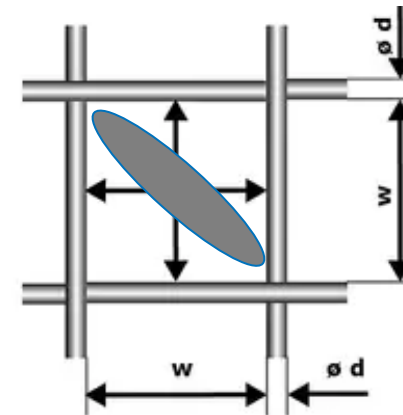
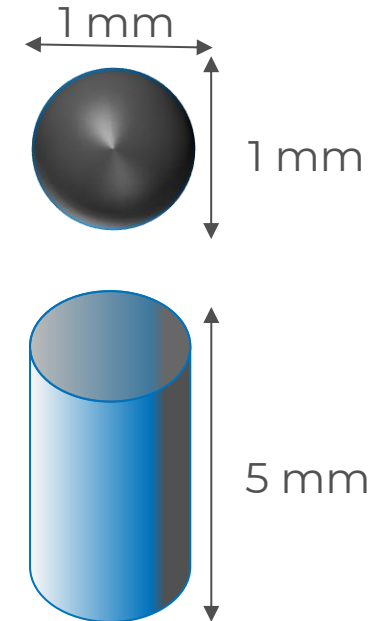
black mass



VIBRATORY SIEVING



ISO 3310
ASTM E11

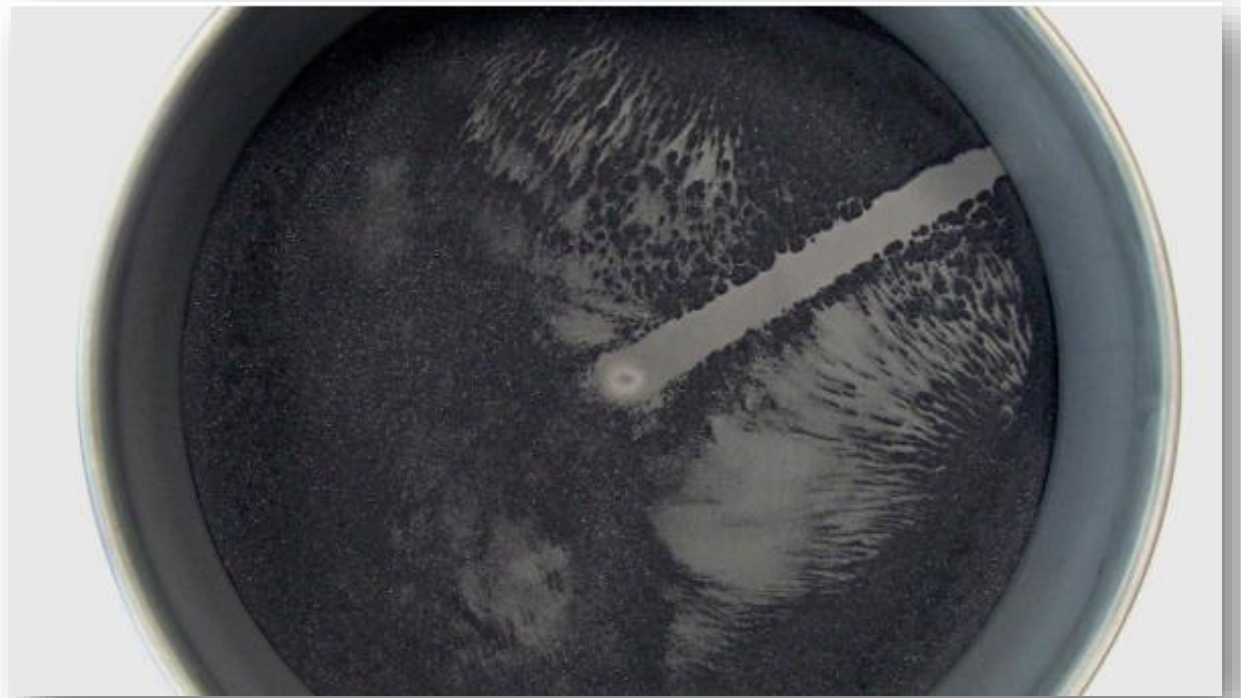




Sieving machine
AS 200 jet pro



Sieving machine
AS 200 jet pro



MILLING AND SIEVING FOR FRACTIONATION

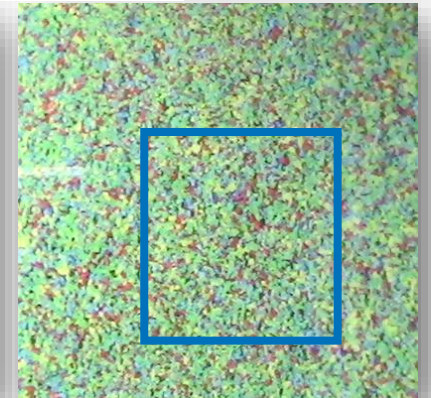
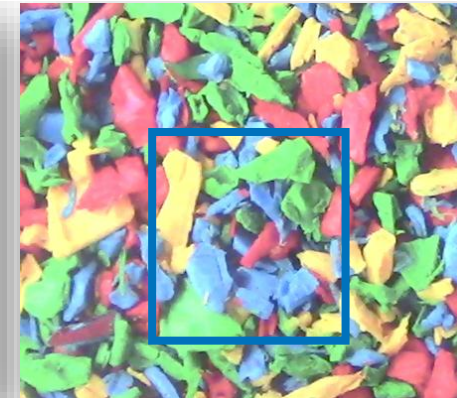
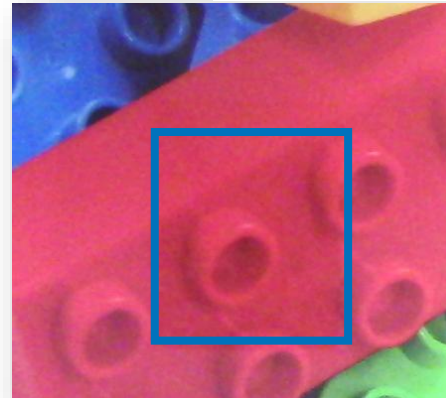


Preparing representative
samples for analysis



Grinding
Homogenization
Sample dividing

reproducible results -
conform to analysis



Preparing representative
samples for analysis



Grinding
Homogenization
Sample dividing

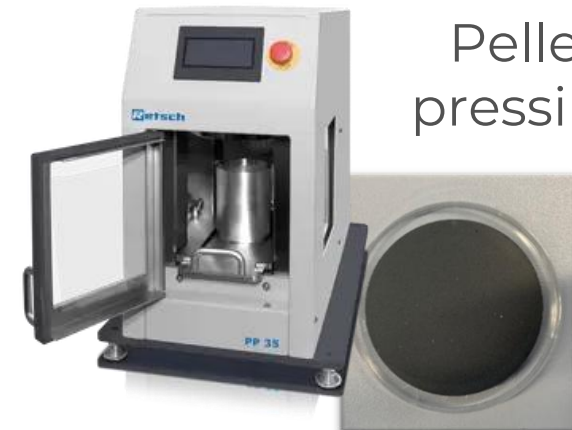
reproducible results -
conform to analysis



Sample
division



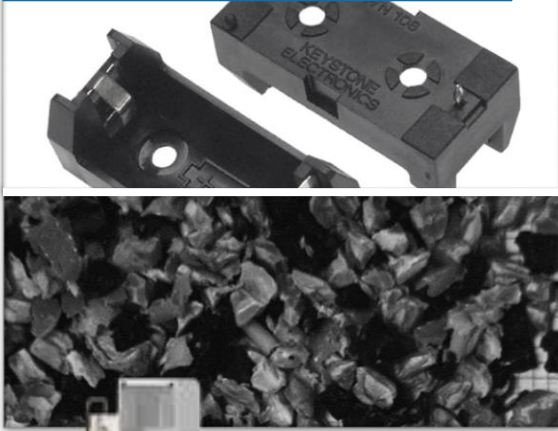
Homogenization



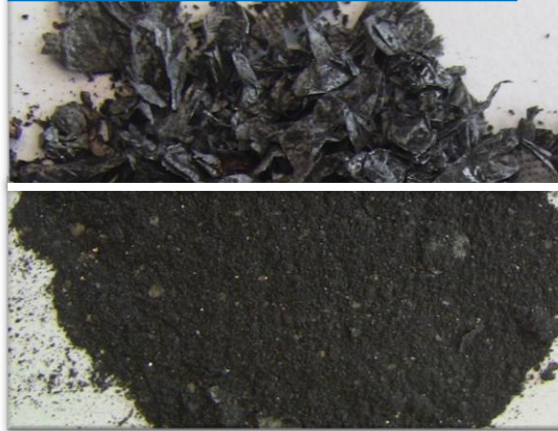
Pellet
pressing

CRYOGENIC GRINDING

Housing parts



Polymeric foil



Metal foil



SM 300



CryoMill



MM 500 control

Preparing representative
samples for analysis



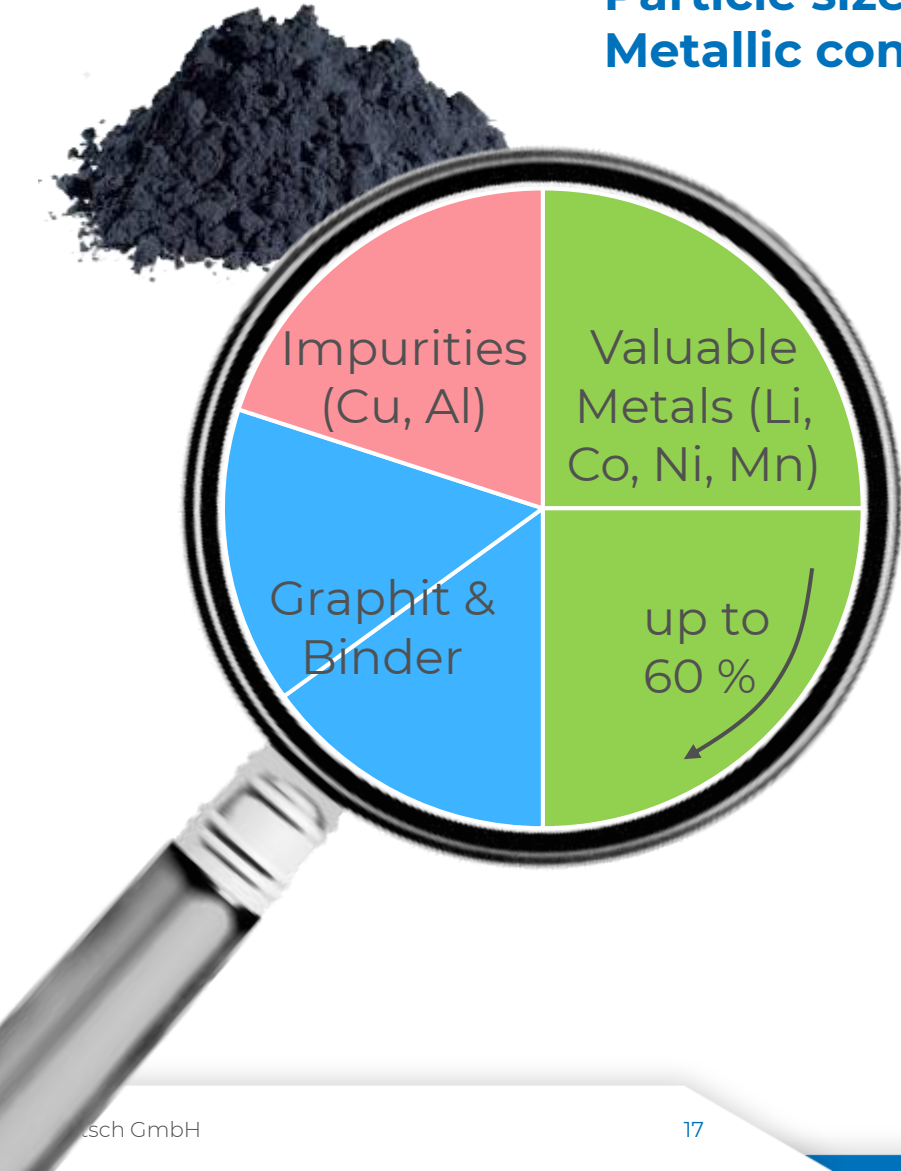
**Grinding
Homogenization
Sample dividing**

reproducible results -
conform to analysis



CHALLENGING THE HOMOGENIZATION OF BLACK MASS

Particle size-dependent composition
Metallic components



MICROTRAC

a **VERDER** company

Porosity & Catalysis



Surface Area
& Pore Size



Catalysis



Density

Size & Shape



Particle size



Particle
shape

Stability & Shelf Life



Particle size



Zeta potential

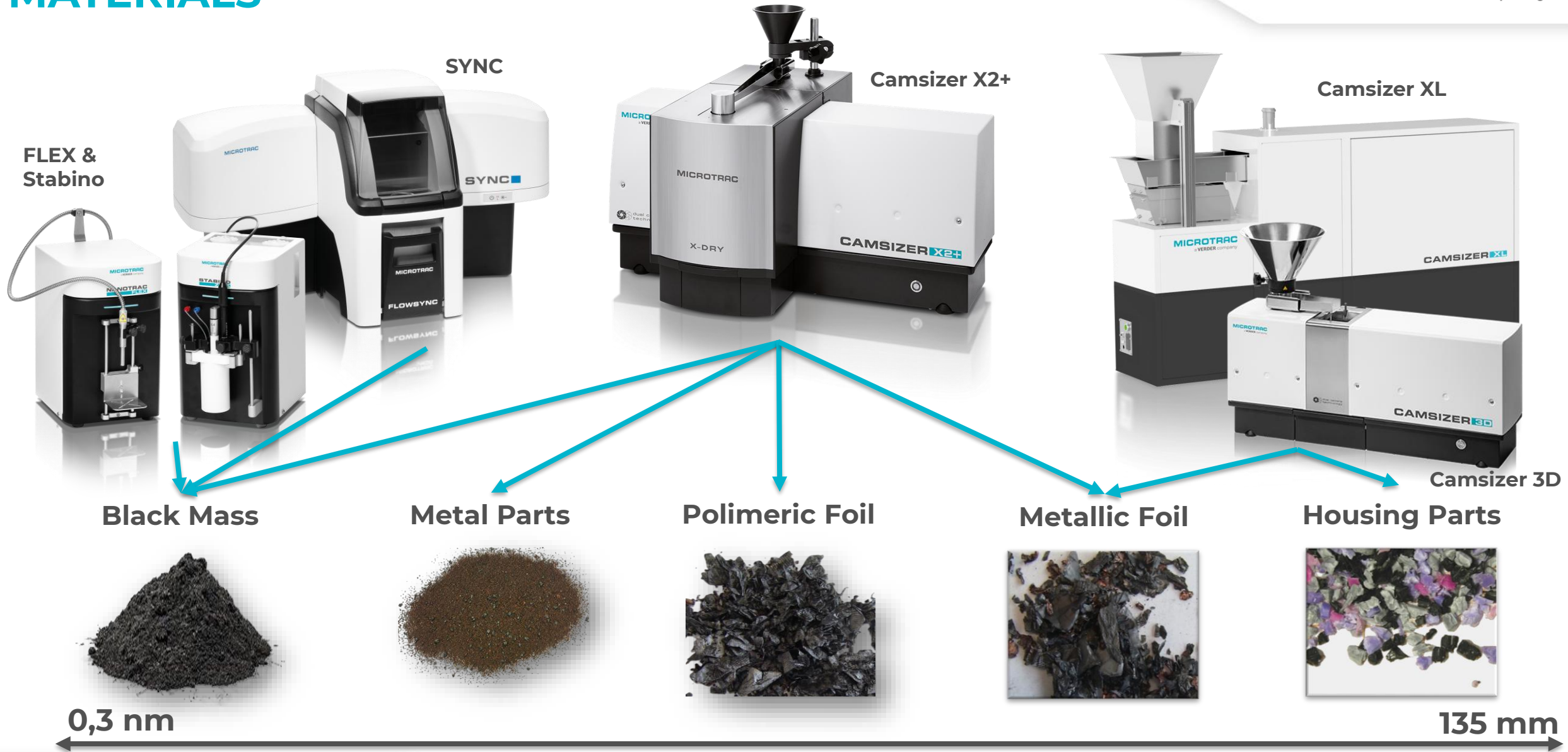


Stability &
Durability

Your strong partner for the comprehensive
characterization of **particulate systems** and **formulations**

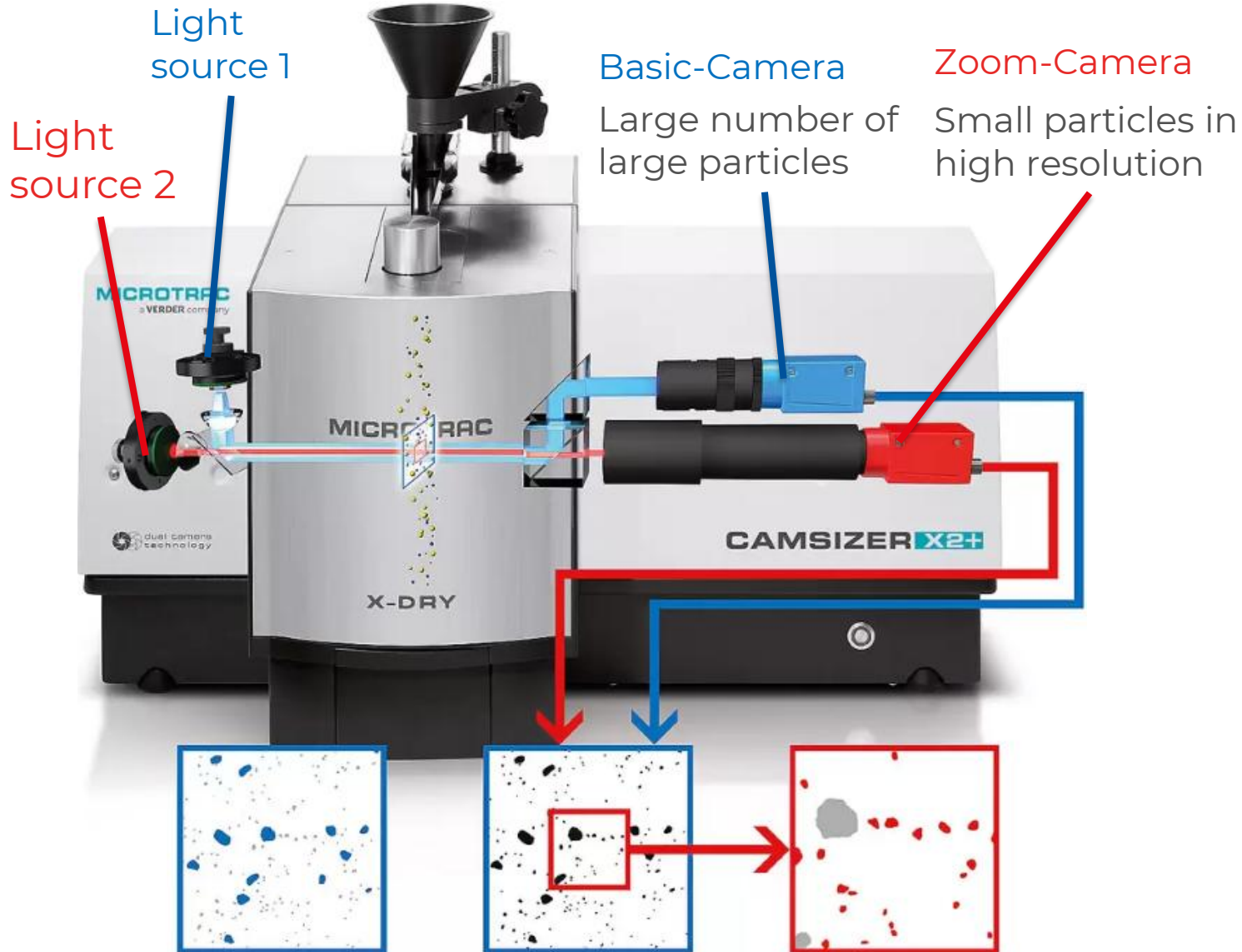
QUALITY CONTROL OF RECYCLES BATTERY MATERIALS

MICROTRAC
a VERDER company



CAMSIZER X2+ - HOW IT WORKS

MICROTRAC
a **VERDER** company

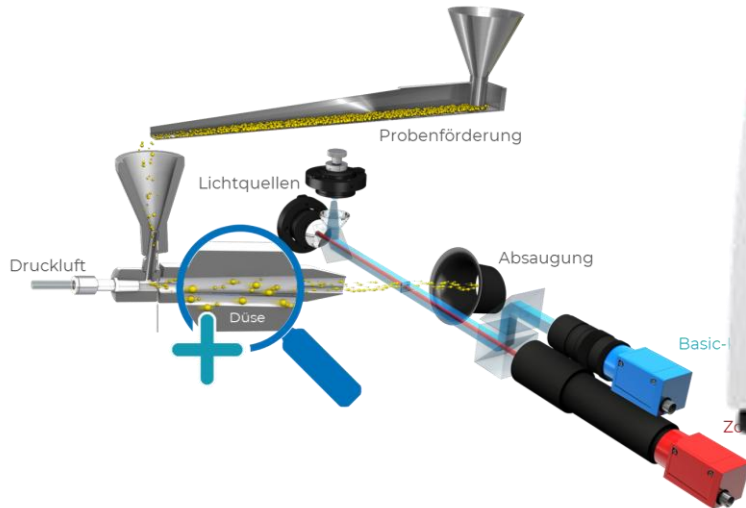


- Dynamic image analysis with **patented 2-camera system** (ISO 13322-2)
- Dry measurement with almost **no sample preparation**
- **Automatically controlled sample feeding** with integrated particle dispersion
- Particle size and shape analysis from **0.9 μm to 8 mm**
- Measurement times of only **1 to 5 min**
- Real-time evaluation of more than **400 frames per second**
- **Modular concept** with quick change between modules
- Reliable detection of **oversized and undersized particles**

 dual camera
technology

CAMSIZER X2+ MODULAR DESIGN

X-Jet



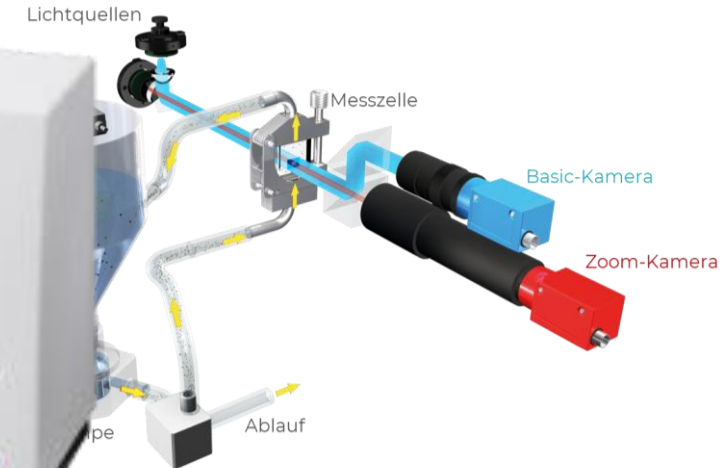
- For fine powders and agglomerating materials
- Dry dispersion by compressed air: pressure from 0.0 bar (vacuum only) to 4.5 bar

X-Fall



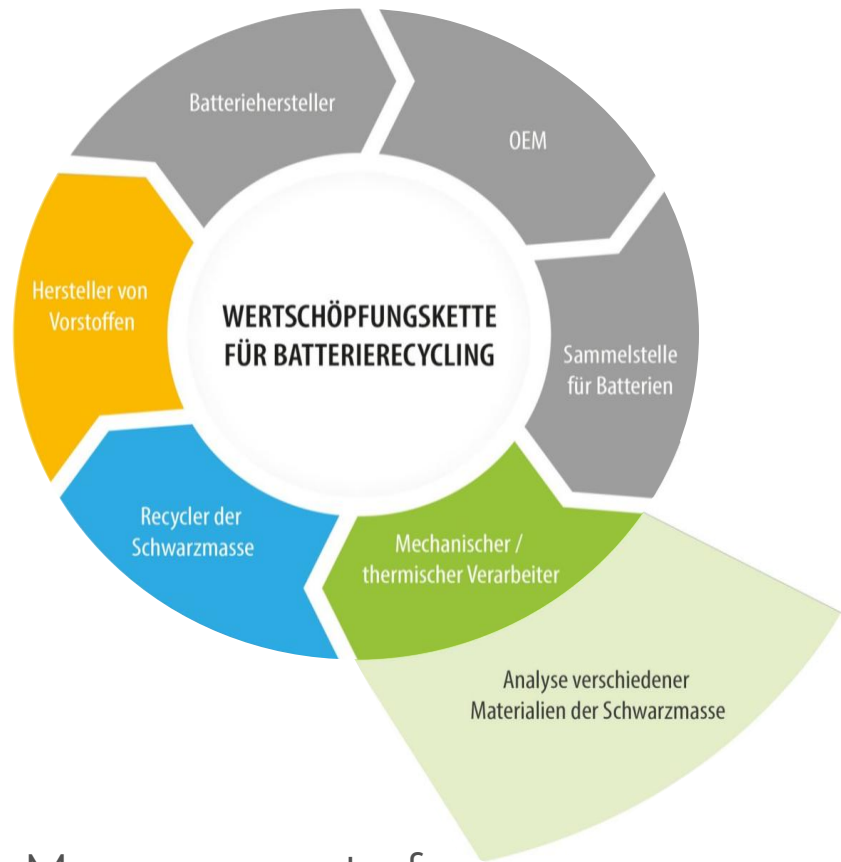
- Sample recovery after analysis

X-Flow



- Sample recovery after analysis
- Ideal for a wide range of solvents





Measurement of:

- Black mass
- Metal powder (Cu and Cu/Al)
- Shredded aluminum foils

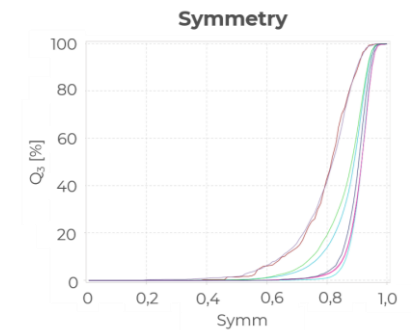
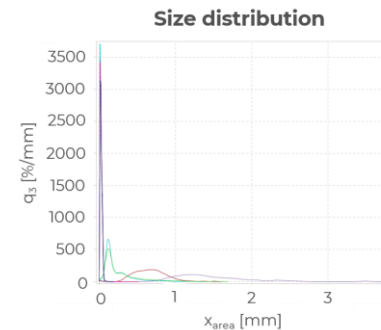
Black mass



Metal powder



Foils

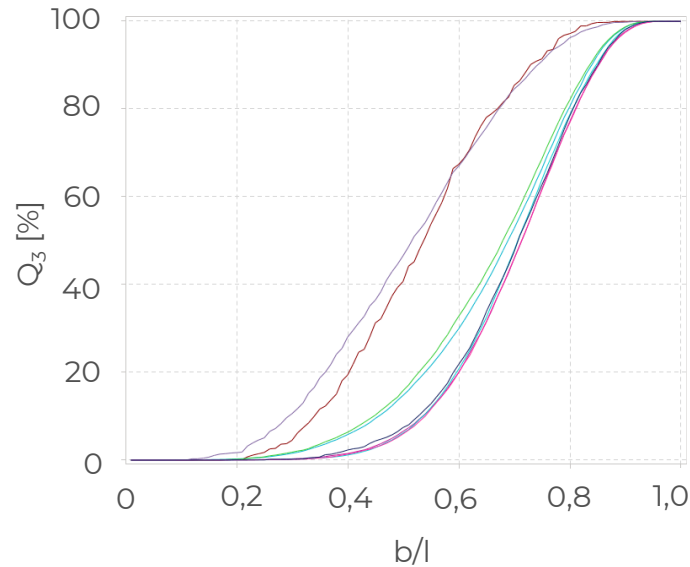


Presentation of results from a variety of measurements from different manufacturers and treatment methods.

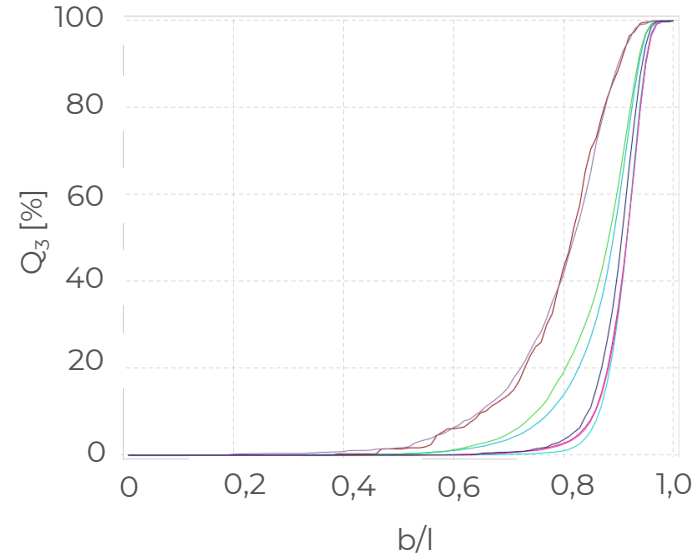
Particle size and particle shape distribution of the analyzed samples shown as $Q_3(x)$.

The particle size is based on the parameters X_{area} (spherical equivalent), the particle shape is based on the parameters $Symm$ (symmetry)

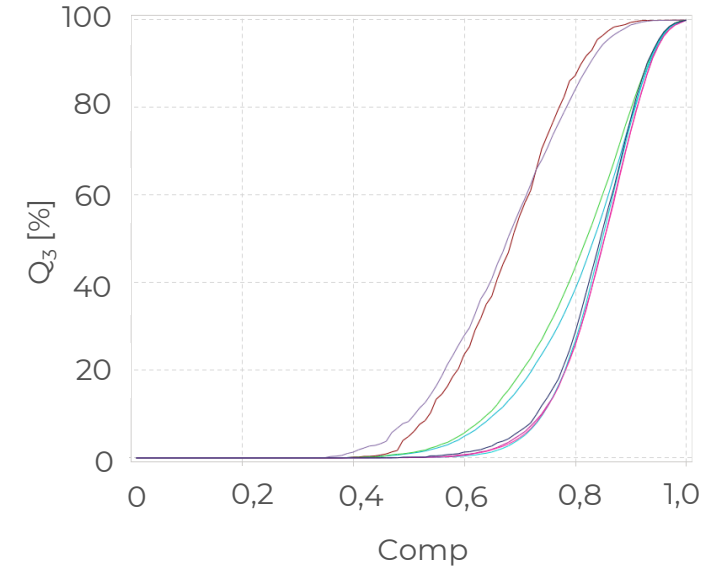
Aspect Ratio



Symmetry



Compactness

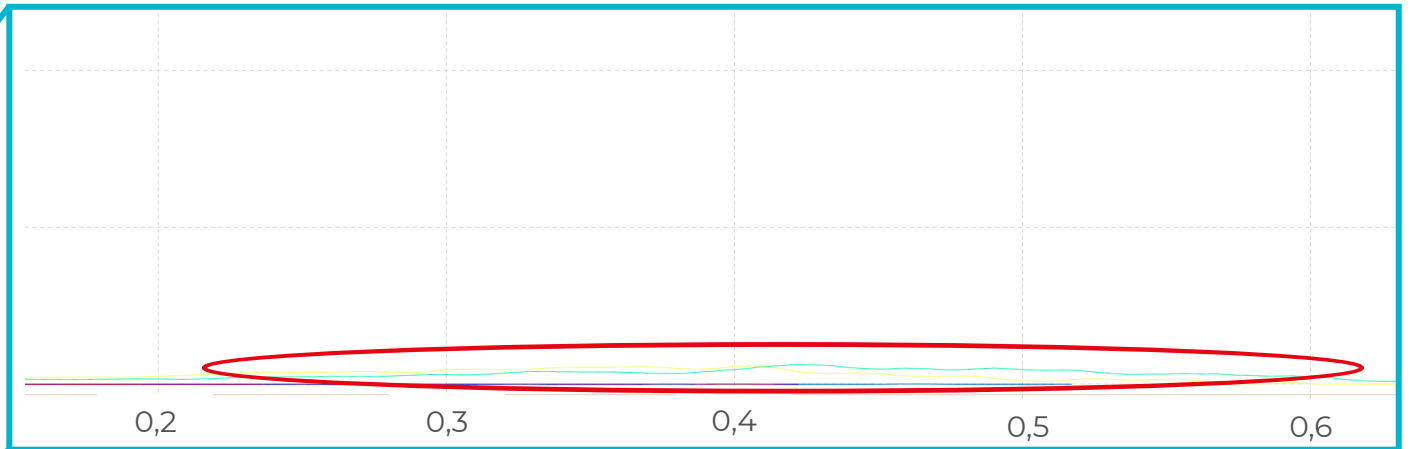
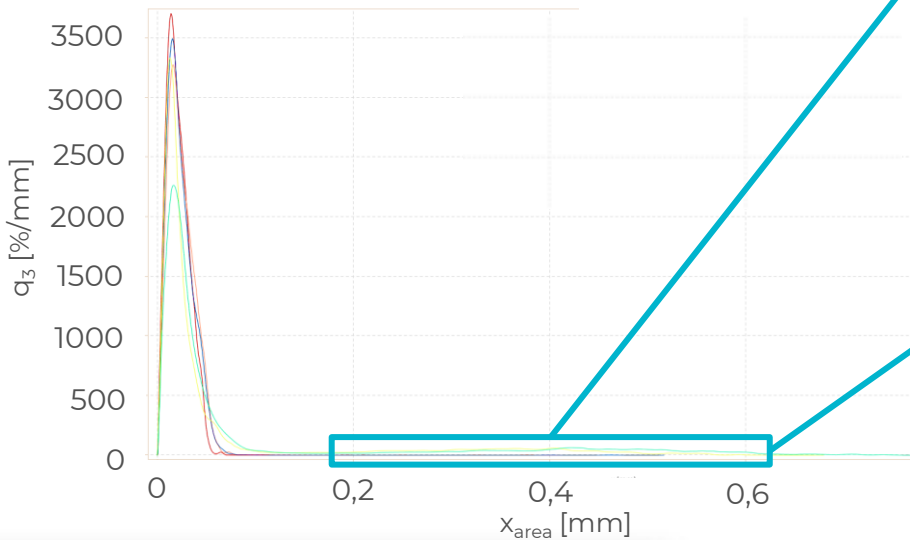
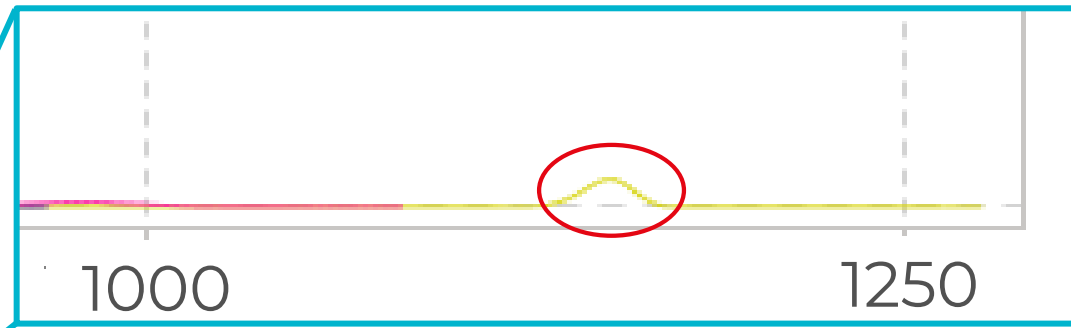
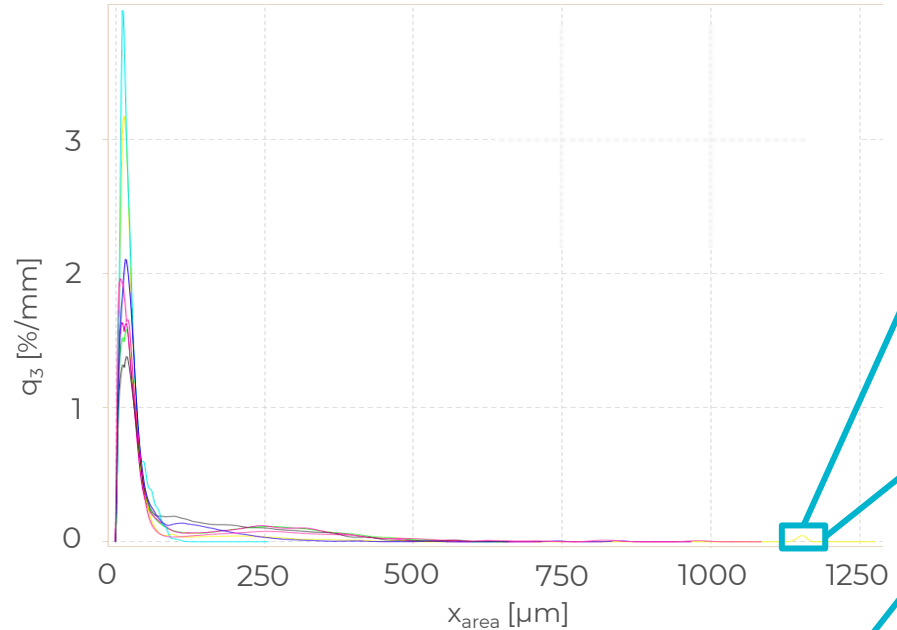


The **symmetry** of the particle shape is a **robust indicator of the mechanical state of the particles** in black mass and **reacts directly to fracture, delamination and re-agglomeration**.

In contrast, aspect ratio and compactness primarily describe the outer silhouette and are more strongly influenced by orientation, projection and segmentation.

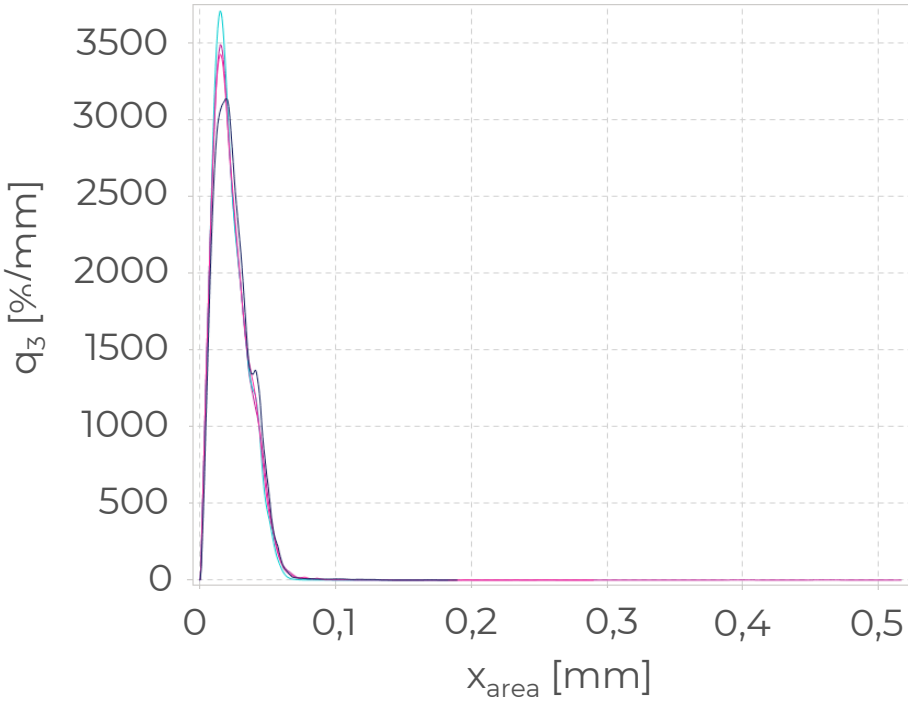
Symmetry is direction-independent and therefore better suited to reliably detect process-induced structural changes.

OVERSIZE DETECTION BLACK MASS



Since individual particles are detected in dynamic image analysis, the detection limit is significantly lower than in laser diffraction. The **detection limit of oversize** is **about 0.5 %** for **dynamic image analysis**, while it is about 2 % for laser diffraction.

Size distribution



Black mass 1

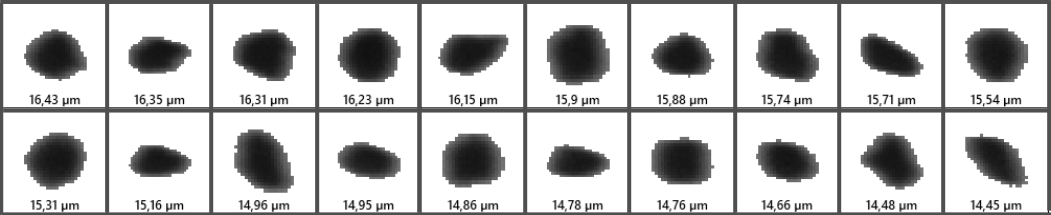
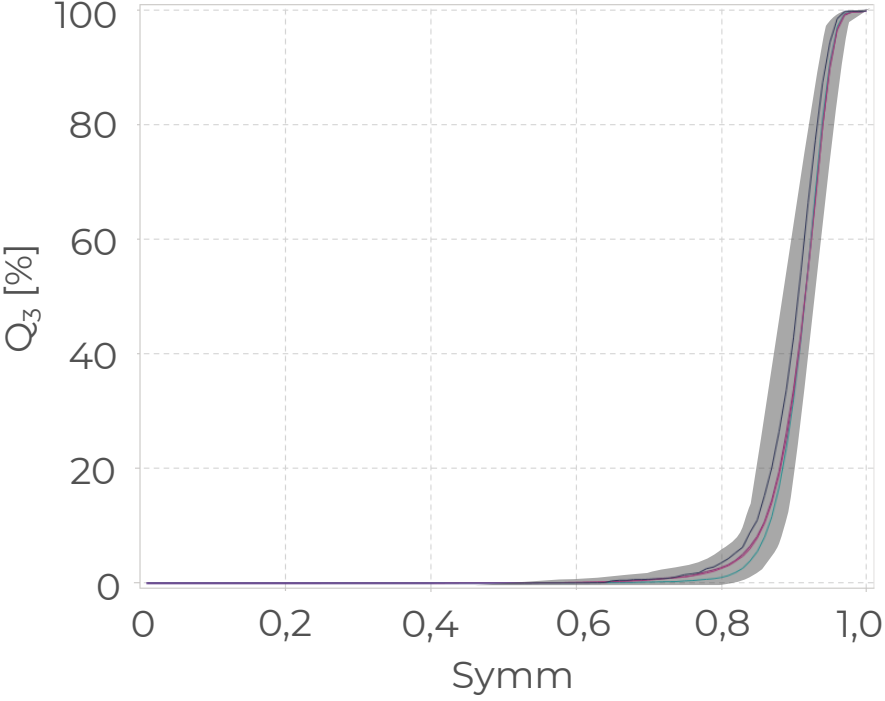


Abb1: Particle images of the test sample Black mass sample 1 (selection)

Symmetry



Black mass 2

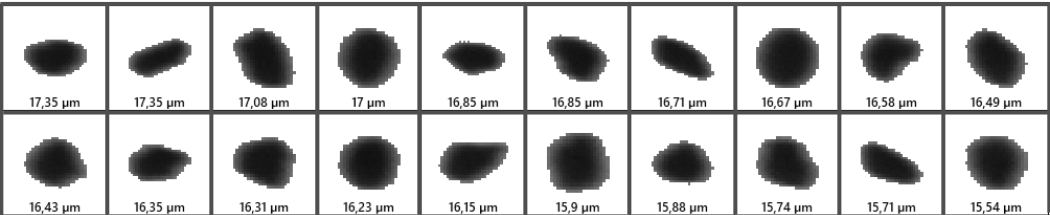
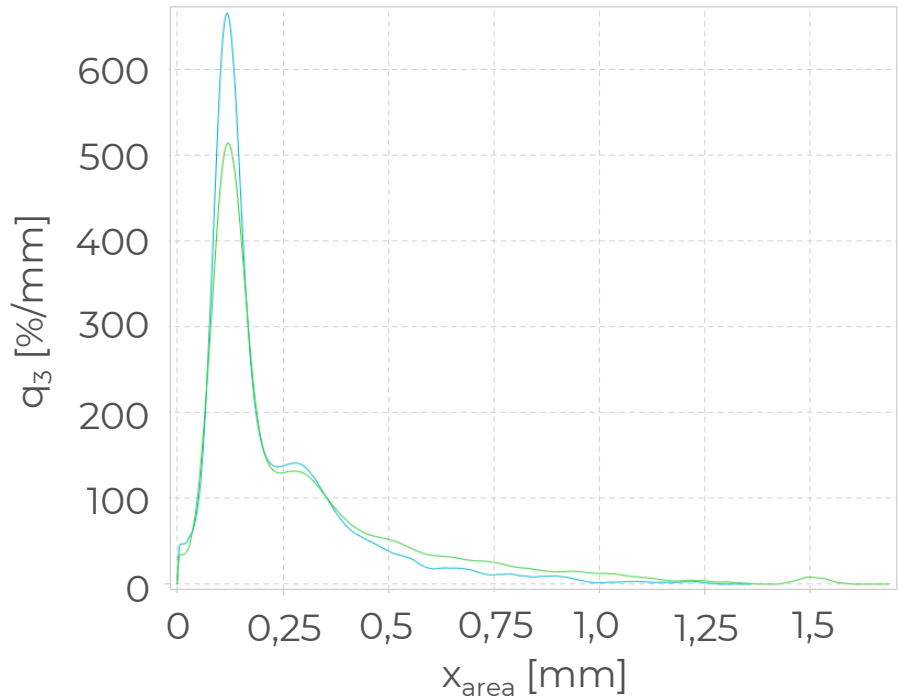


Abb2: Particle images of the test sample Black mass sample 2 (selection)

Size distribution



Metal powder 1

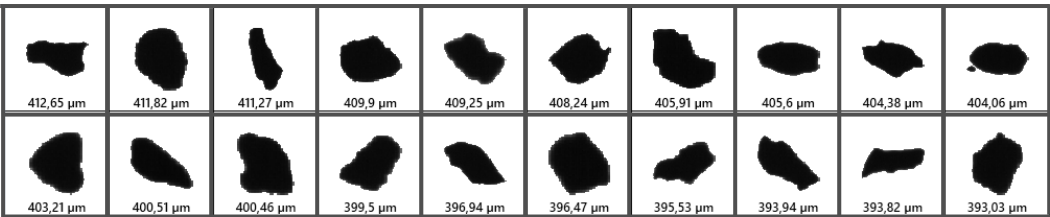
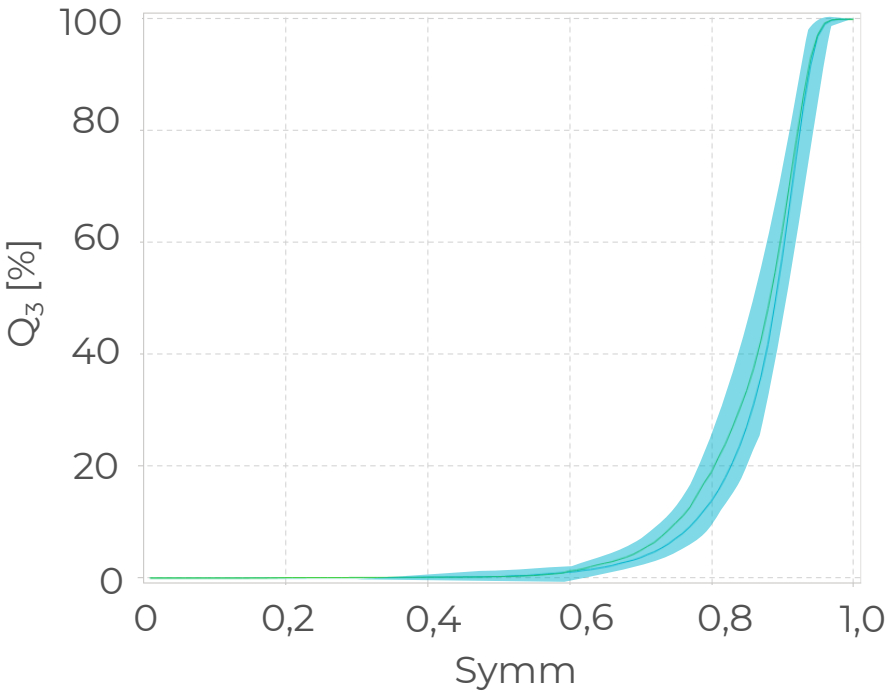


Abb3: Particle images of the test sample metal powder sample 1 (selection)

Symmetry



Metal powder 2

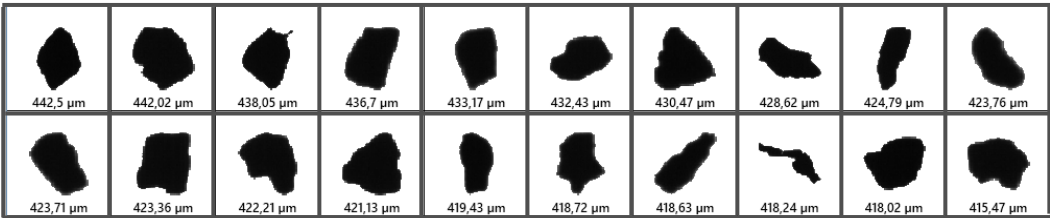
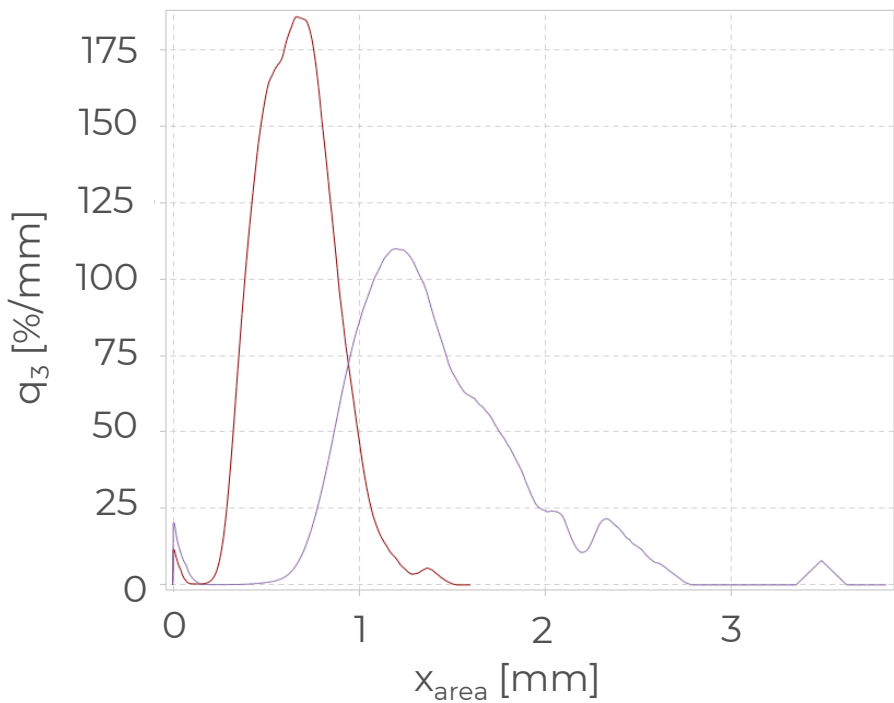


Abb4: Particle images of the test sample metal powder sample 2 (selection)

Size distribution



Foil 1

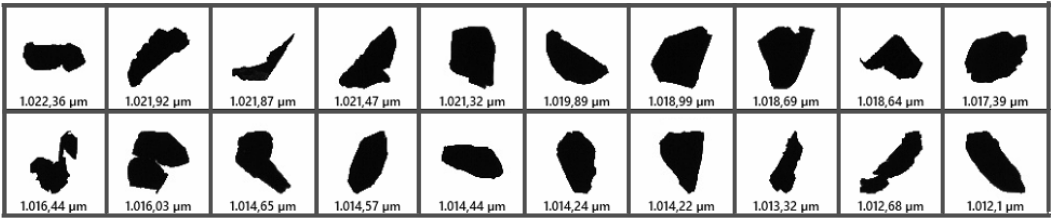
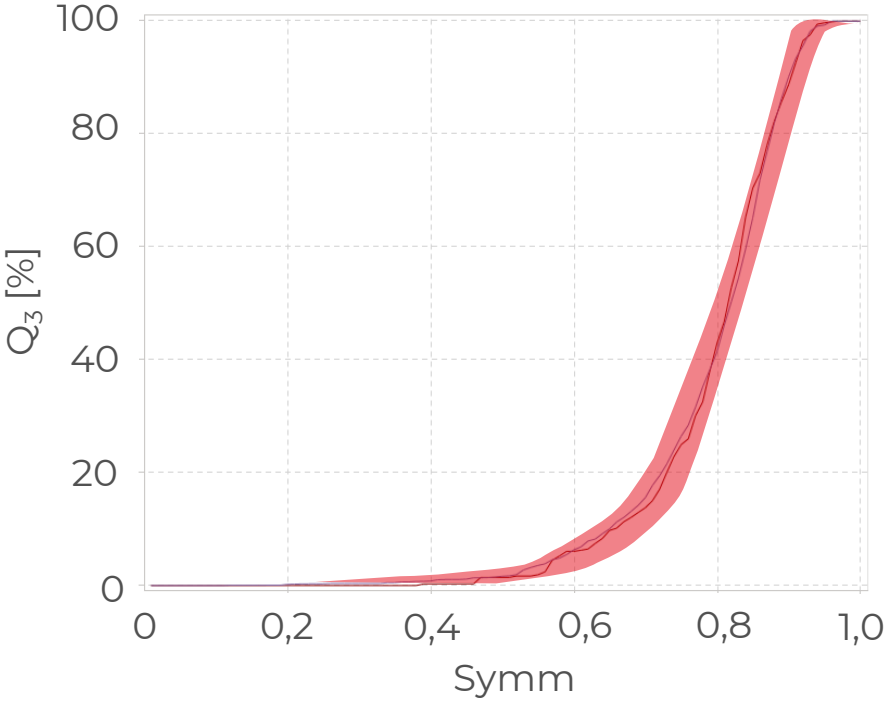


Abb5: Particle images of the test sample foil sample 1 (selection)

Symmetry



Foil 2

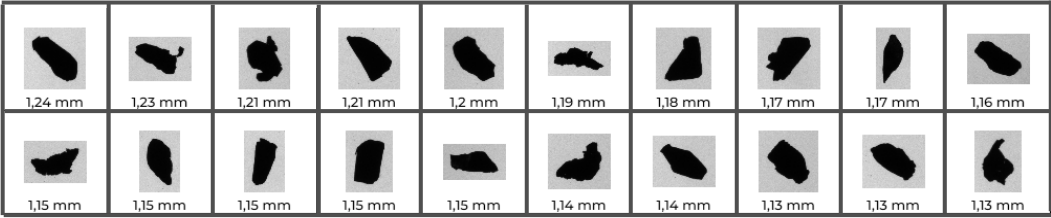
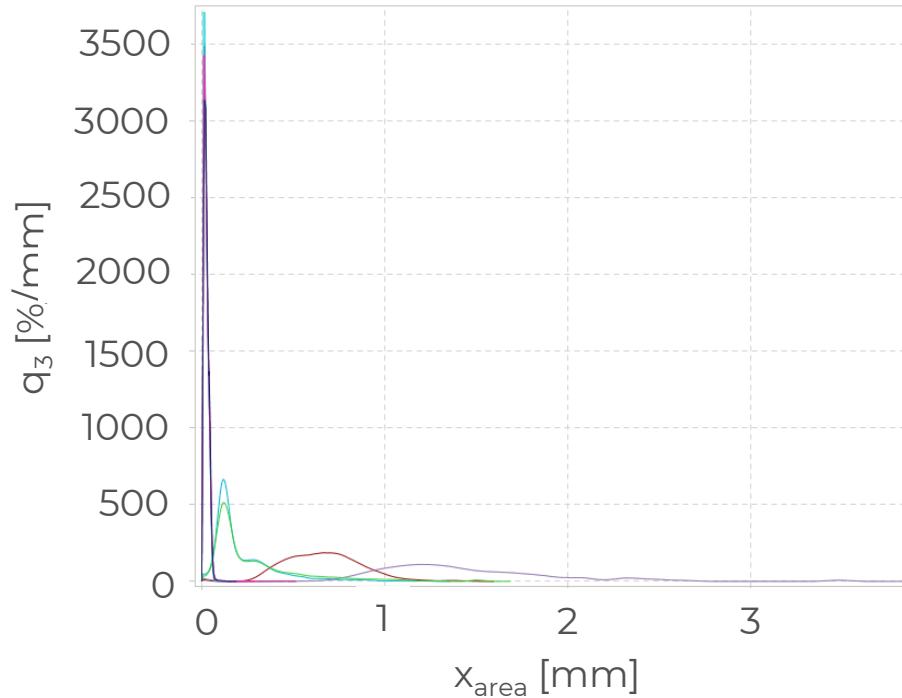
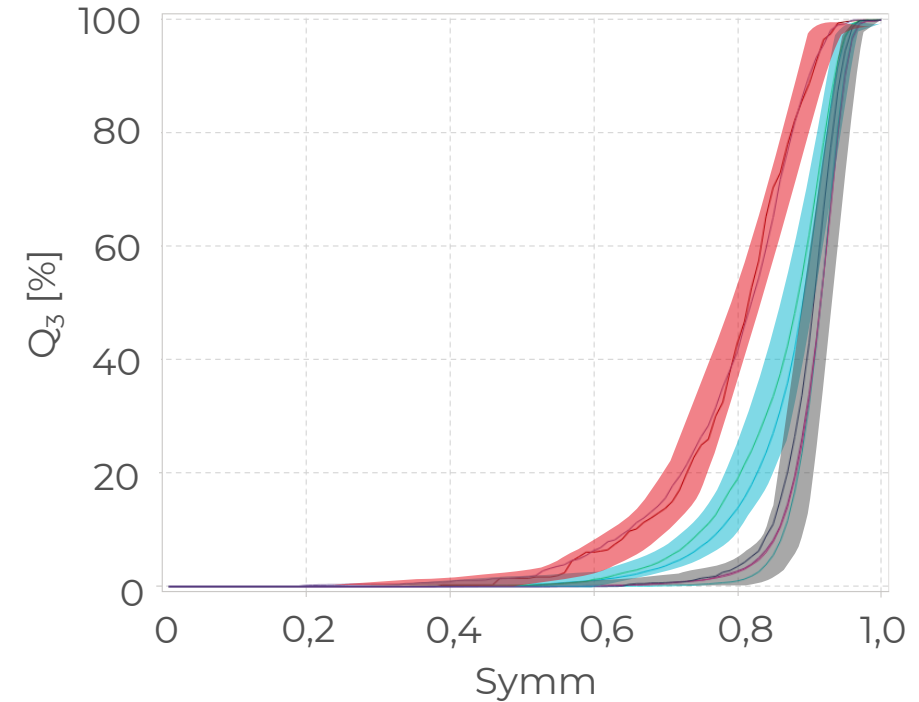


Abb6: Particle images of the test sample foil sample 2 (selection)

Size distribution



Symmetry



- All materials could be measured without any problems by means of compressed air dispersion. The **2-camera optics** of the **Camsizer X2+** enable reliable analysis of both **fine materials (black mass and metal powder)** and **coarser particles (aluminum foils)** – even with **wide particle size distributions**.
- Typical measurement times are **2–5 minutes**, depending on the desired statistics.
- Use & Benefits:
 - Reliable detection of oversize particles
 - Clear differentiation between black mass, foil and metal powder through defined measurement corridors



VERDER



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**Thank you
for your attention!**

