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Transilvania University of Brasov - Romania
Materials Science and Engineering Faculty

Image analysis methods for cross-contamination detection in raw powders for powder bed fusion

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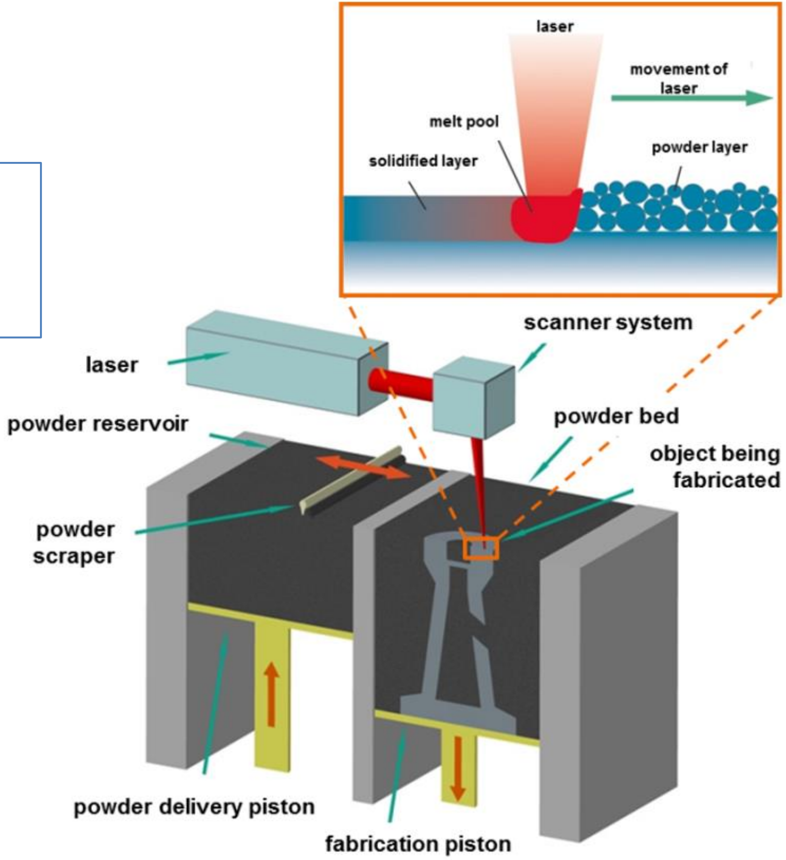
UNIVERSITÀ POLITECNICA DELLE MARCHE

Introduction | Powder Bed Fusion

Powder Bed Fusion (PBF)

The thermal energy (from a **laser** or **electron beam**) selectively **fuses** regions of a **powder bed** in a layer-by-layer fashion, according to a CAD model



PROBLEMS

- High surface roughness
- Porosity (poor densification)
- Residual thermal stresses
- Heterogeneous microstructure
- Feedstock cross-contamination

Introduction | Cross-Contamination

Additive Manufacturing 24 (2018) 13–19

Contents lists available at ScienceDirect

Additive Manufacturing

journal homepage: www.elsevier.com/locate/addma

Repercussions of powder contamination on the fatigue life of additive manufactured maraging steel

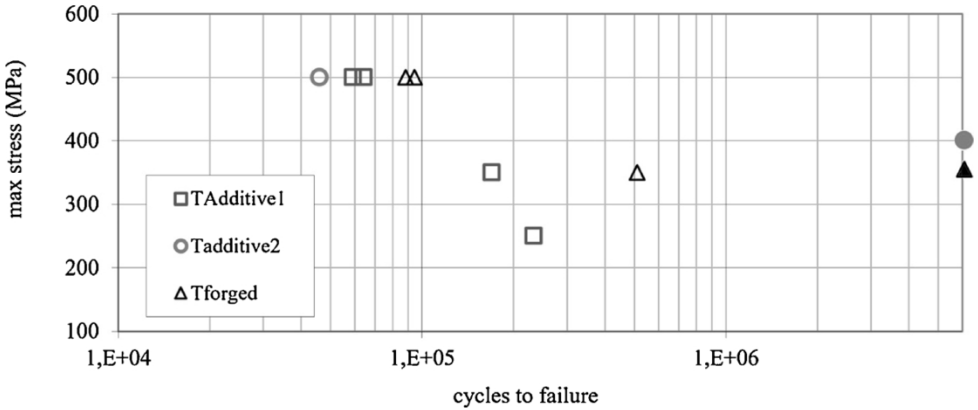
A. Gatto, E. Bassoli, L. Denti*

Specimens considered in the experimental plan.

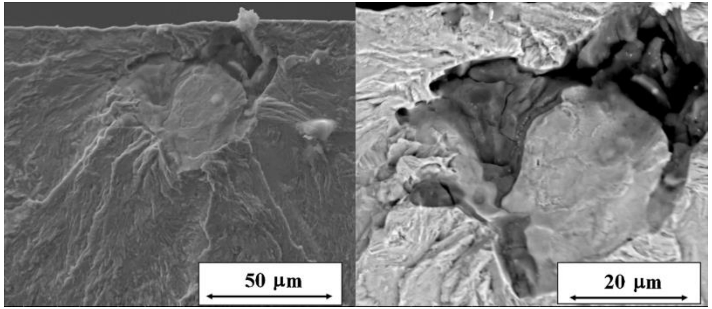
Designation	No. of specimens	Material	Technology	Standard	Age hardening
$T_{Additive1}$	4	18Ni-300	PBF	ASTM E466	6 h at 490 °C
$T_{Additive2}$	2				
T_{Forged}	5		FORGING		

Additive1 = powder from lot 1.
Additive2 = powder from lot 2.

AXIAL FATIGUE LIFE
const. amplitude, R=0



SEM



SE BSE

Ti- and Al-based oxides

H2020 DREAM Project

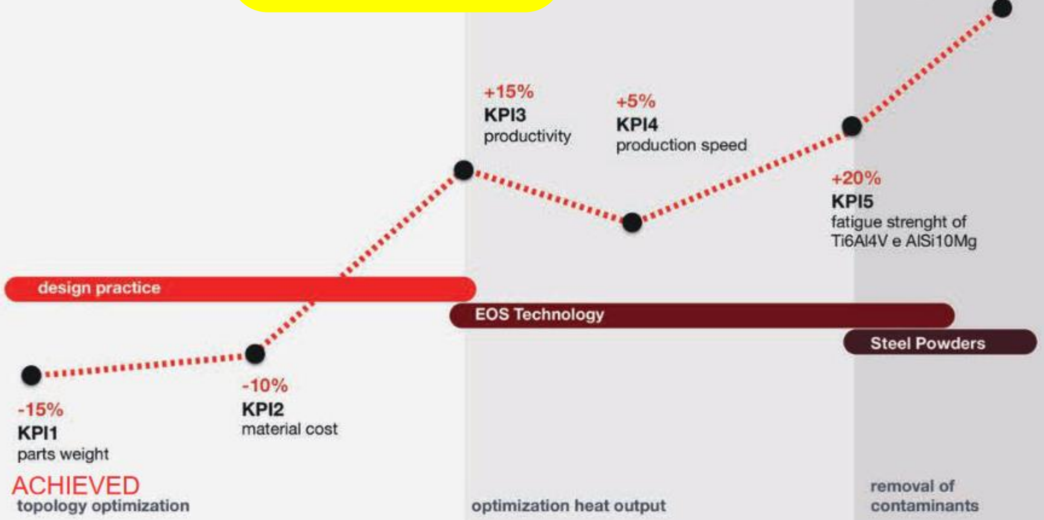
KPI1
at least 15% weight reduction of parts optimized in topology and for Additive Manufacturing

KPI2
reduction of more than 10% of material cost

KPI3
increase over 15% of productivity achieved for PBF process

KPI4
increase over 5% of production speed of laser PBF systems

KPI5
increase above 20% of fatigue strength of metal parts produced with laser PBF (up to +120%)



Business Cases

Medium size prosthetic titanium components

Lightweight automotive Aluminium components

Mould inserts

www.dream-euproject.eu

This project has received funding from the European Union's Horizon2020 research and innovation programme under grant agreement n. 723699

Introduction | Statistical Quantification

EURO PM2018 CONGRESS & EXHIBITION — EPMA 14 - 18 October 2018 Bilbao, Spain

Experimental | Statistical Procedure

1. Put a known quantity of powder on a stub for SEM;
2. COMPOSITION CHECK → Acquire a minimum of 3 EDS microanalysis on large areas (200x)
3. SEM (BSE)-EDS (maps) inspection → At least 50 fields (500x) of the stub area

Statistical Procedure

4. Count the contaminant particles (n) per inspected area
5. Estimate the total number of contaminant particles (TCP) per stub (stub area=122.6 mm^2)
6. Estimate the total number of particles per stub (TOT) by the ImageJ analysis software (average TOT≈10^5 particles per stub)
7. **Calculate contamination** (CC) from the above quantities (CC=TCP/TOT)

Three samples characterized for each condition

D.C. Montgomery, G.C. Runger, Applied Statistics and Probability for Engineers, 6th ed., Wiley, 2014, pp. 792

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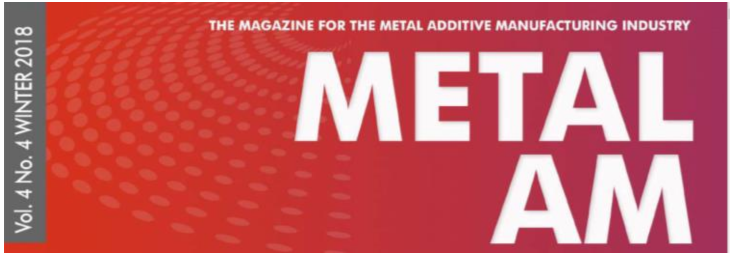


E. Santecchia, P. Mengucci, A Gatto, E. Bassoli, L. Denti, F. Bondioli, G. Barucca – EuroPM18 Proceedings

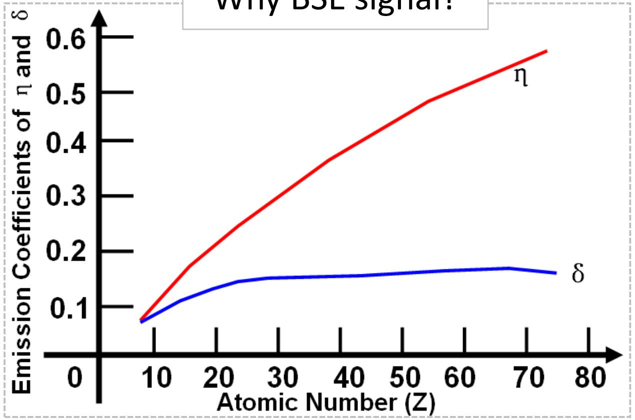
What is next?

- Faster procedures for cross-contamination estimation (less than 50 micrographs!)
- Automatic detection -> Machine Learning

Experimental | Characterization Techniques

- **Scanning Electron Microscopy (SEM)**
  Zeiss Supra 40 (Field emission) SE-ET, SE-in lens, BSE
- **Energy Dispersive Spectroscopy (EDS)**
  Bruker Quantax Z200, quantitative analysis software
- **X-ray Diffraction**
  Bruker D8 Advanced, Cu-kα, Bragg-Brentano geometry
- **ImageJ Software**

Why BSE signal?



SEM Working Parameters

Detector	Backscattered Electrons (BSE)
Aperture	60 µm
Magnitude	500x
Working Distance	8.3 mm
Accelerating Voltage	15 keV

Experimental |Powder Samples

Virgin Powders

- Maraging Steel (EOS MS1*) → 18% Ni Maraging 300 (US) |1.2709 (EU)
- Ti6Al4V (EOS Titanium Ti64*) → ISO 5832-3, ASTM F1472, and ASTM B348

Contaminated Samples

Sample	Virgin Powder	Controlled Contamination	
		Type	Quantity [wt.%]
MS+0.5Ti64	MS1	Ti64	0.5
MS+1Ti64	MS1	Ti64	1
Ti64+0.5MS	Ti64	MS	0.5
Ti64+1MS	Ti64	MS	1

**EOS GmbH Electro Optical Systems (www.eos.info)*

Contaminated batch: 5 g

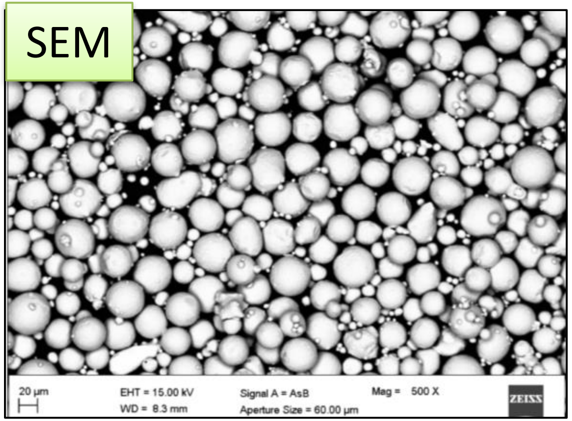
Density (EOS Datasheet)

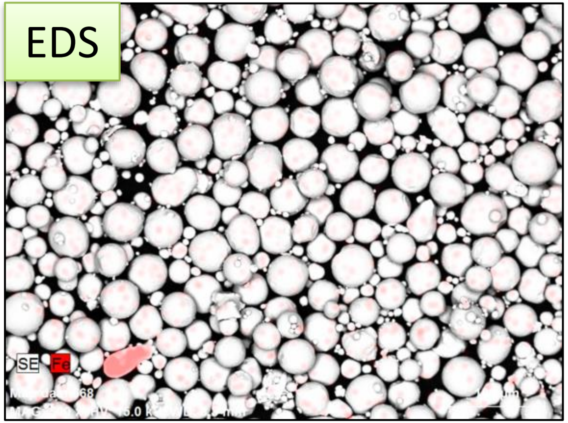
MS1 → 8.0-8.1 g/cm^3

Ti64 → 4.41 g/cm^3

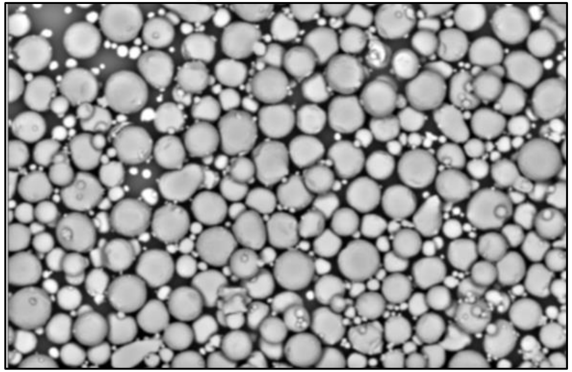
3 Powder samples analyzed for each condition
5 Micrographs used to quantify the contamination

Experimental | SEM image analysis - ImageJ

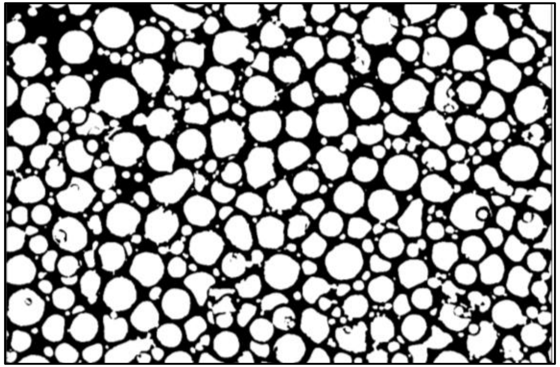




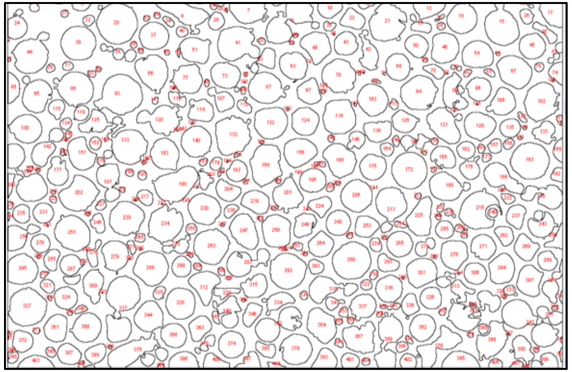
FFT Bandpass Filter



Threshold



Analyze Particles



Experimental | Statistical Quantification Methods

Contrast Ratio

«Analyze Particles»
Area occupied by the particles (virgin and contaminants)

+

EDS elemental map

Weight Ratio

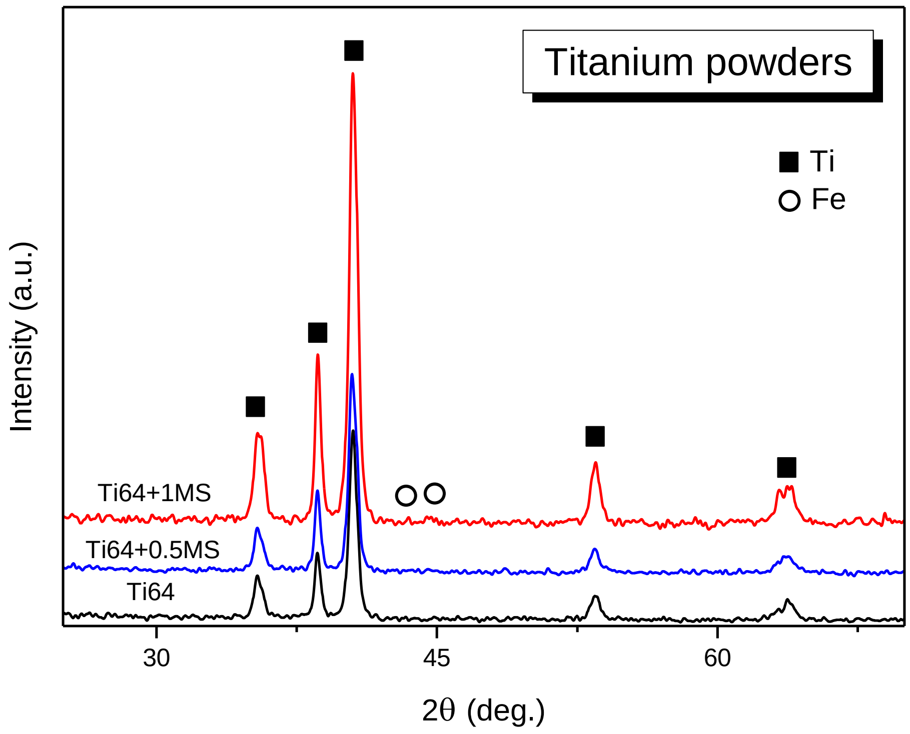
«Analyze Particles»
Radius of each particle in the micrograph → Volume

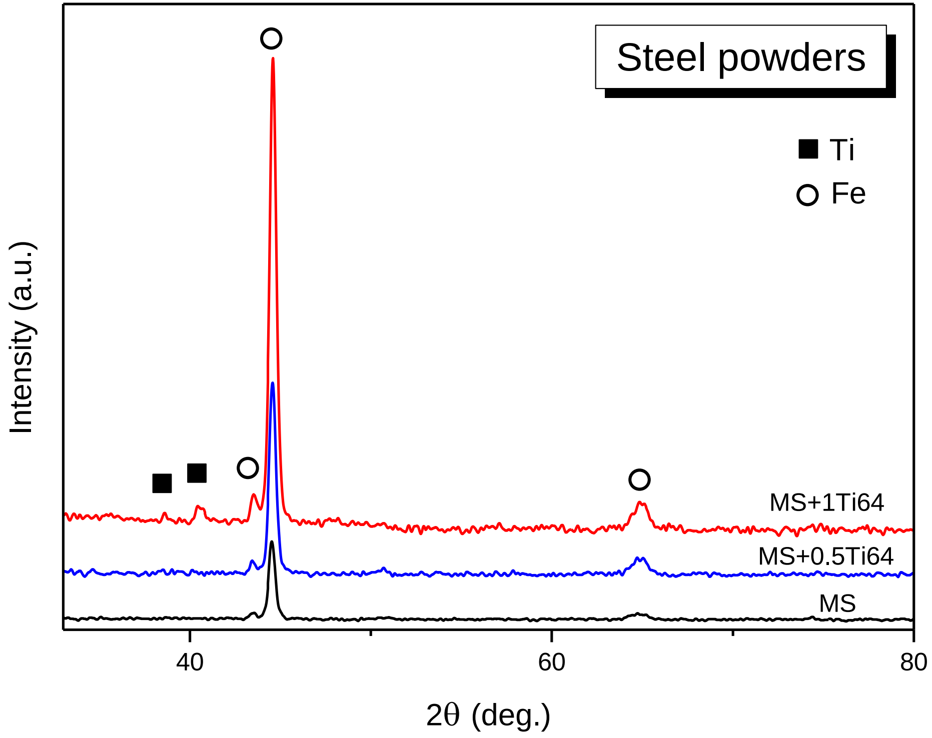
+

EDS elemental map

Hypothesis: Sphericity of contaminants

Results | X-ray Diffraction



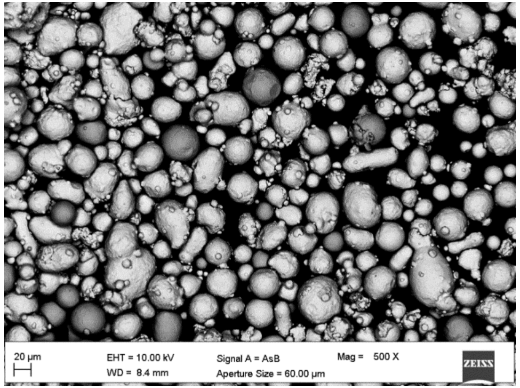


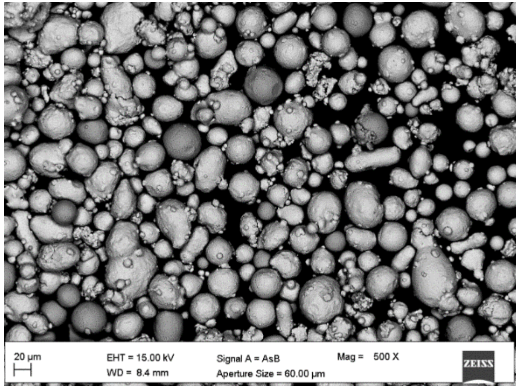
Cross-contamination detected in the MS+1Ti64 sample only!

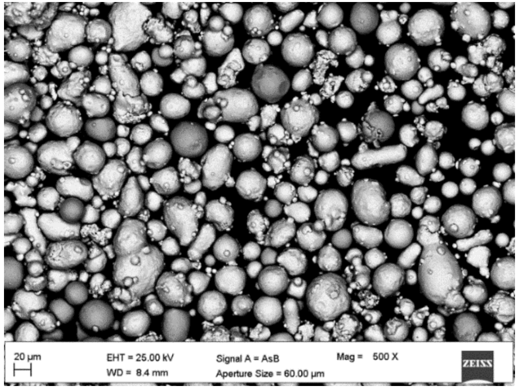
Results | SEM KeV Selection

10 keV | 15 keV | 25 keV

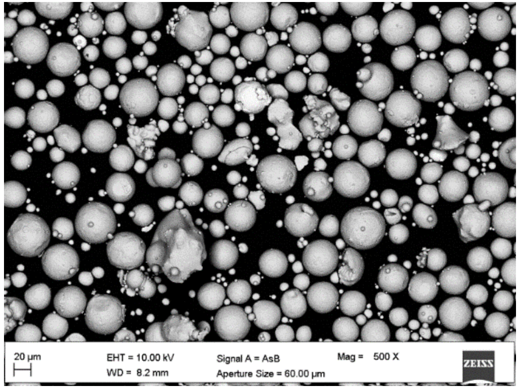
MS+1Ti64

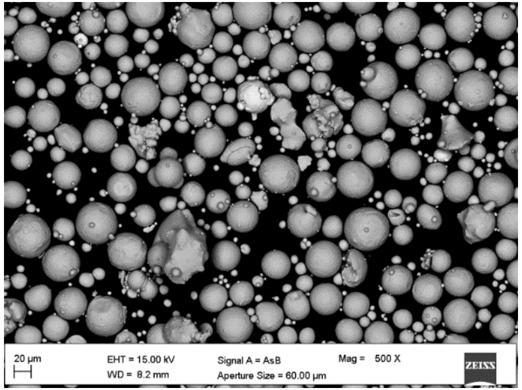


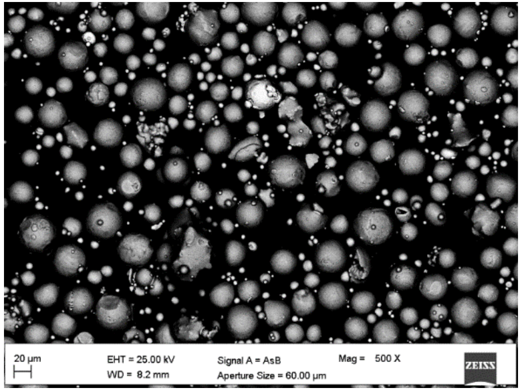




Ti64+1MS

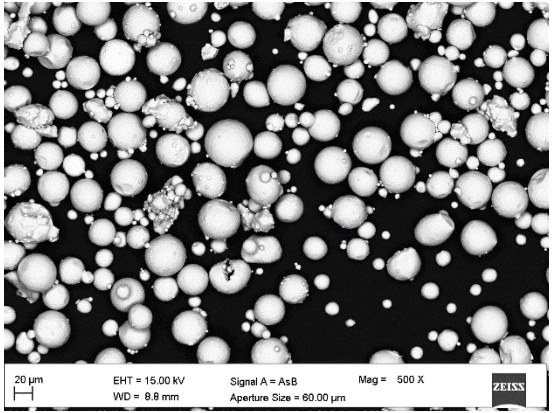


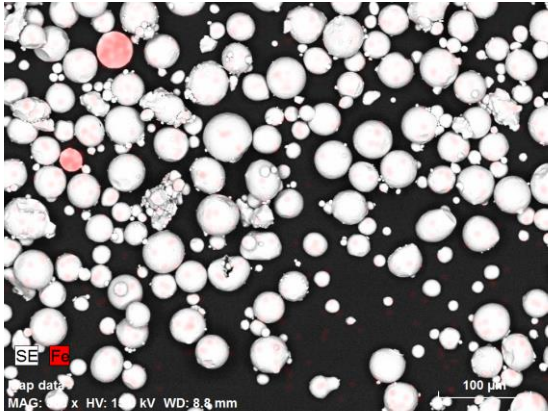




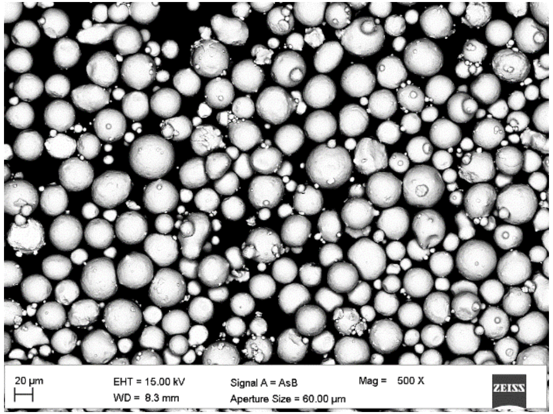
Results | Ti64 virgin powder

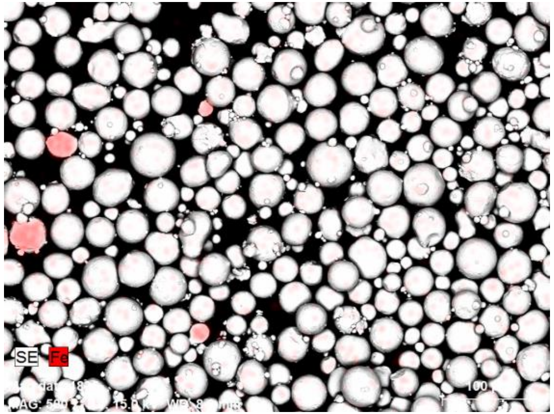
Ti64+0.5MS





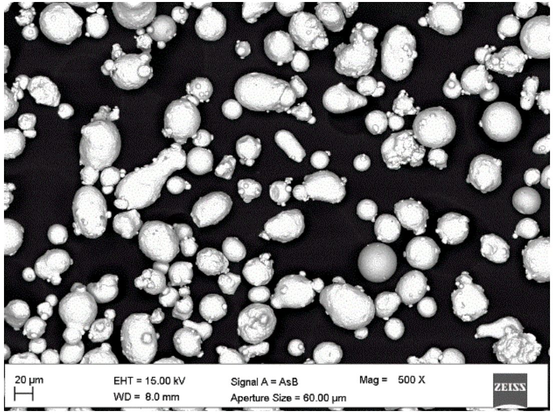
Ti64+1MS

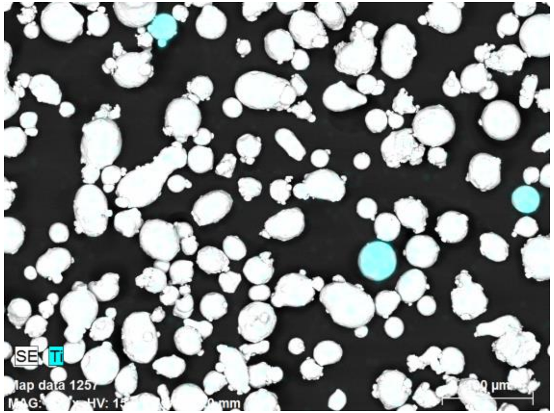




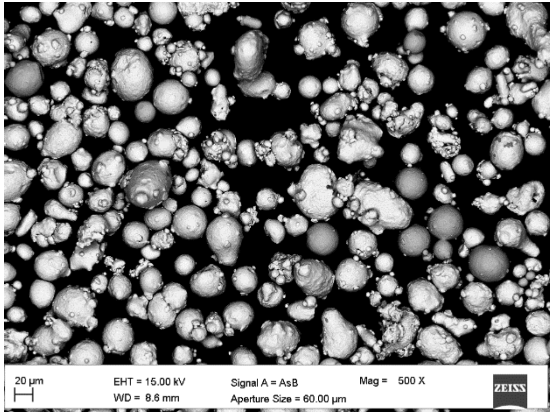
Results | MS virgin powder

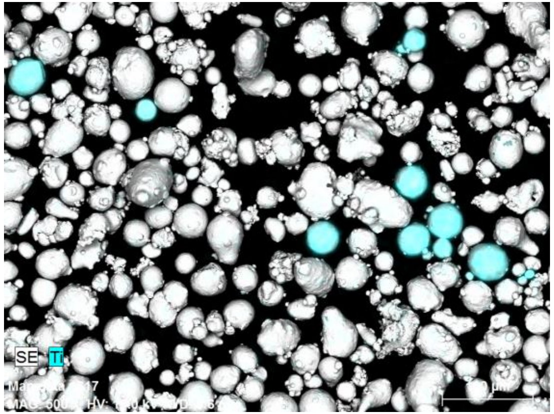
MS+0.5Ti64





MS+1Ti64





Results | Cross-Contamination Quantification

Sample Name	Contrast Ratio (%)	Weight Ratio (%)
MS+0.5Ti64	2.0 ± 0.2	2.5 ± 0.6
MS+1Ti64	3.6 ± 0.3	3.1 ± 0.8
Ti64+0.5MS	1.1 ± 0.4	1.5 ± 0.6
Ti64+1MS	2.0 ± 0.2	3.2 ± 0.7

Purely phenomenological

Linked with the physical properties of the powders

Coherence with the level of introduced contamination!

Conclusions

- Cross-contamination is hardly detectable by conventional XRD equipment
- The weight ratio procedure overestimates cross-contamination amounts
- Results of the contrast ratio procedure are in good agreement with the ratio of introduced cross-contamination amounts
- By tuning the SEM parameters it is possible to optimize the information of the micrographs for machine learning

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HORIZON 2020

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Research on Additive Manufacturing

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