Additive Manufacturing: Effective Control of Metal Powder Quality

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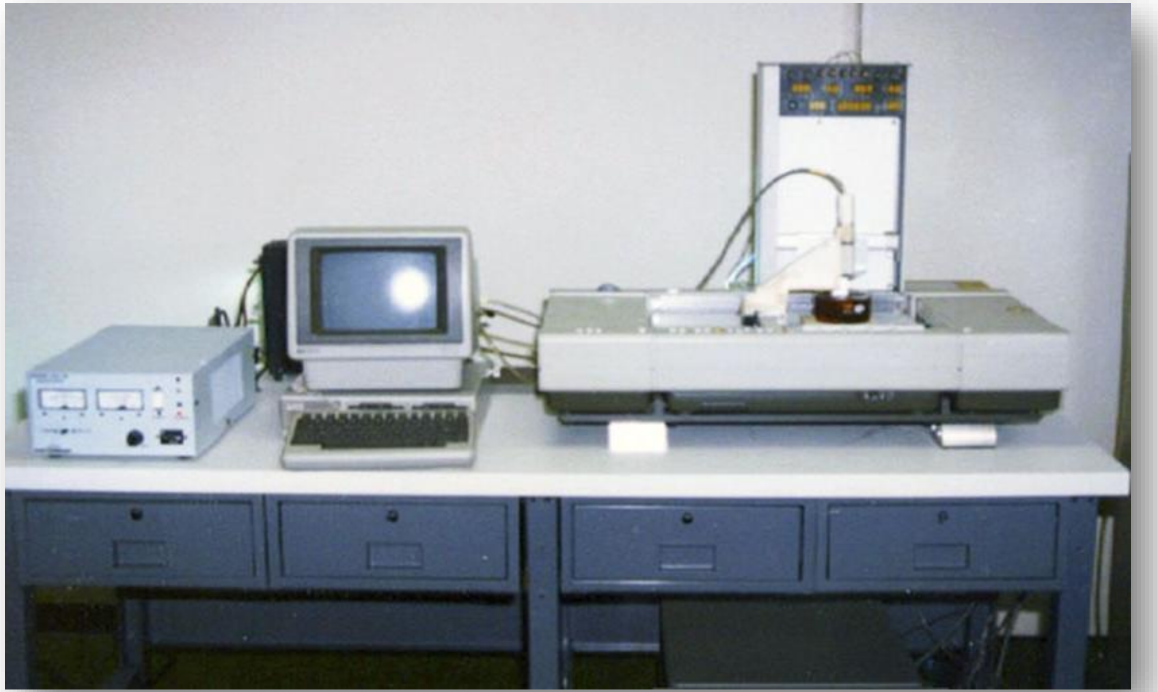
3D Printing – History

- 1980: First patent by Japanese Dr. Kodama Rapid prototyping (**photosensitive resin was polymerized by an UV light**)
- 1986: The first patent by 3D system corporation
- 1988: First SLA-1 machine
- 1990: First EOS Stereos system
- 1997: The first commercial EBM platform by Arcam
- 2000s: The beginning 3D printings of popularization
- 2000: a 3D printed working kidney is created (transplanted in 2013)



The SLA 1

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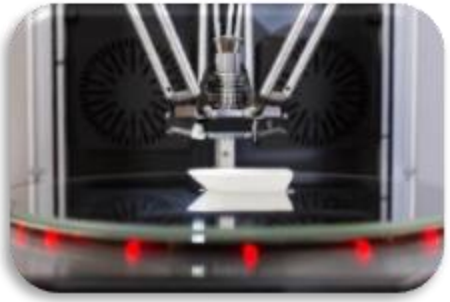
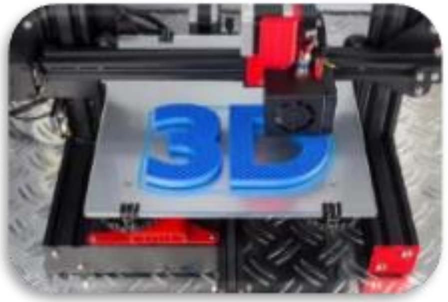
Additive Manufacturing – Applications

- Automotive
- Aerospace
- Defense
- Medical & Dental
- Implants
- Complex Industrial components
- Prototypes
- Consumer goods





- Missing items for your action figures

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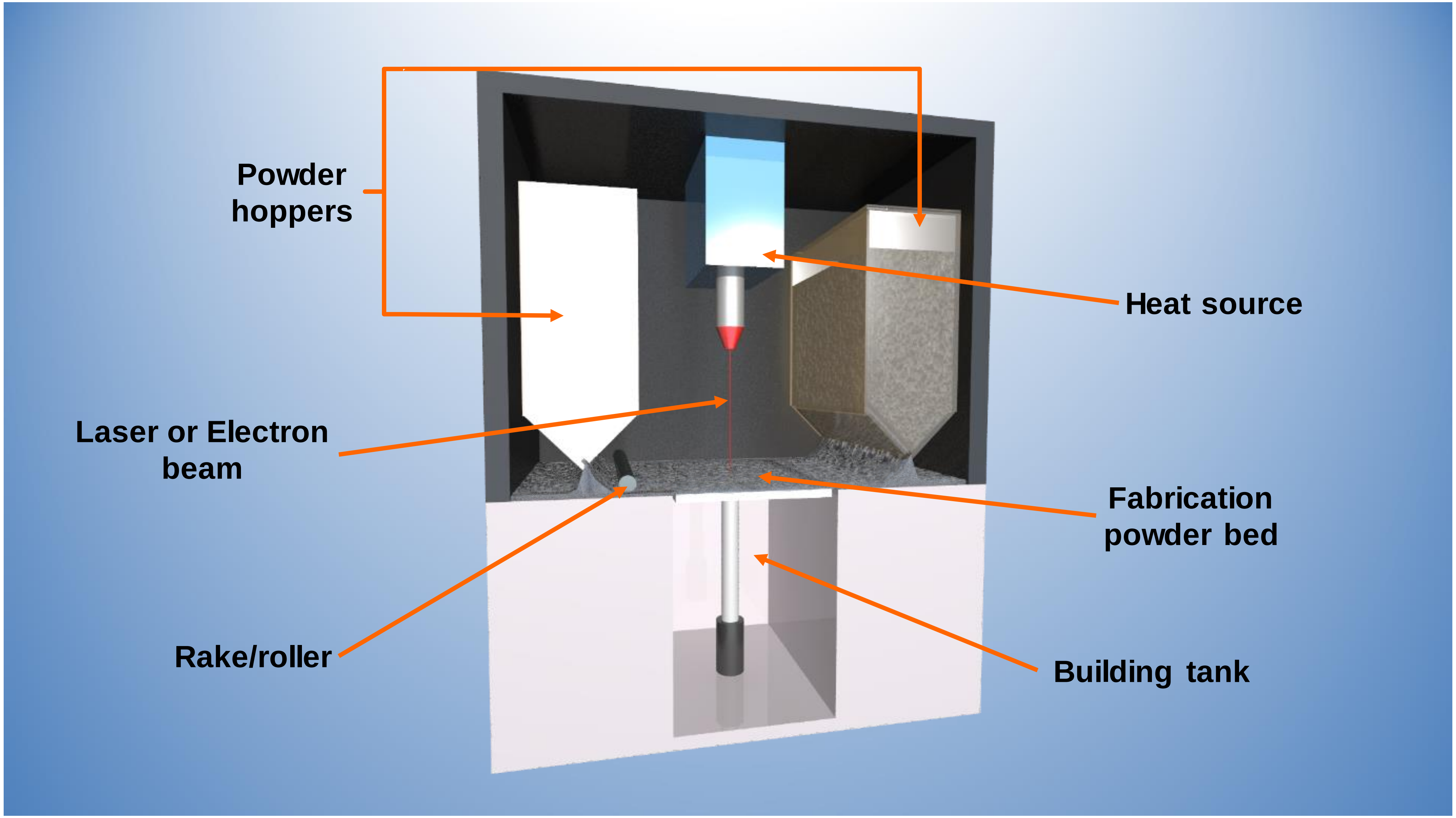
General Instrument Schematic



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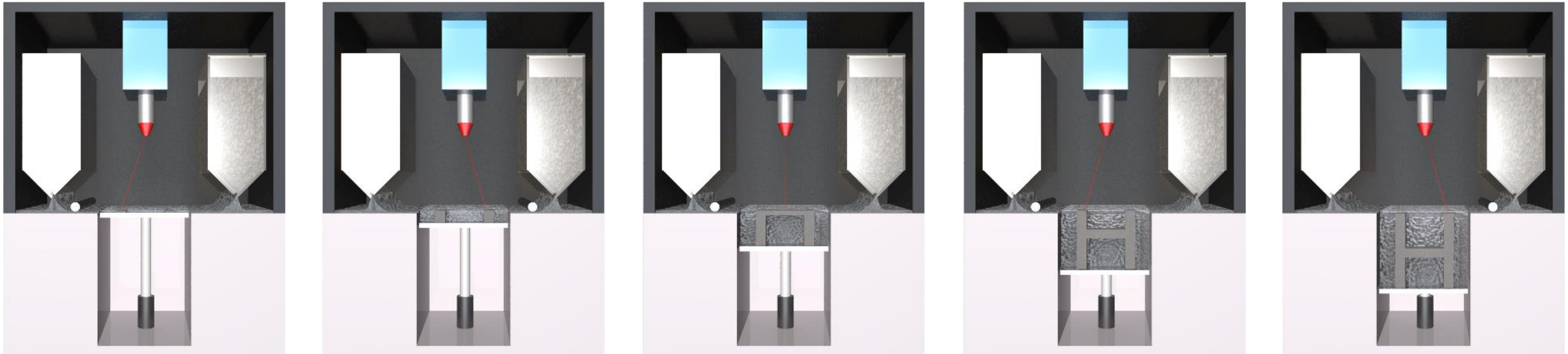
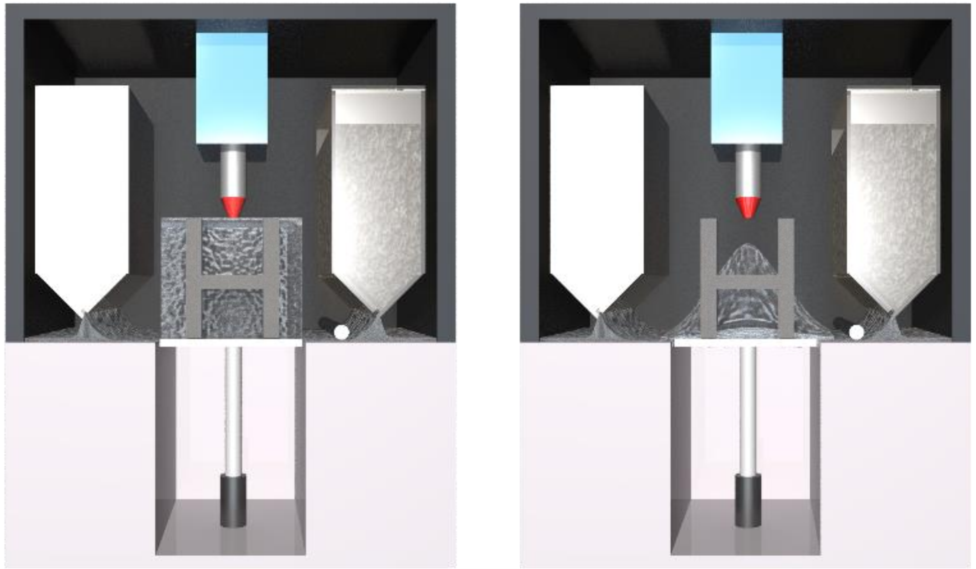
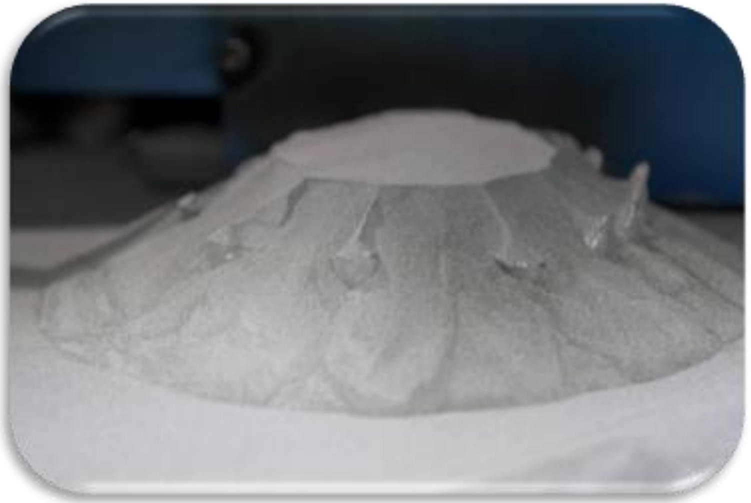
Additive Manufactory - Steps







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Heat Source – DMLS Vs EBM

Direct Metal Laser Sintering (DMLS)

Advantages:

- A broad range of metal materials (>14).
- Finer layer thickness (typically 20–40 µm).
- Better accuracy as a result – typically DMLS produces a smoother surface finish than EBM.
- Print under nitrogen or argon atmosphere

Disadvantages:

- Print under nitrogen or argon atmosphere
- Longer printing times
- The objected is more attached to the support block structures

Electron Beam Melting (EBM)

Advantages:

- Faster than DMLS
- Low level of internal defects.
- Good material properties, in particular fatigue properties.
- No limitations in chemical composition.
- Minimal residual stresses due to high process temperature
- Little waste material: virtually all excess powder can be recycled.

Disadvantages:

- Print under vacuum at high temperatures (650° - 1000° C)
- Thicker layer (50–70 µm for EBM).
- Longer cooling time
- Raw DMLS parts have a surface finish comparable to fine investment cast parts.

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Quality control test methods (2014)

Designation: F3049 – 14

Standard Guide for Characterizing Properties of Metal Powders Used for Additive Manufacturing Processes[1]

B215 Practices for Sampling Metal Powders

B213 Test Methods for Flow Rate of Metal Powders Using the Hall Flowmeter Funnel

B822 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering

E1447 Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by Inert Gas Fusion Thermal Conductivity/Infrared Detection Method

E539 Test Method for Analysis of Titanium Alloys by X-Ray Fluorescence Spectrometry

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Powder contamination & Impact

Contamination

Impact on process and final composition

Effect on mechanical properties

- Storage
- Oxidation
- Mixing of powders
- Foreign particulate

No benchmark for reuse
Impact on final product not well known

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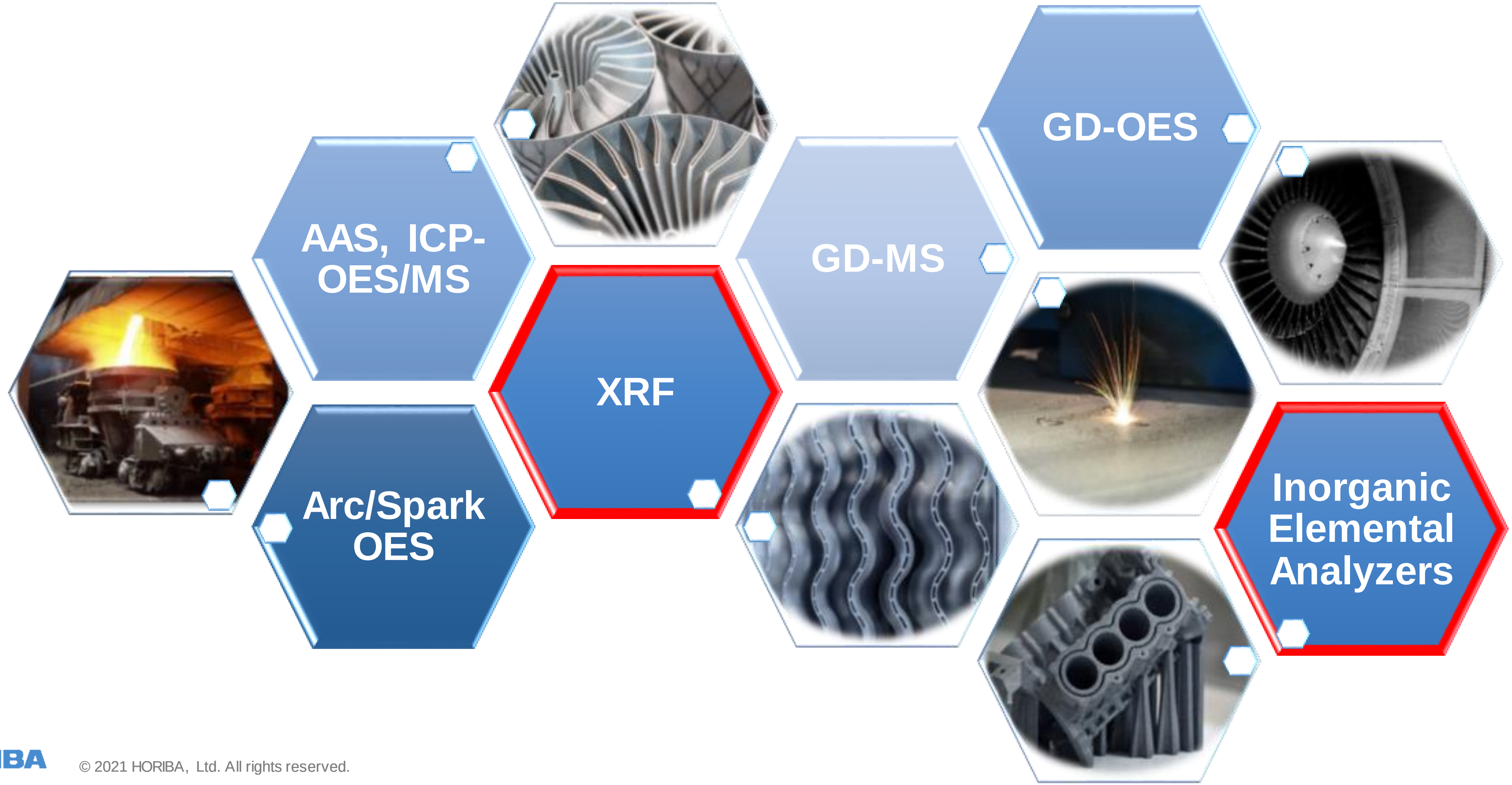
How to assess elemental contamination?



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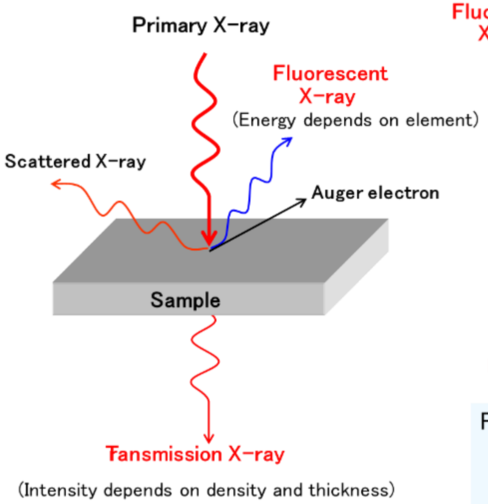
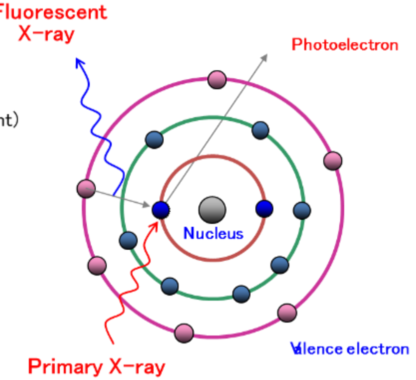
X-Ray Fluorescence

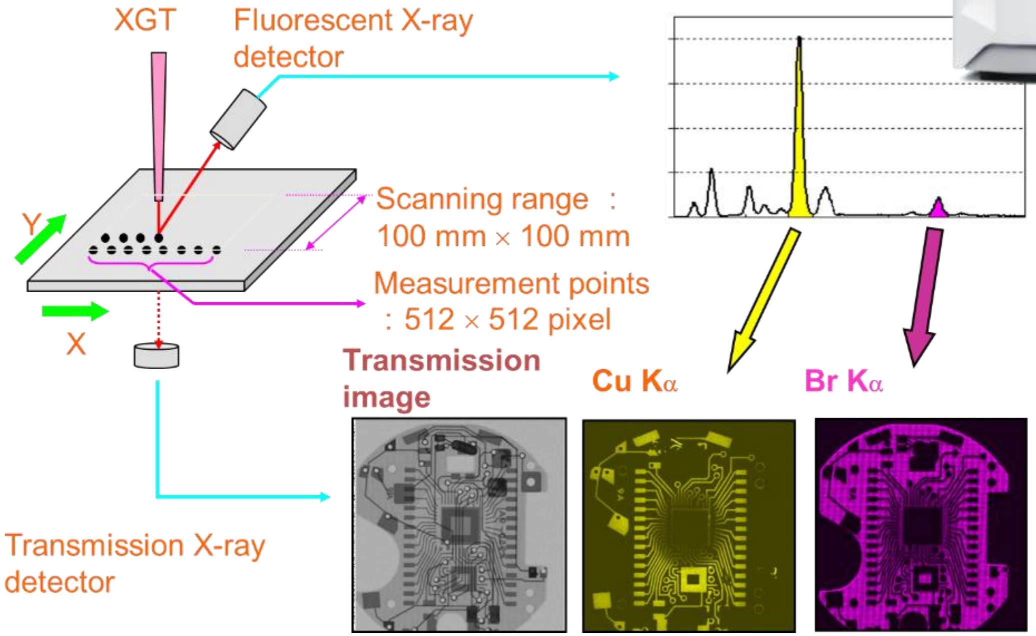


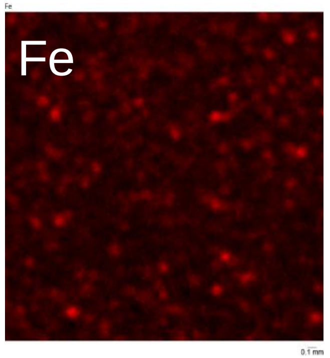


Fluorescent X-ray
Energy → Qualitative
Intensity → Quantitative

Qualitative and Quantitative Analysis

Microscopy and Mapping
Elemental Distribution
Elemental Identification





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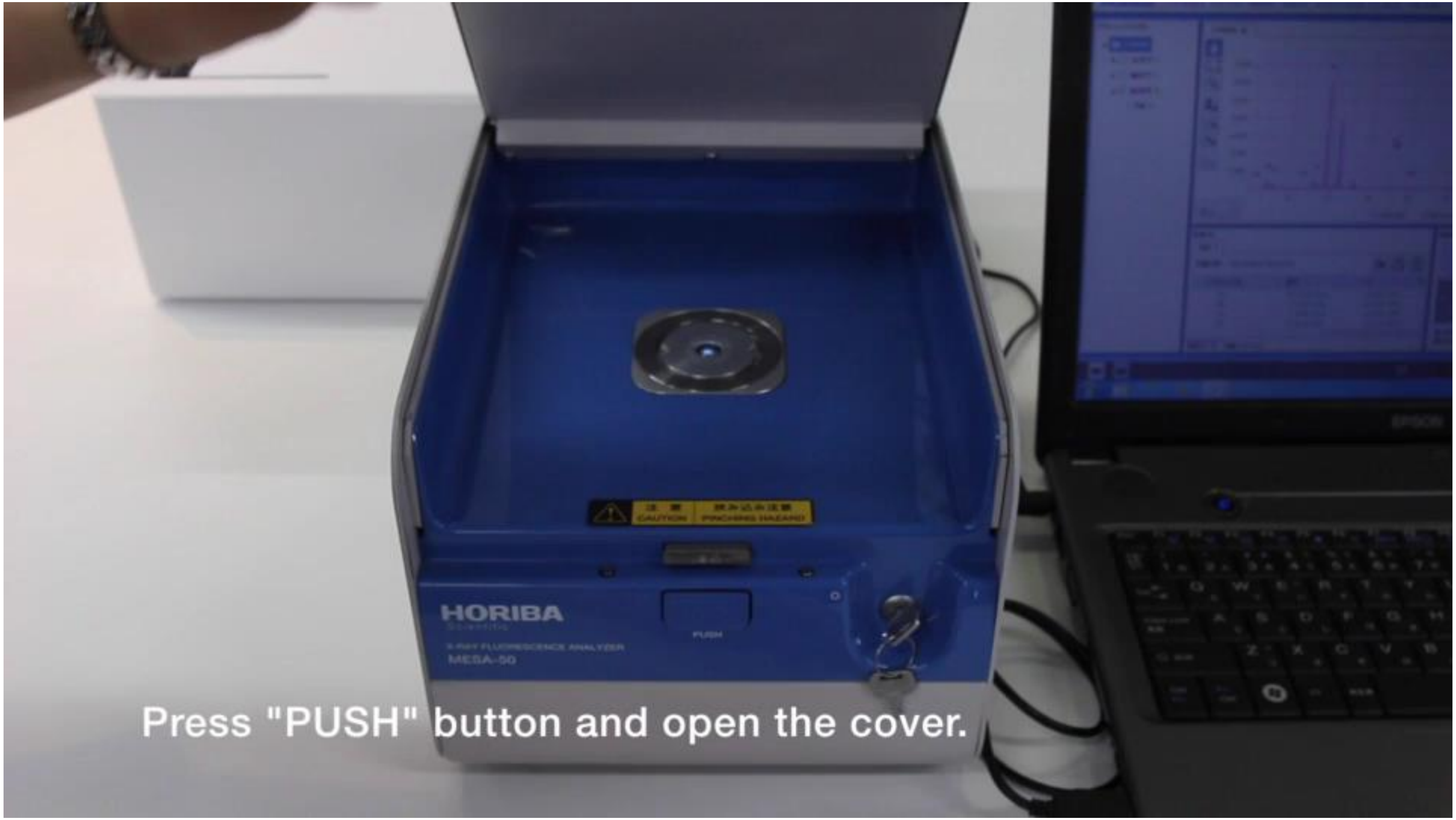
MESA-50 – Operation



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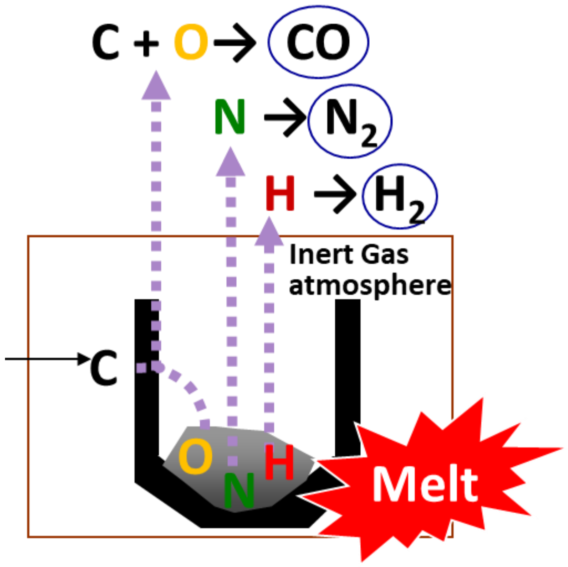
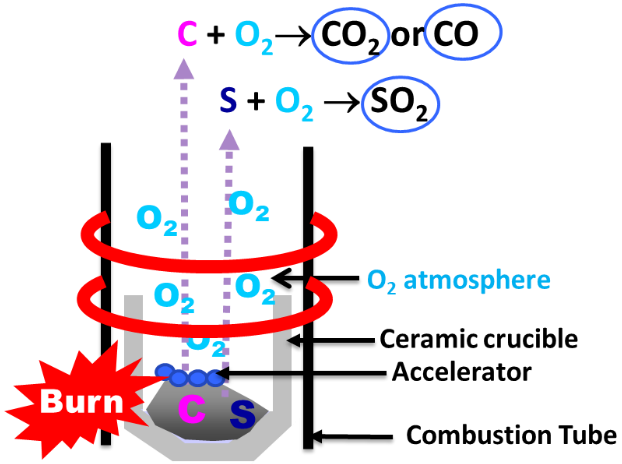
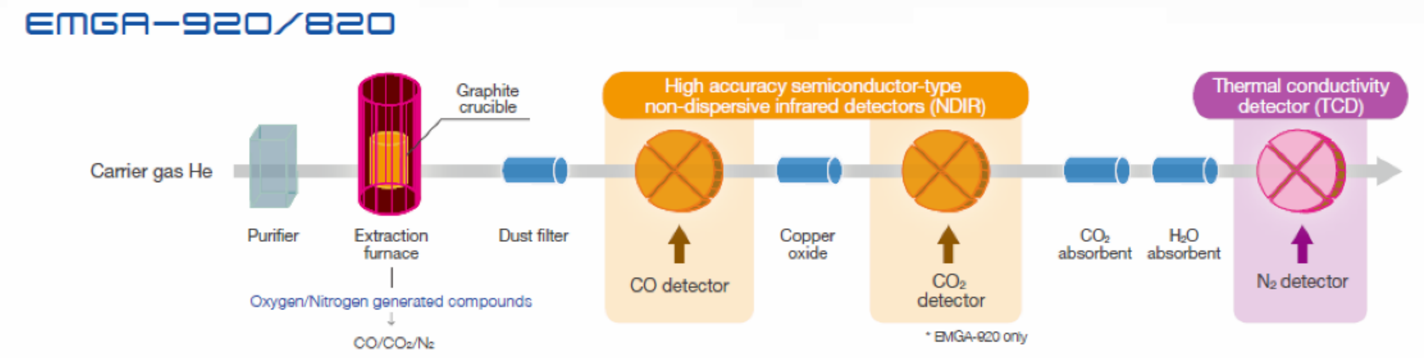
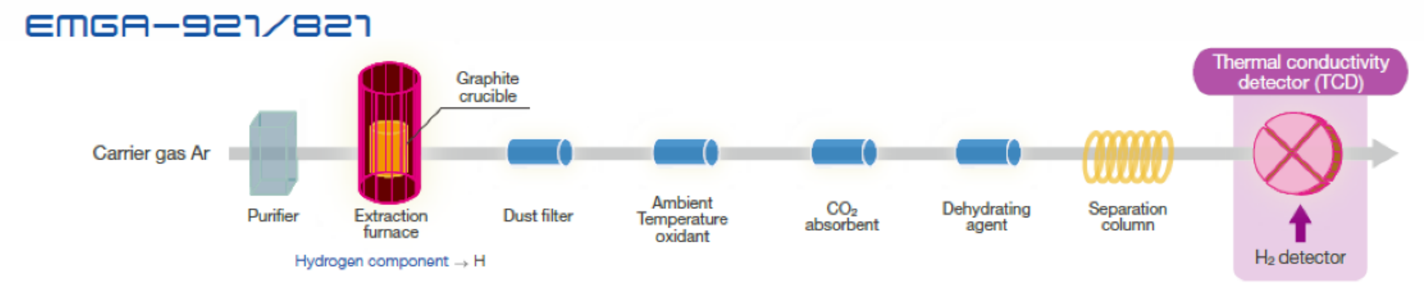
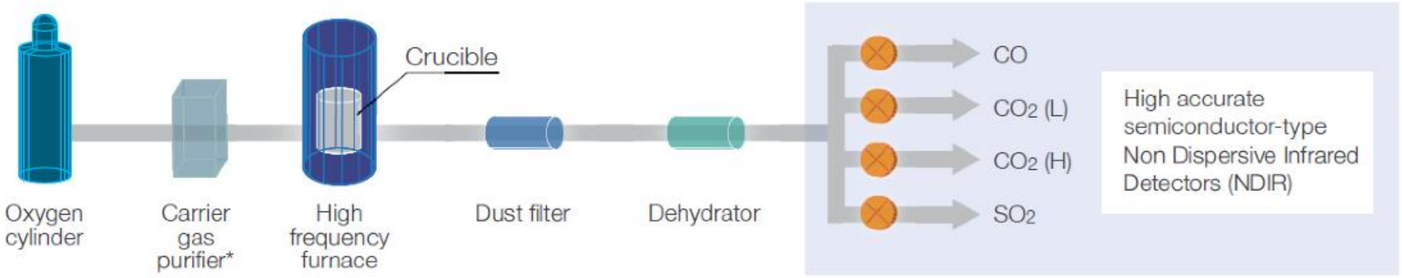
Inorganic elemental analyzers – C/S, O/N & H











*Optional for EMIA-Pro, Standard for EMIA-Expert

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EMIA Series – Operation

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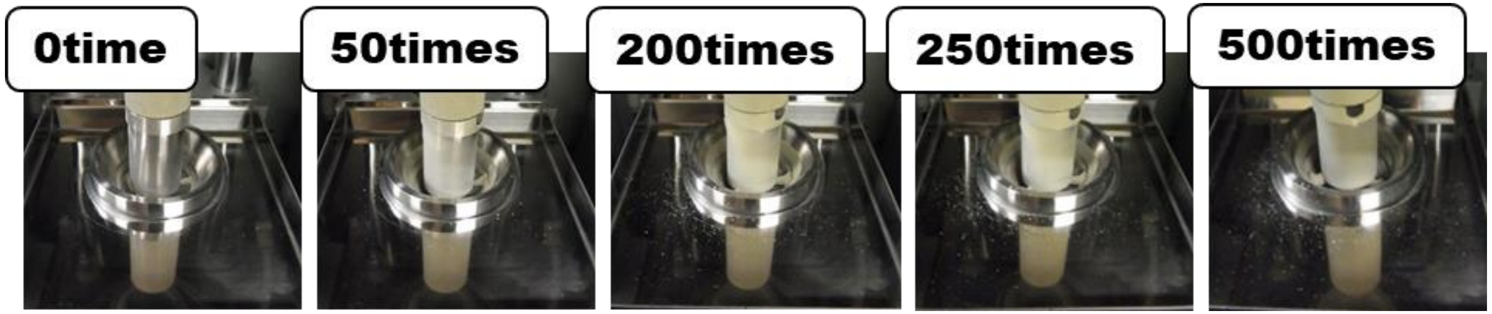
EMIA Series – Unique cleaning mechanism

200 analysis without maintenance

- Patented new cleaning technology & Innovative furnace design
- High stability due to dustless furnace

Fast cycle time

- 70 seconds
- 52 analysis/hour



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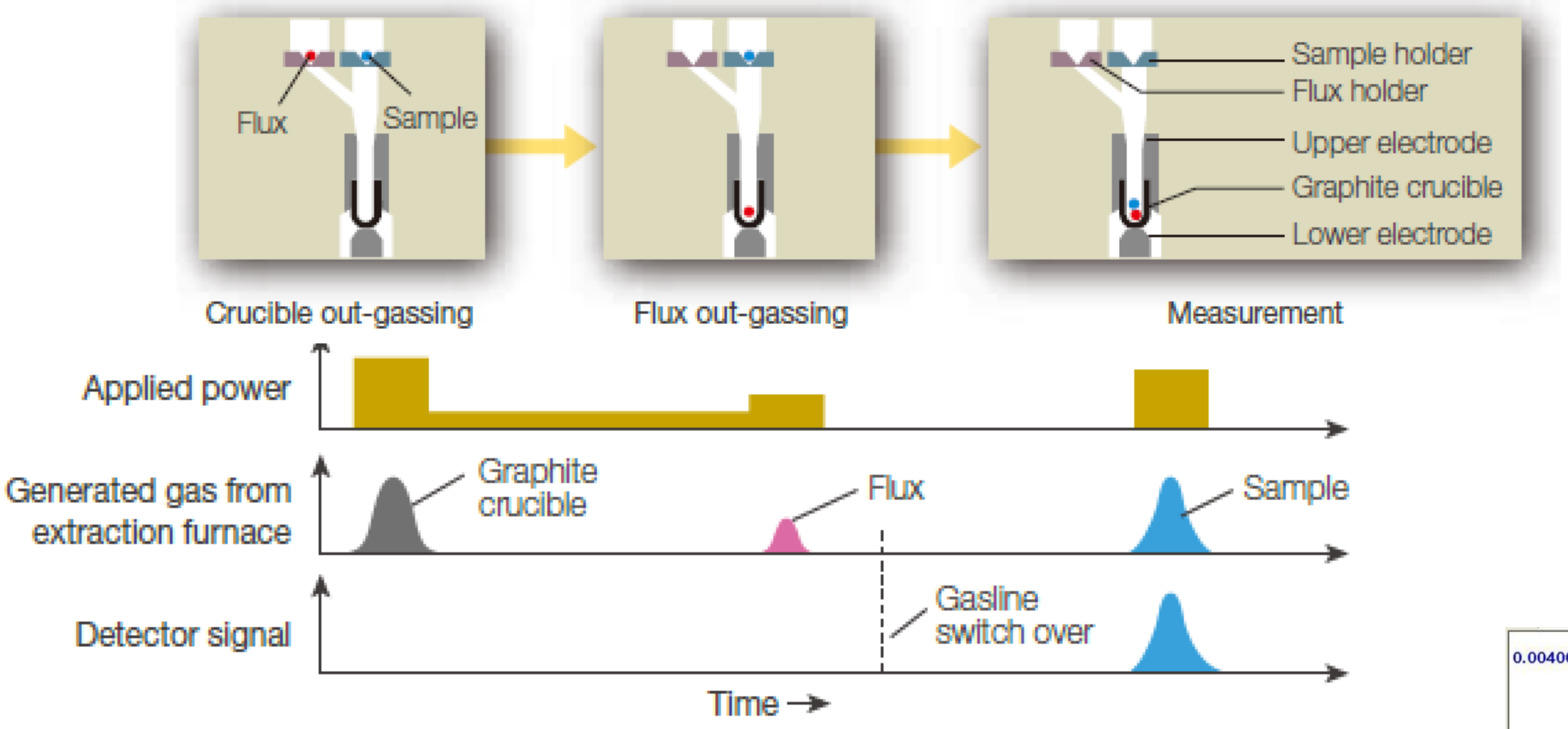
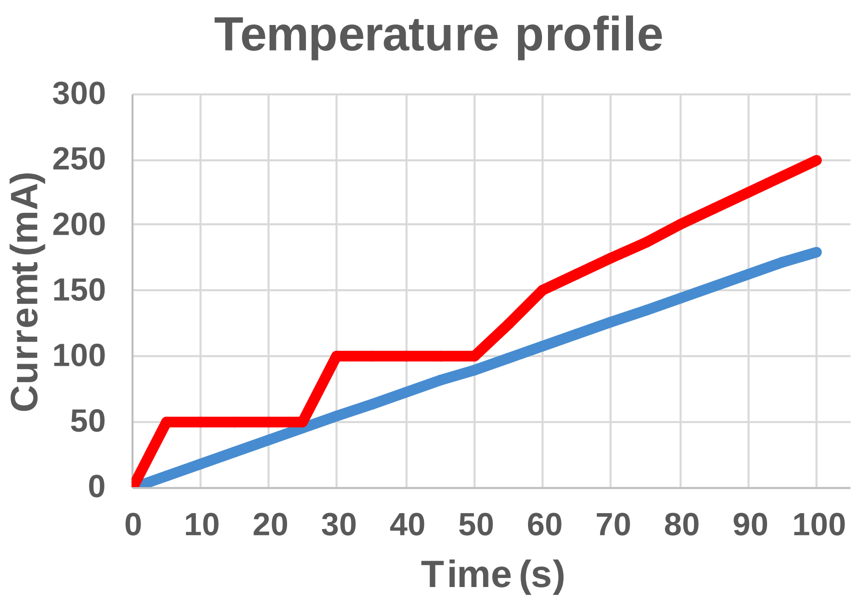
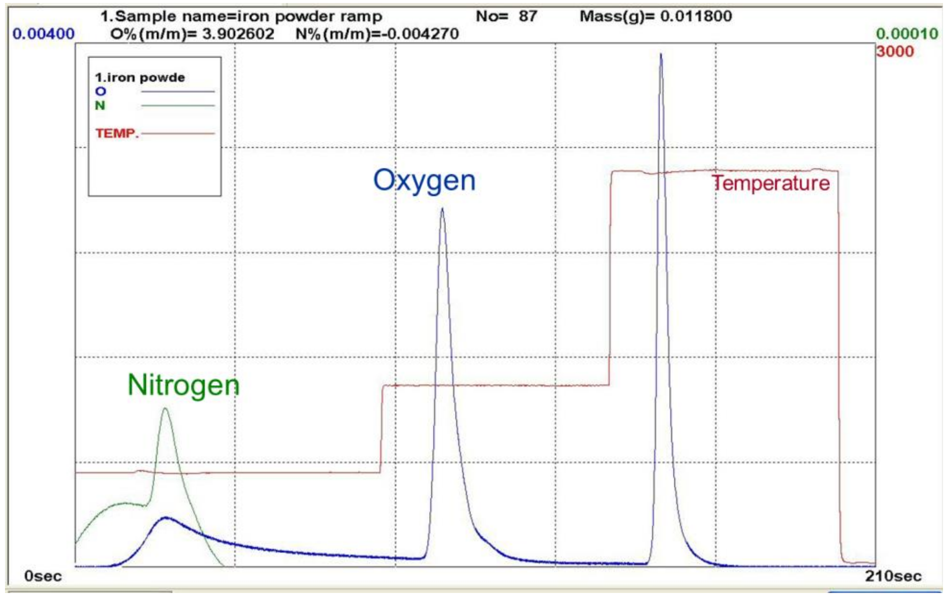
Dual loading & temperature control







Independent outgassing of crucible and flux

Blank reduction for high accuracy

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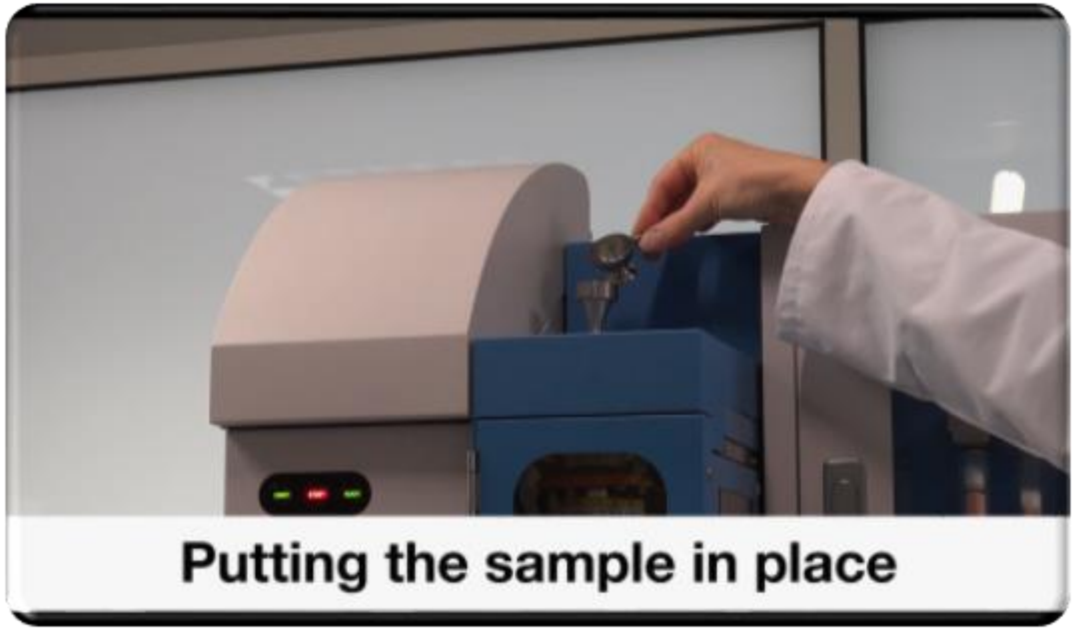
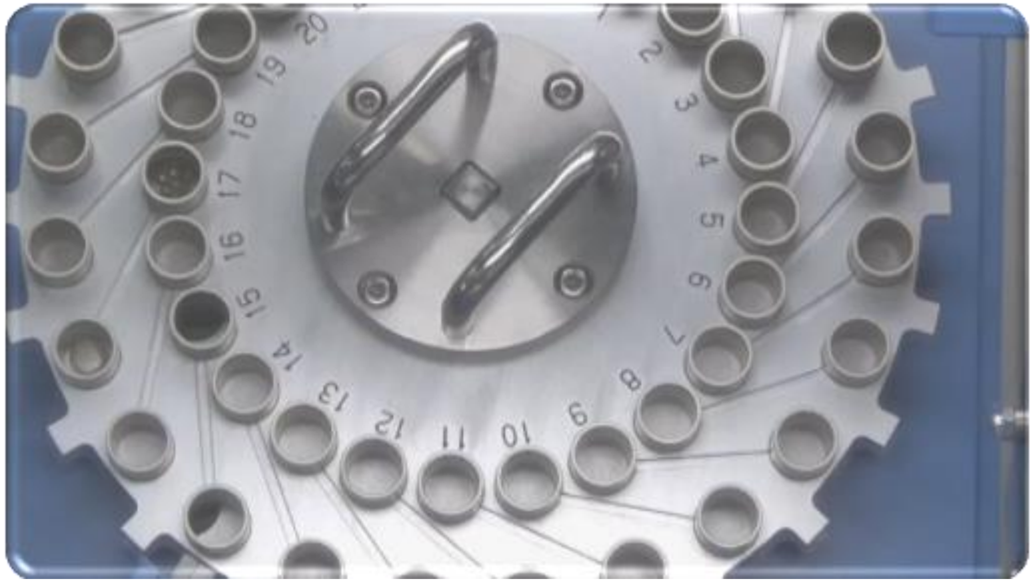
EMGA Series – Operation





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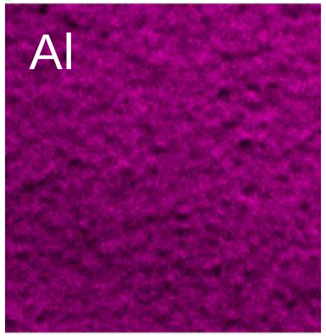
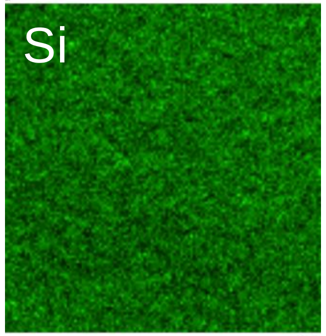
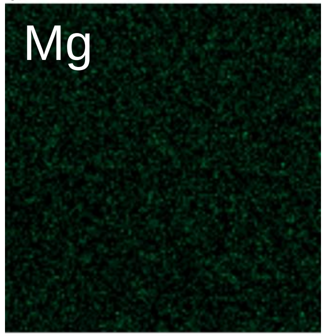
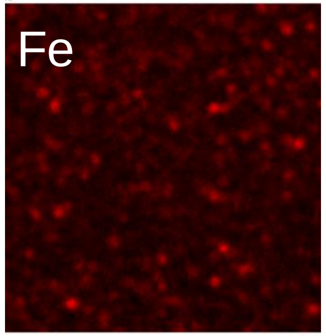
AlMg10Si

Element	Specification Mass %	Result Mass %
Al	Approx. 87-91 (Balance)	84
Si	9.0-11.0	15.0
Mg	0.25-0.45	0.79
Fe	<0.25	0.50
N	<0.20	<0.0002
O	<0.20	0.11









Element	Result Mass ppm	SD
C	100	3
S	244	11
H	38.3	1.0

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Ti6Al4V

Element	Specification Mass %	Result Mass %
Ti	Approx. 88-91 (Balance)	90
Al	5.50-6.50	5.50
V	3.50-4.50	4.48
C	<0.08	0.0123
O	<0.13	0.125
N	<0.05	0.0134
H	<0.012	0.0034

Run	Mass (g)	O% (m/m)	N% (m/m)
1	0.1155	0.125	0.0134
2	0.1153	0.128	0.0135
3	0.1027	0.122	0.0135
Average		0.125	0.0134
SD		0.003	0.0001
RSD%		2%	0.4%

Run	Mass (g)	C% (m/m)
1	0.1999	0.0123
2	0.2019	0.0124
3	0.1816	0.0122
Average		0.0123
SD		0.0001
RSD%		0.8%

Run	Mass (g)	H% (m/m)
1	0.1986	0.0035
2	0.2106	0.0033
3	0.2022	0.0034
Average		0.0034
SD		0.0001
RSD%		1.7%

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316L S.S.

Element	Specification Mass %	Result Mass %
Fe	Approx. 61-72 (Balance)	63
Cr	16.00-18.00	17.10
Ni	10.00-14.00	13.05
Mo	2.00-3.00	2.67
Mn	<2.00	1.27
Si	<1.00	0.76
N	<0.10	0.09
O	<0.10	0.03
C	<0.03	0.02
S	<0.03	0.003

Run	Mass (g)	O% (m/m)	N% (m/m)
1	0.3007	0.0300	0.0928
2	0.3000	0.0306	0.0932
3	0.2743	0.0301	0.0932
Average		0.0302	0.0931
SD		0.0003	0.0002
RSD%		1.1%	0.2%

Run	Mass (g)	C% (m/m)	S% (m/m)
1	0.1992	0.0199	0.0029
2	0.1815	0.0200	0.0031
3	0.1870	0.0201	0.0030
Average		0.0200	0.0030
SD		0.0001	0.0001
RSD%		0.5%	3.3%

Run	Mass (g)	H ppm
1	0.1950	7.16
2	0.2097	7.19
3	0.2020	7.16
Average		7.17
SD		0.02
RSD%		0.2%

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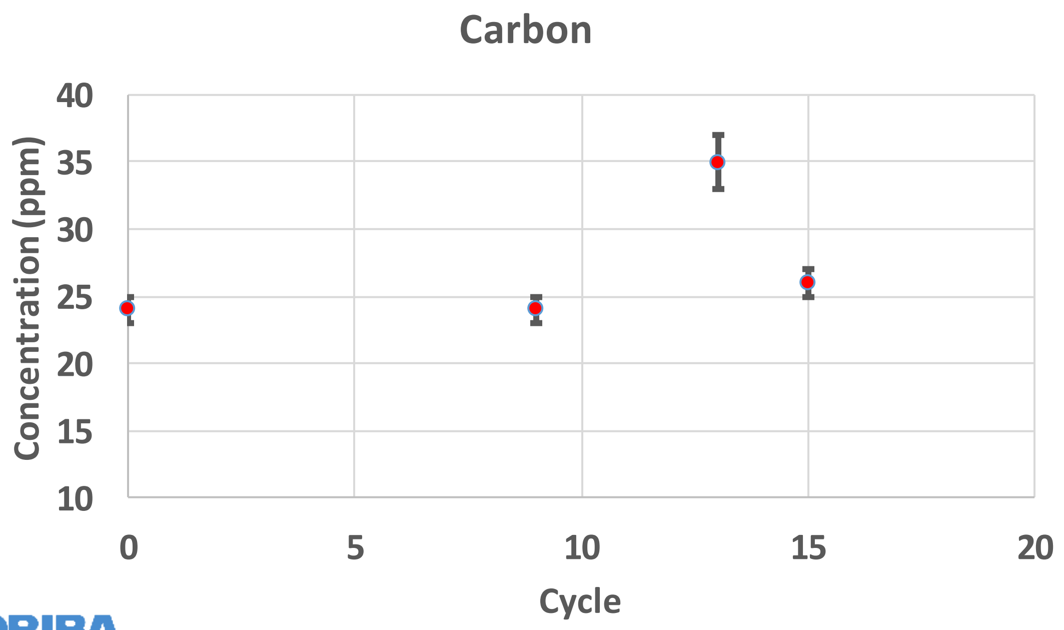
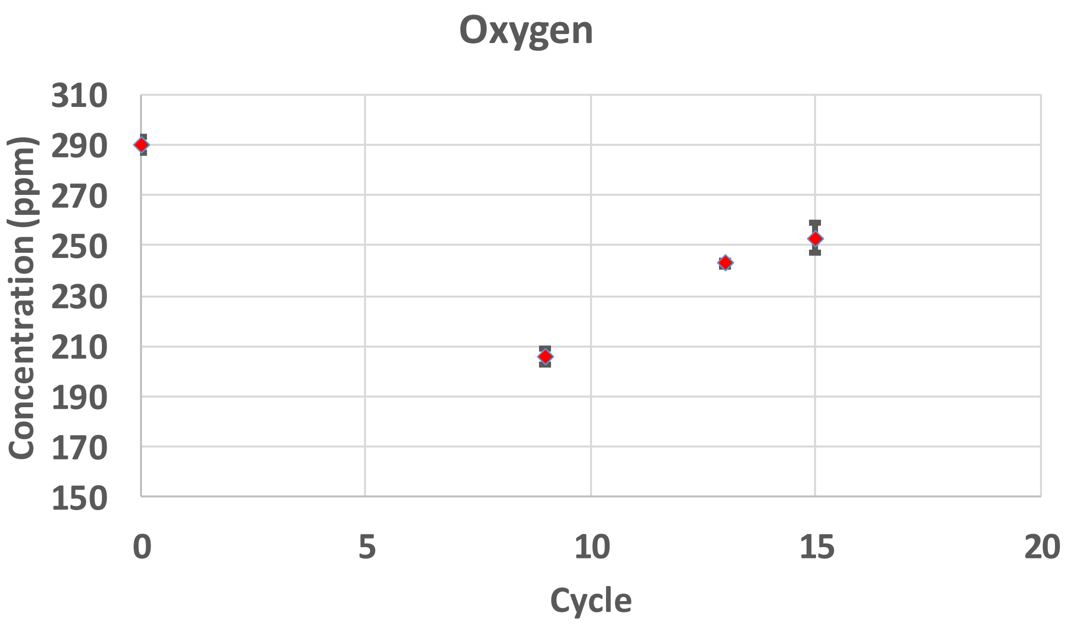
Hastelloy C276 – Monitoring of recycled powder3

Cycle	C ppm	S ppm	O ppm	N ppm	H ppm
0	24	11	290	51	<5
9	24	11	206	64	<5
13	35	13	243	68	<5
15	26	9	253	64	<5





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Size vs Functionality

Properties related to Particle Sizer

Property	With decreasing of particle size
Homogeneity	↑ Increase
Elastic Behavior	↓ Decrease
Resistance	↑ Increase
Brightness	↑ Increase
Opacity	↓ Decrease
Agglomeration	↑ Increase

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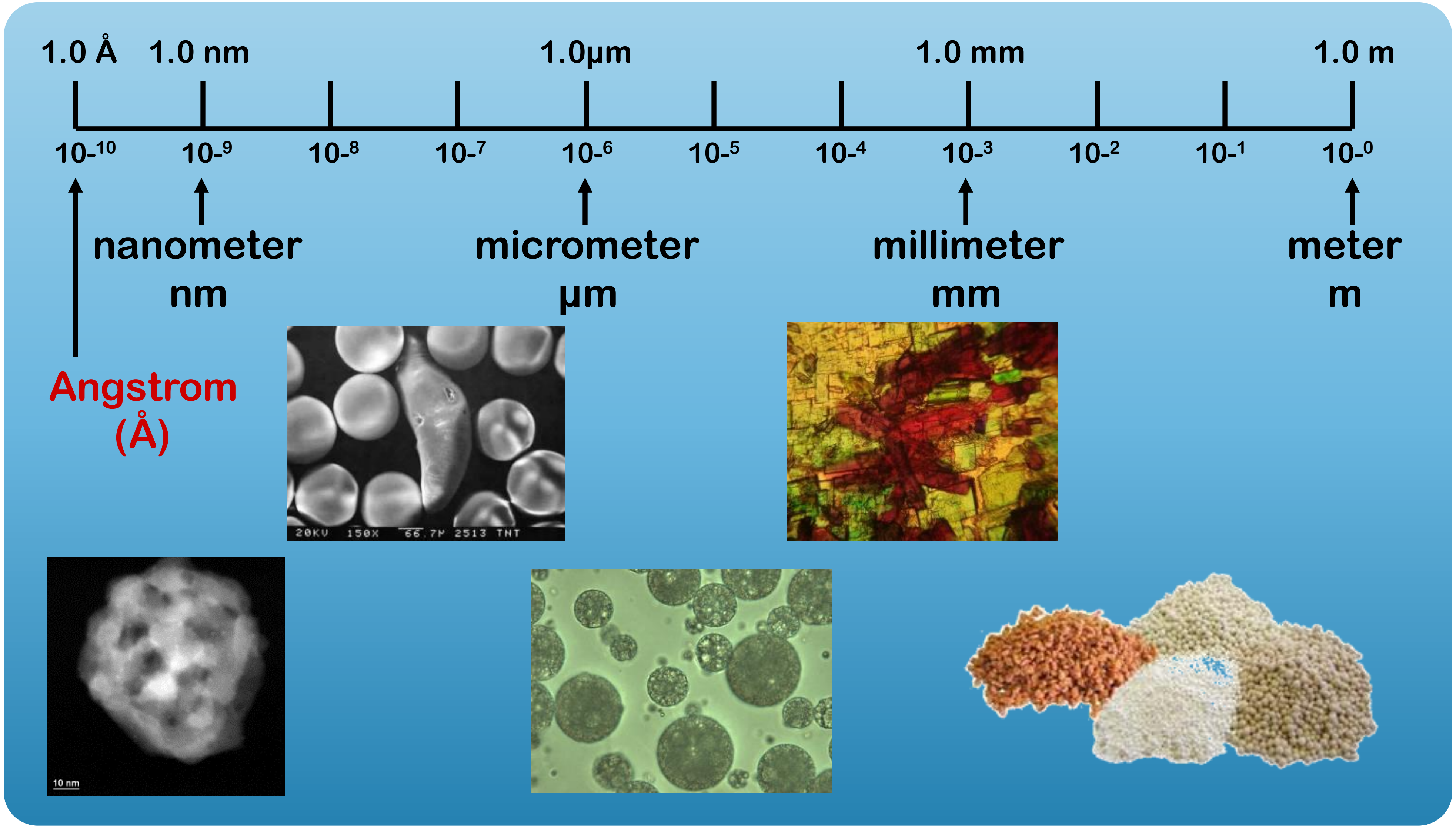
Scale



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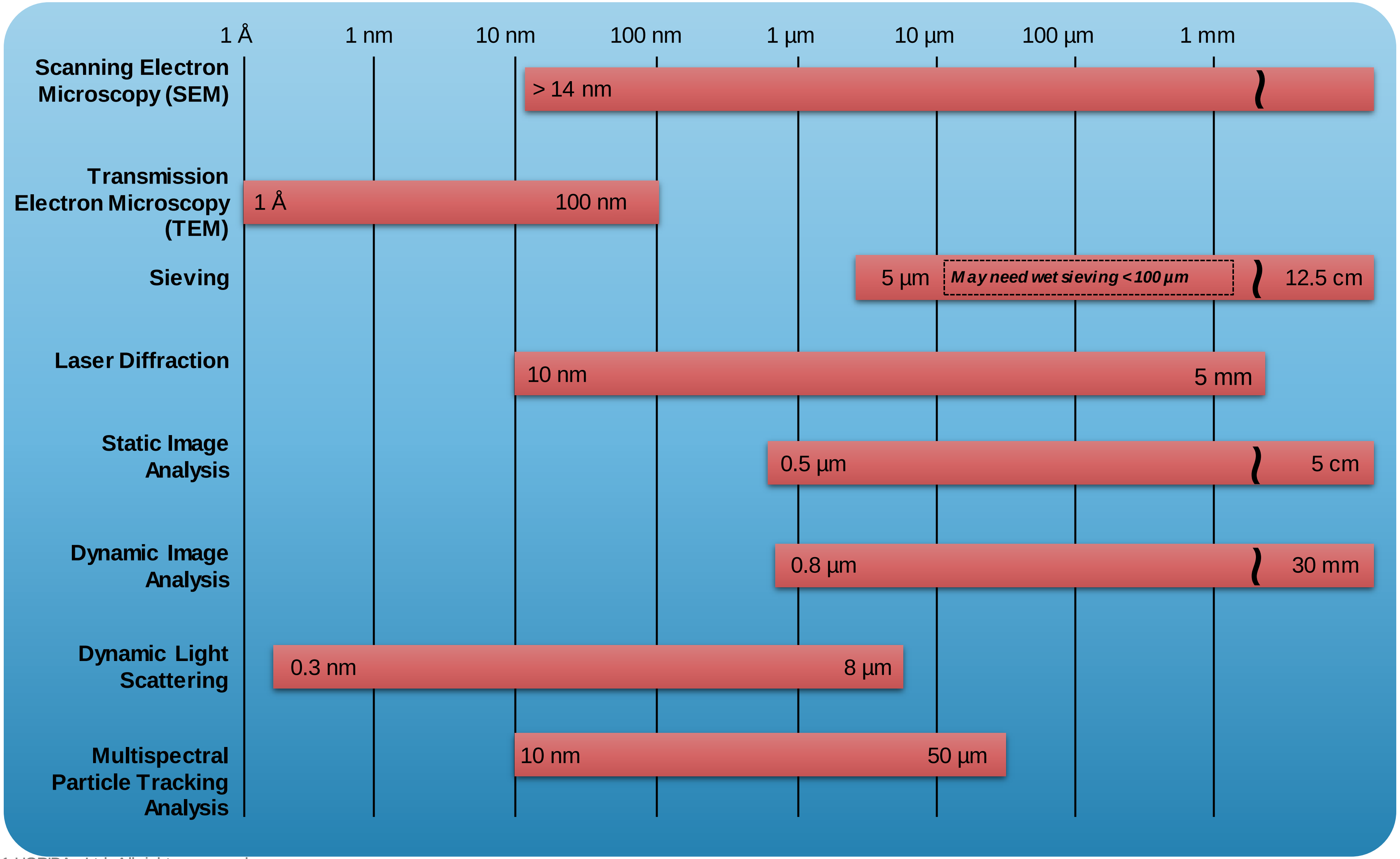
Measurement Range of the Main Techniques for Particle Characterization



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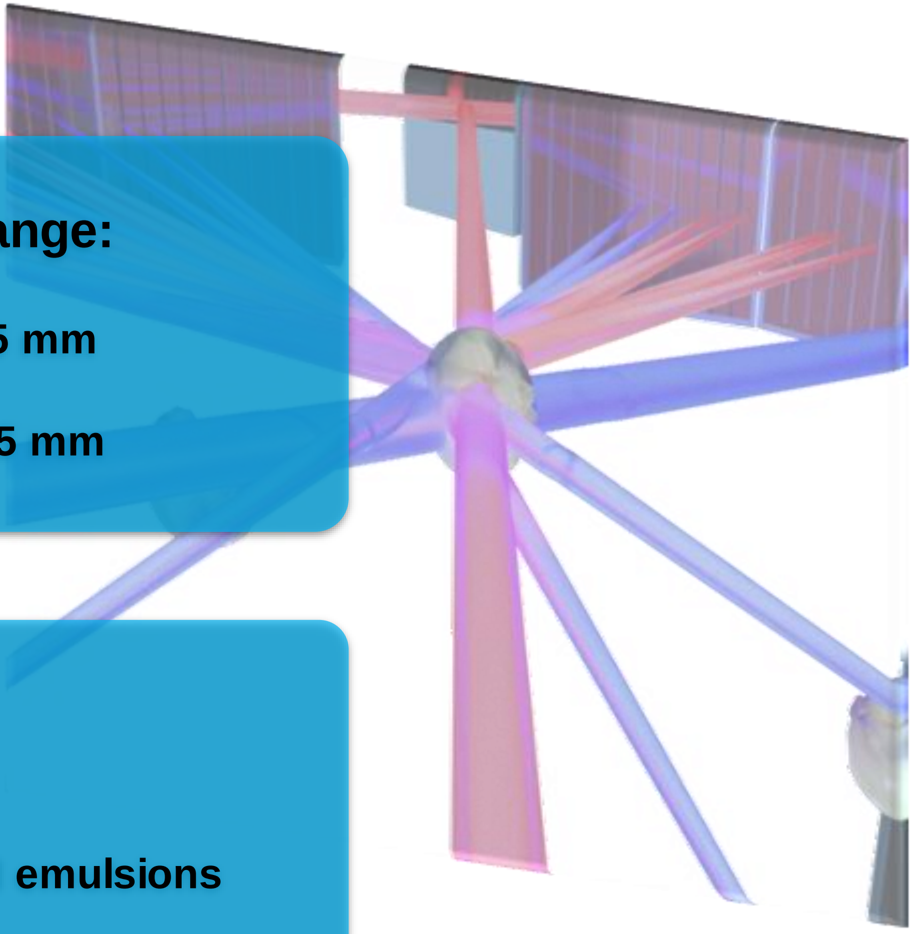
Laser Diffraction (Static Light Scattering)



Measurement Range:

- Dry dispersion: 0.1 µm – 5 mm
- Wet dispersion: 10 nm – 3.5 mm

Sample:

- Powders, suspensions and emulsions

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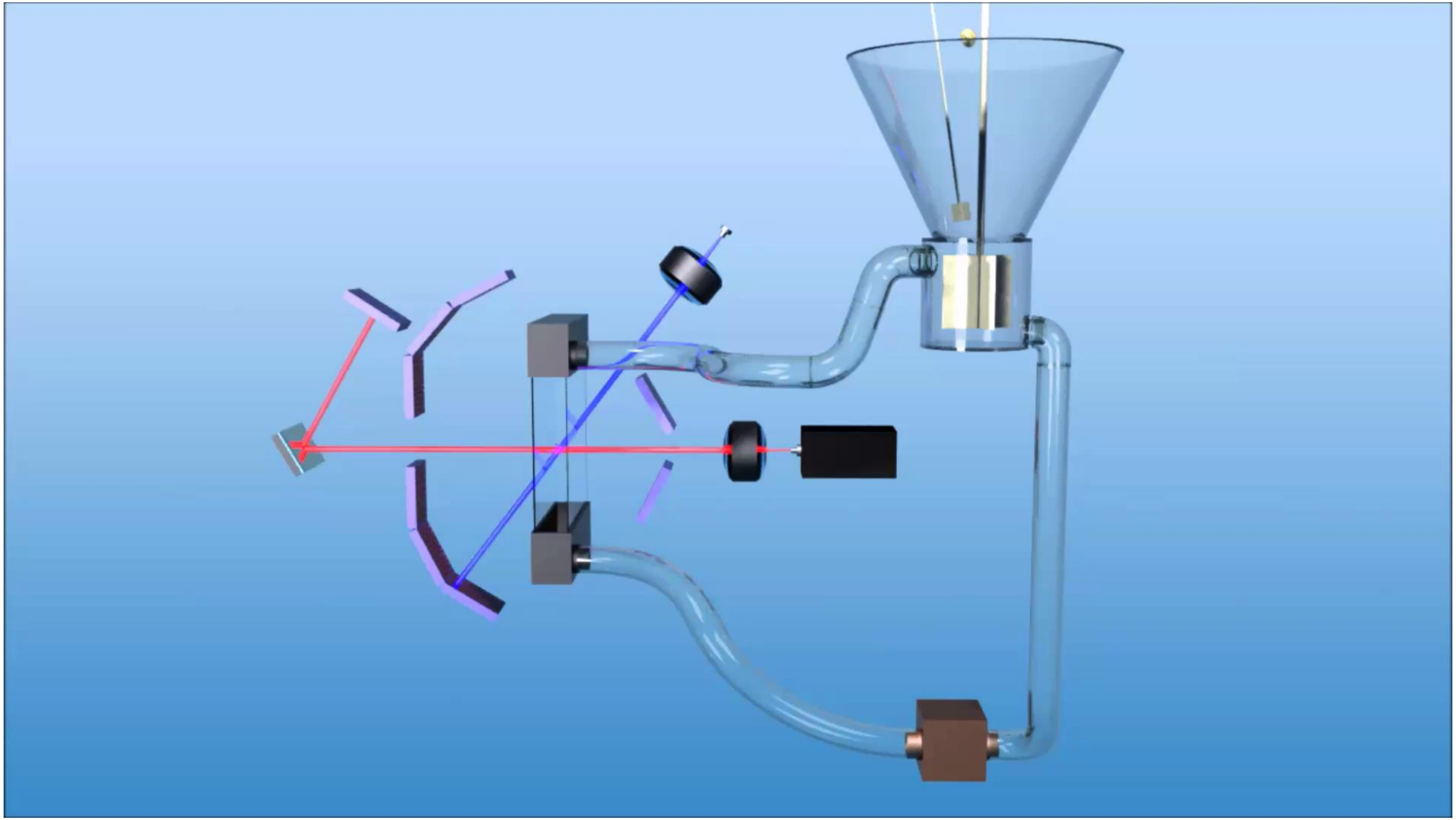
Double Beam Laser Diffraction Instrument Schematic



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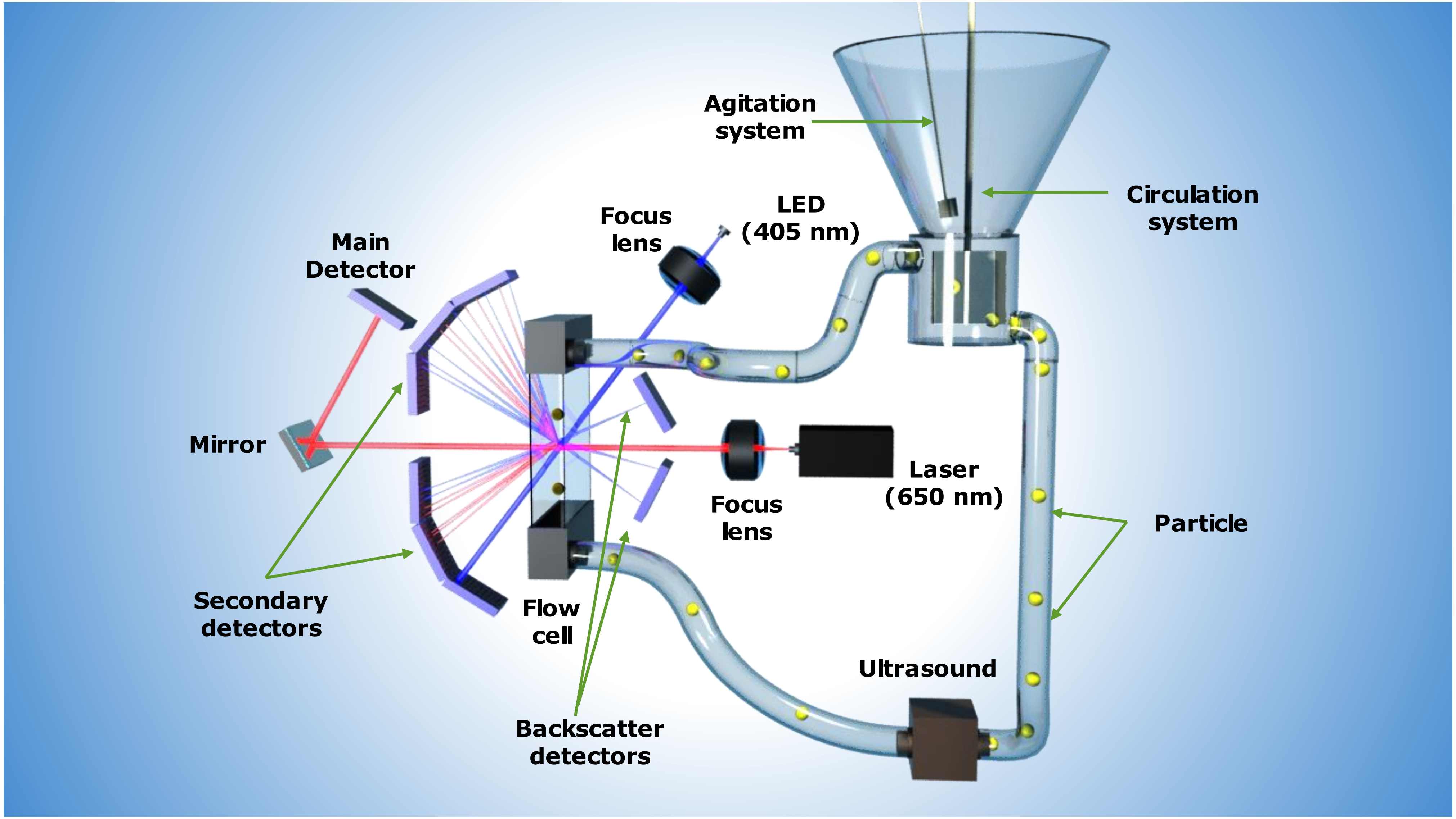
Double Beam Laser Diffraction Instrument Schematic



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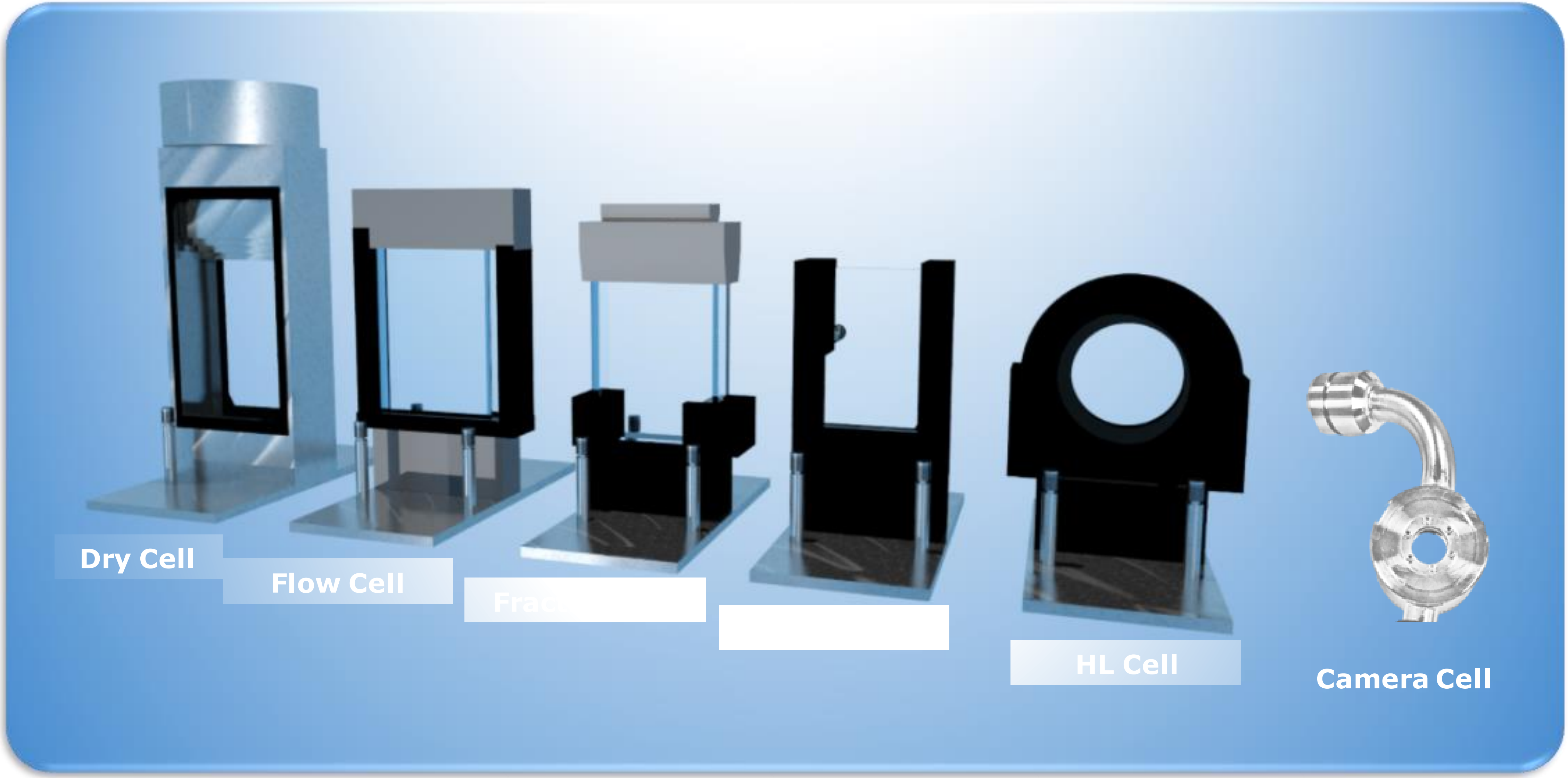
Different Measurement Options



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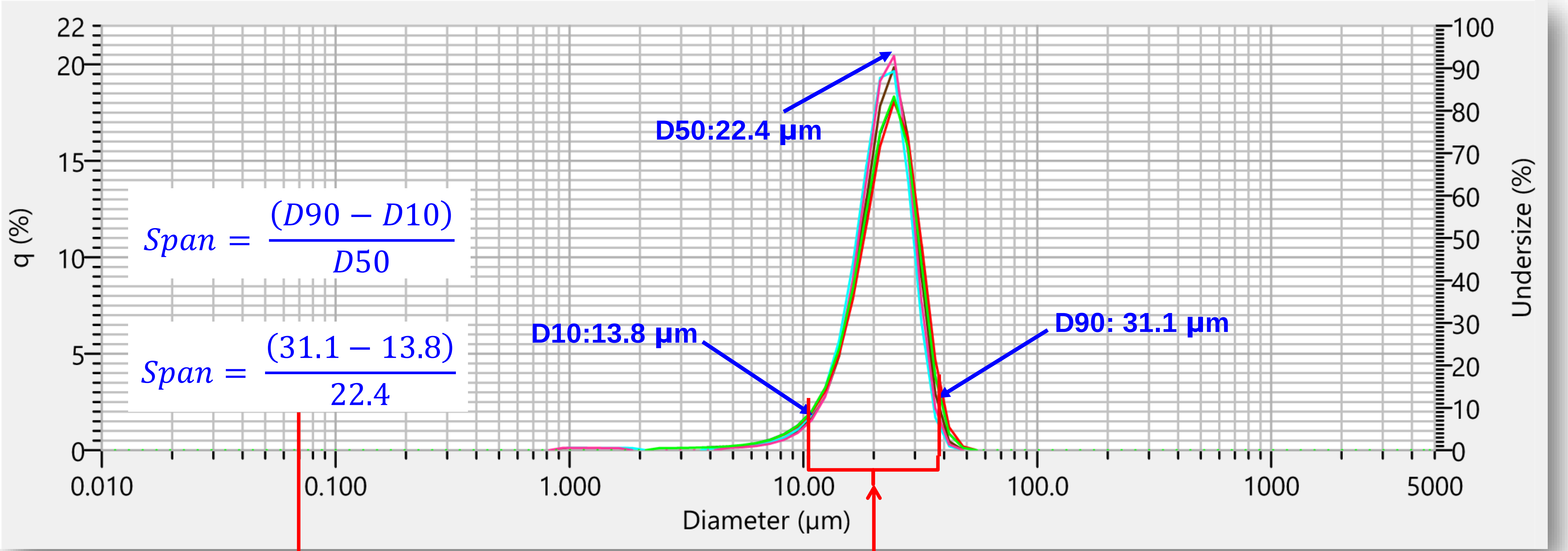
Fresh sample – Particle size distribution



$$Span = \frac{(D90 - D10)}{D50}$$

$$Span = \frac{(31.1 - 13.8)}{22.4}$$

$$Span = 0.772$$

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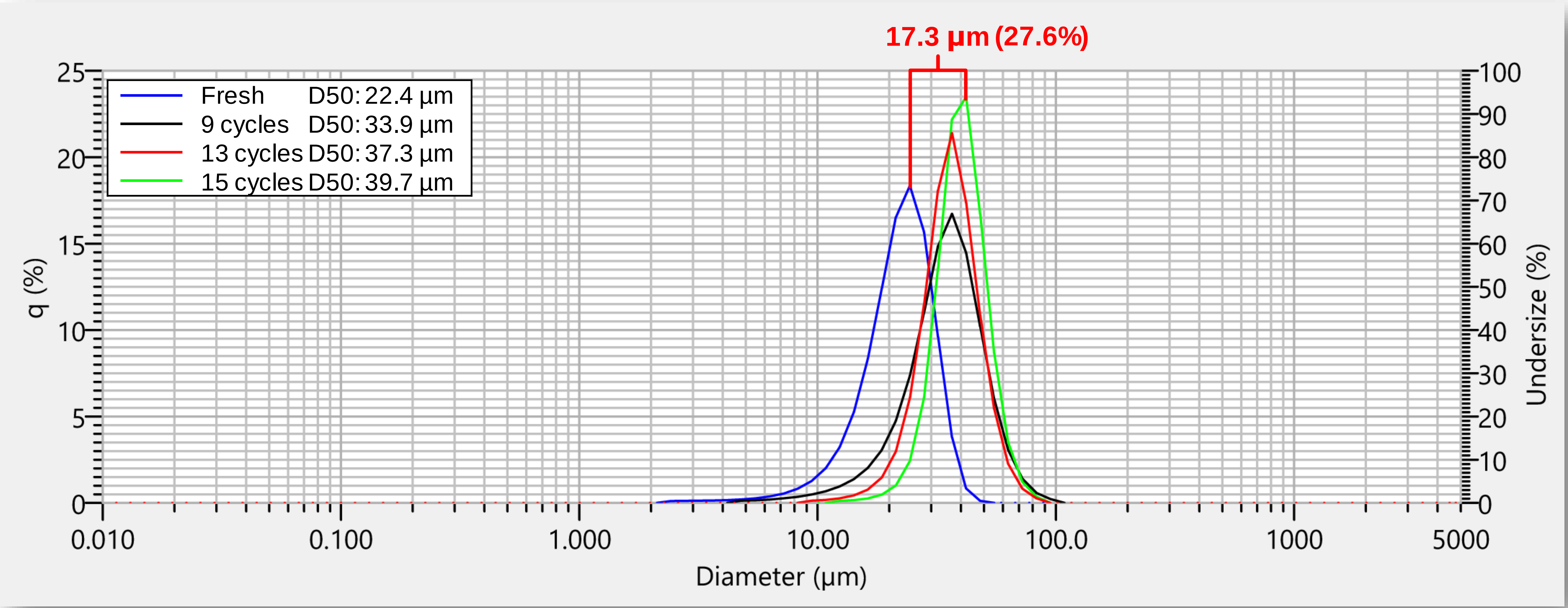
Distribution shift due to recycling



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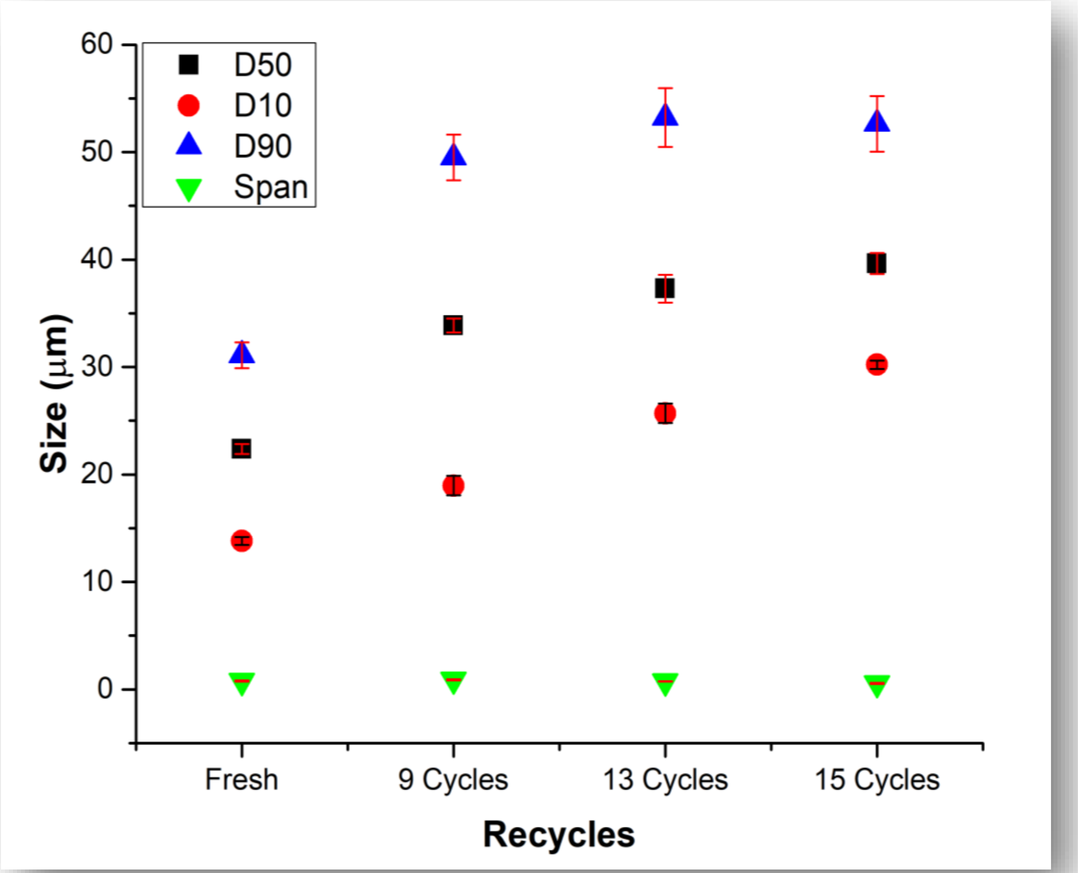
Results Evaluation

Cycles	D50	CoV	D10	CoV	D90	CoV	Span	CoV
	µm	%	µm	%	µm	%		%
Fresh	22.4	2.1	13.8	2.6	31.1	3.8	0.772	6.7
9 cycles	33.9	1.9	19.0	4.7	49.5	4.3	0.901	6.0
13 cycles	37.3	3.5	25.7	3.5	53.2	5.2	0.737	3.9
15 cycles	39.7	2.5	30.2	1.3	52.6	4.9	0.565	10.7



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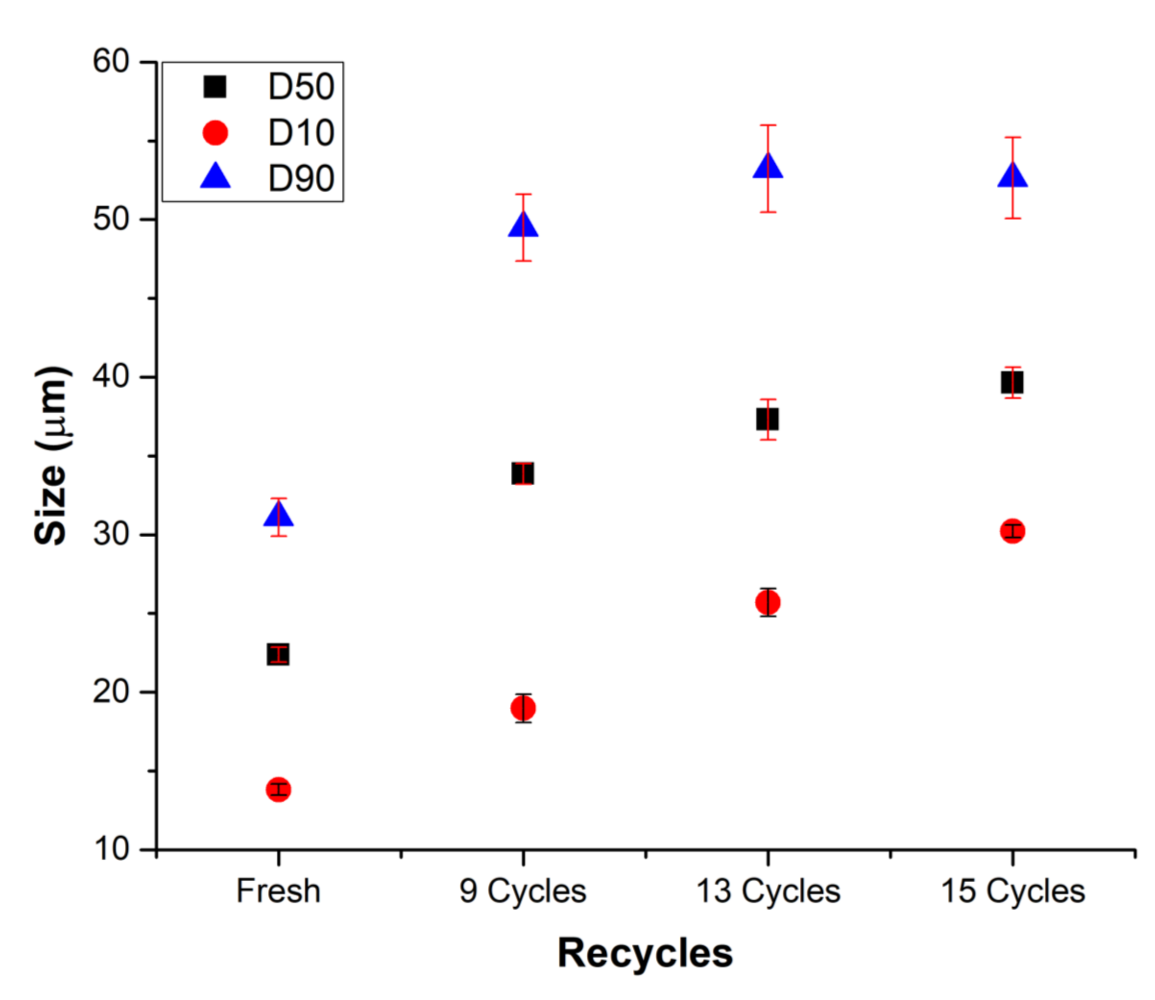
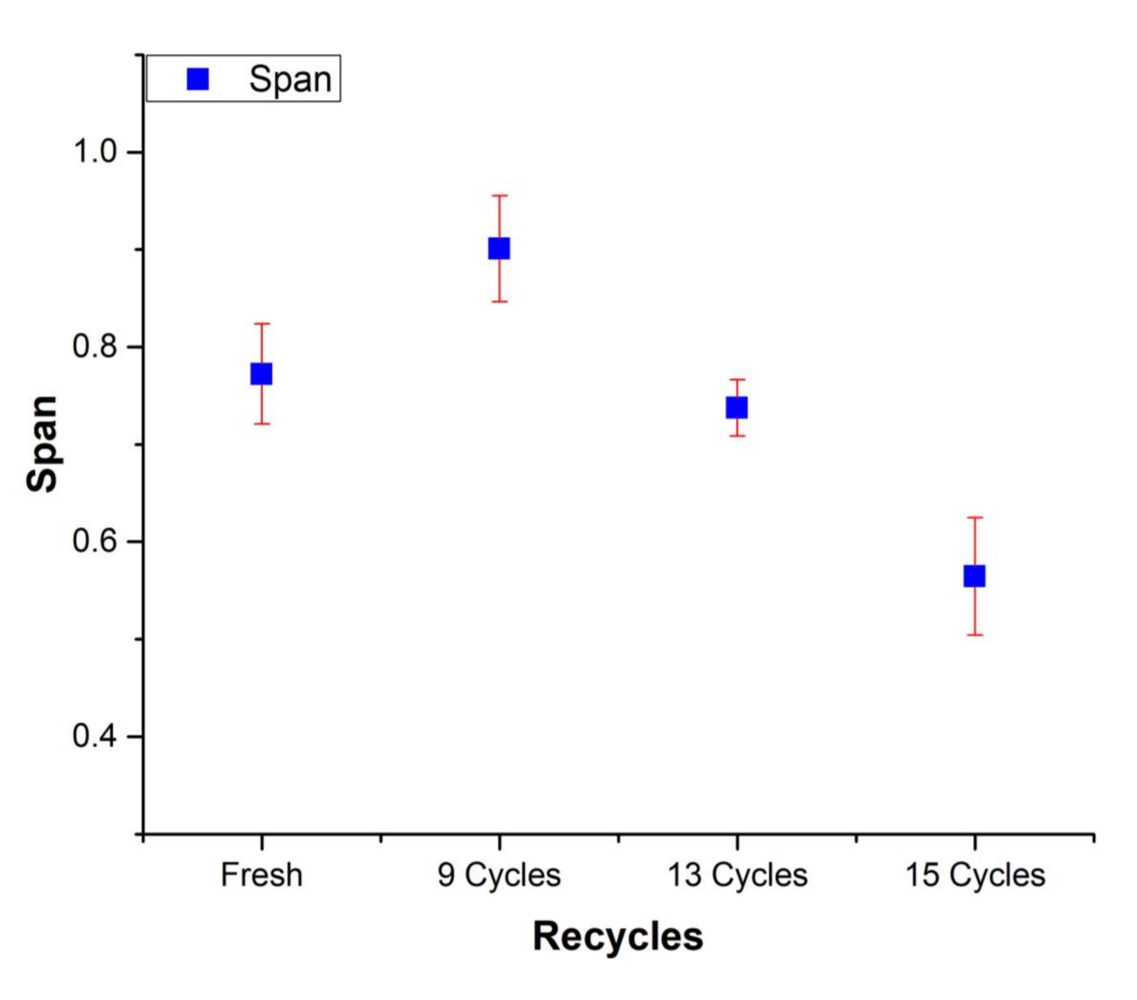
Results Evaluation





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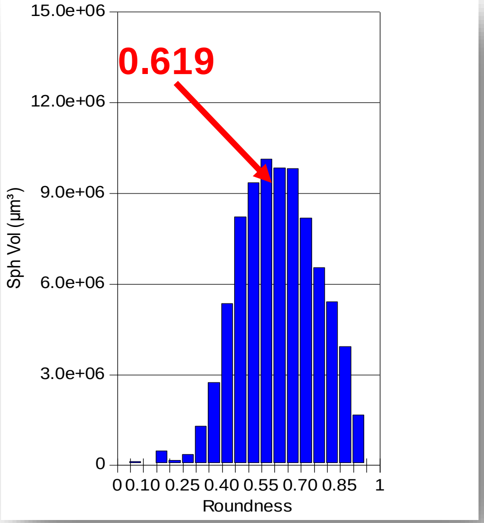
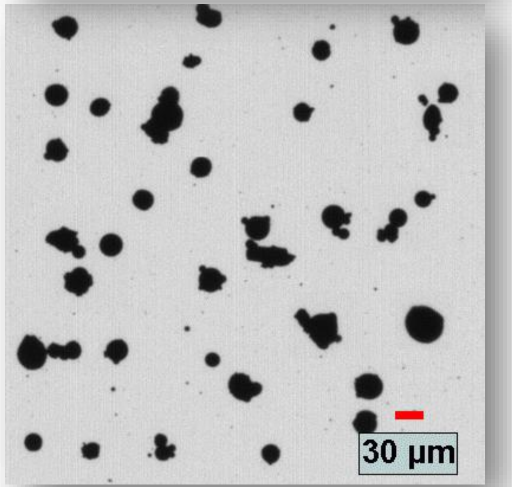
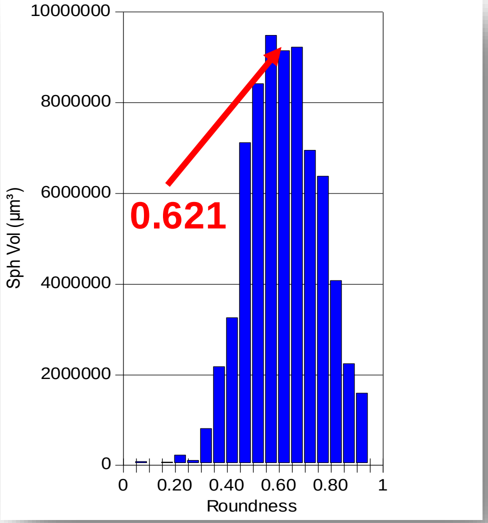
Shape Analysis – Roundness*

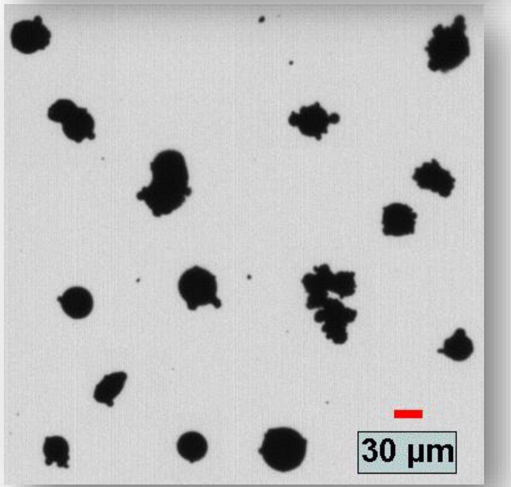
Fresh



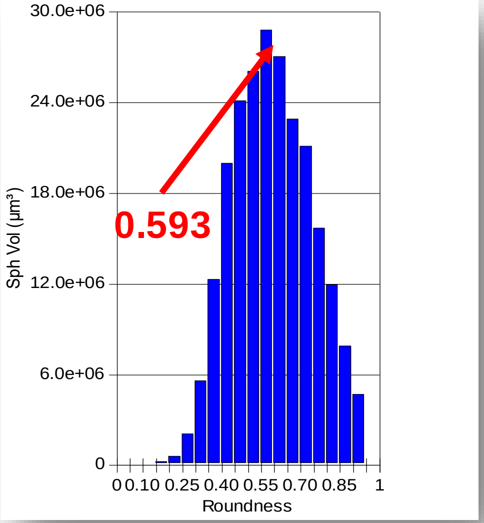


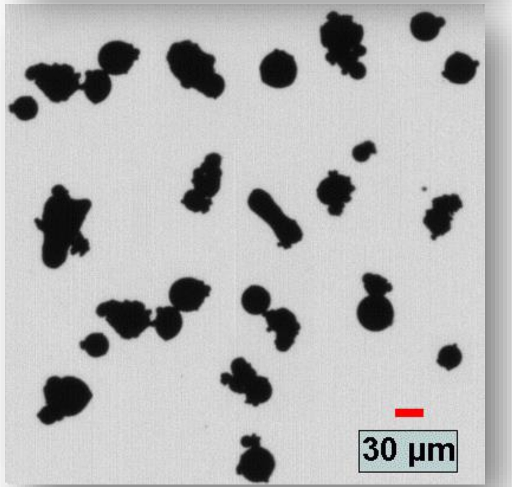
9 Cycles



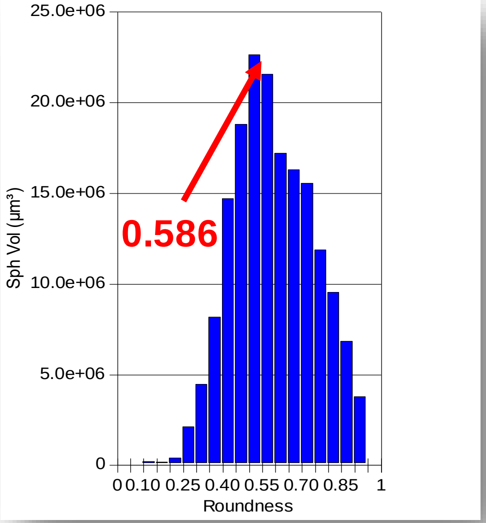


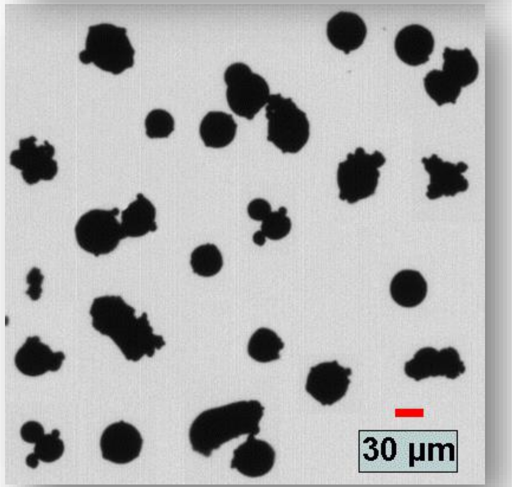
13 Cycles





15 Cycles





*Roundness = closer to 1 more spherical is the particle

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Conclusion

Additive Manufacturing Process

- Quality of final product related to the quality of fresh powders and its recycles as recommended by ASTM F3049
- Elemental composition impact mechanical properties
- Particle size affects process and final product properties
- Recycling of powders implies higher risk of contamination and evolution of powder characteristics

Elemental Analysis

- XRF and inorganic elemental analyzers are appropriate for additive manufacturing raw material and process control and can provide results within minutes
- Both techniques involve minimal to no sample preparation and can be used for R&D purposes as well as for industrial lab/QC in factories
- Efficiency and quality can be ensured and improved thanks to appropriate quality control of powders
- ROE and benchmark on reuse of powders and its effect on the final product can ultimately be established

Particle Size Analysis

- The particle size and shape analysis are essential to evaluate the size distribution of fresh and recycles powders.
- The quality of the final product is drastically impacted by the powder particle size.

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Omoshiro-okashiku

Joy and Fun

Terima kasih

நன்றி

Gracias 谢谢

Danke

Tack ska du ha

Σας ευχαριστώ πάρα πολύ

धन्यवाद

Grazie

THANK YOU

شُكْرًا

ขอบคุณครับ

Большое спасибо

Obrigado

ありがとうございました

Cảm ơn

Merci

감사합니다

Dziękuję

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