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Development of a reliable method for contamination detection in raw metal powders for additive manufacturing

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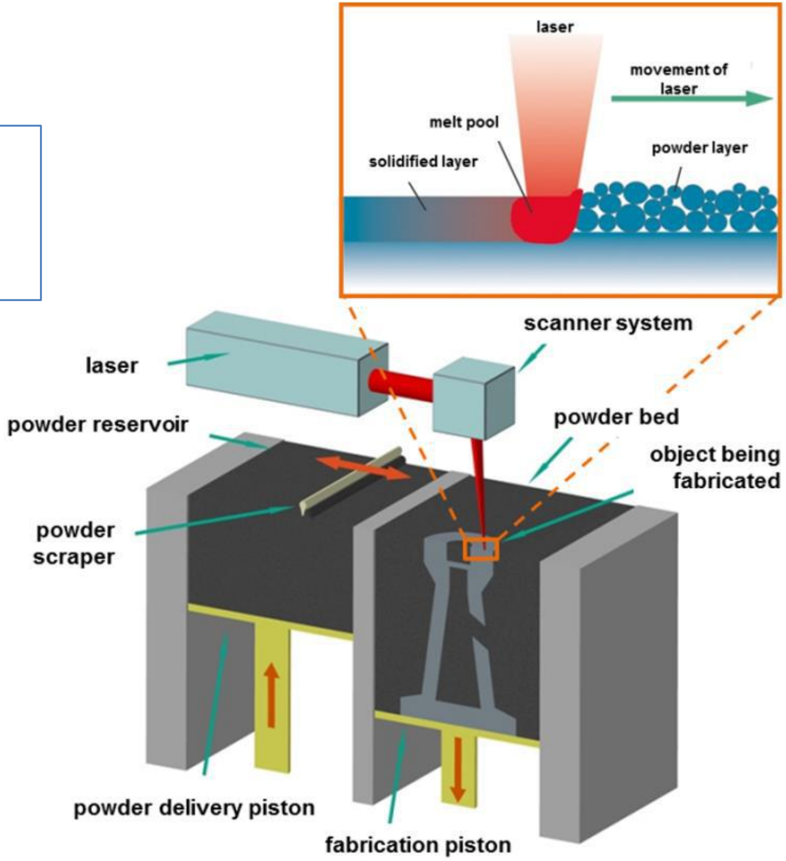
UNIVERSITÀ POLITECNICA DELLE MARCHE

INSTM

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

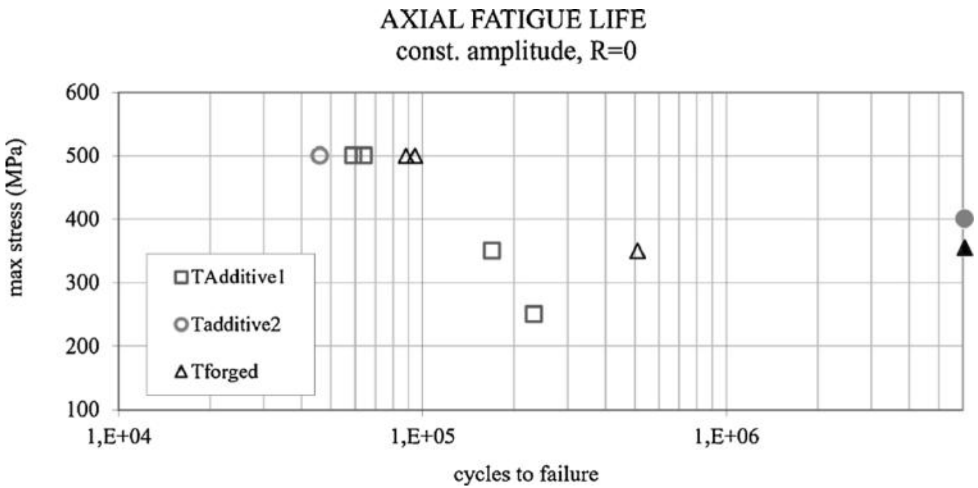
Repercussions of powder contamination on the fatigue life of additive manufactured maraging steel

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

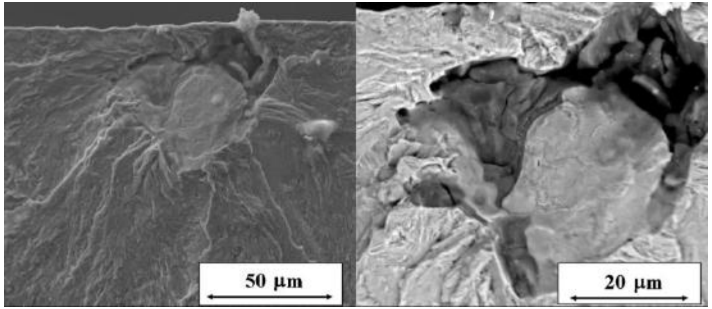
Specimens considered in the experimental plan.

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.



SEM



SE BSE

Ti- and Al-based oxides

H2020 DREAM Project

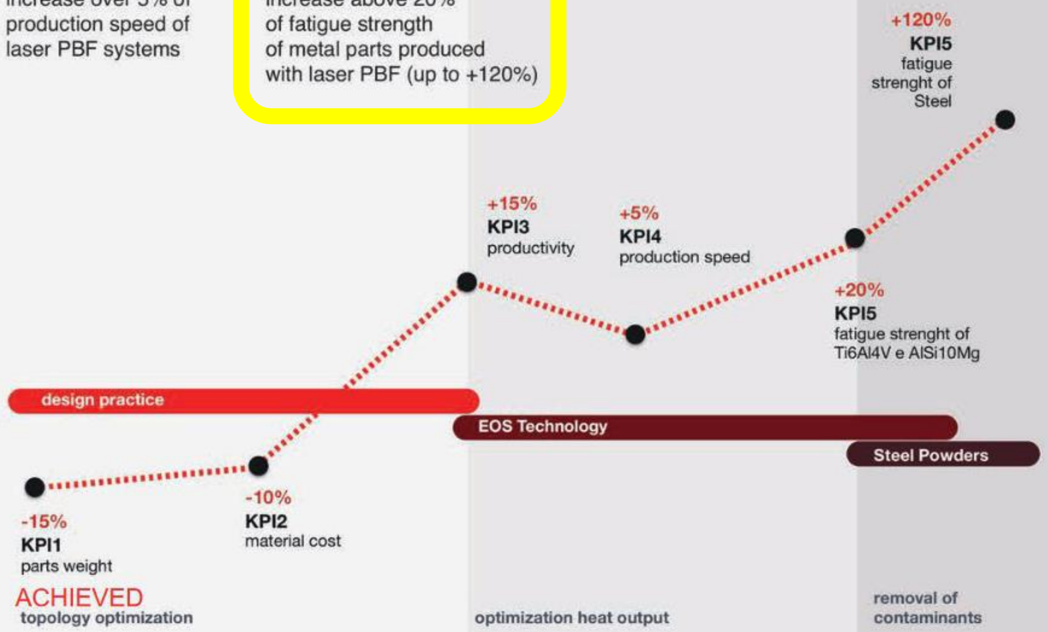
KPI1
at least 15% weight reduction of parts optimized in topology and for Additive Manufacturing

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

ADLER ORTHO FRANCE

beWARRANT

eos

POLY-SHAPE

MIND.4D

www.dream-euproject.eu

PHOTONICS21

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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

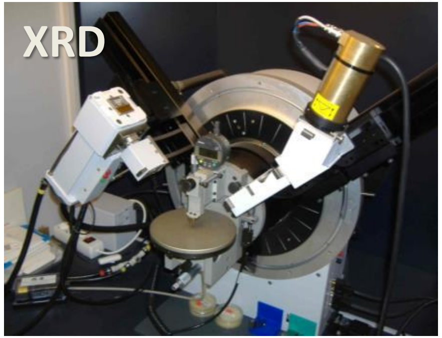
Contaminated Samples

Contaminated batch: 100 g

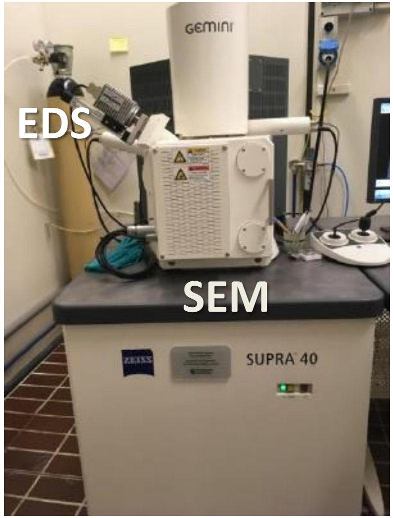
Sample Name	Virgin Powder	Controlled Contamination		Possible Contamination Source
		Type	Quantity [wt.%]	
MS1_Ti64	MS1	Ti64	0.5	Contamination through sieving equipment, tools, gloves or AM machine that are previously used with Ti64
MS1_Oxi	MS1	TiO_2, Al_2O_3	< 0.5	Production batch with titanium oxide and aluminium oxide inclusions
Ti64_MS1	Ti64	MS1	0.5	Breakage of the steel recoater blade or contamination from AM machine
Ti64_ZrO_2	Ti64	ZrO_2	0.5	Breakage of ceramic recoater blade

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

Experimental | Characterization Techniques



- **X-Ray Diffraction (XRD)**
  Bruker D8 Advance, Cu-kα, Bragg-Brentano geometry
- **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



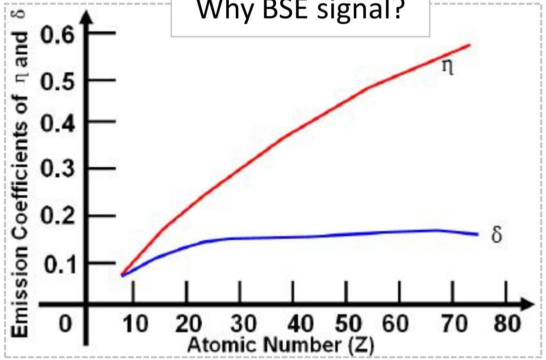
SEM Working Parameters

Aperture	60 µm
Detector	Backscattered Electrons (BSE)
Magnitude	200x (comp. check)
	500x (statistics)
Working Distance	8.3 mm
Accelerating Voltage	15 keV (statistical procedure)
	20 keV (comp. check)

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

Why BSE signal?



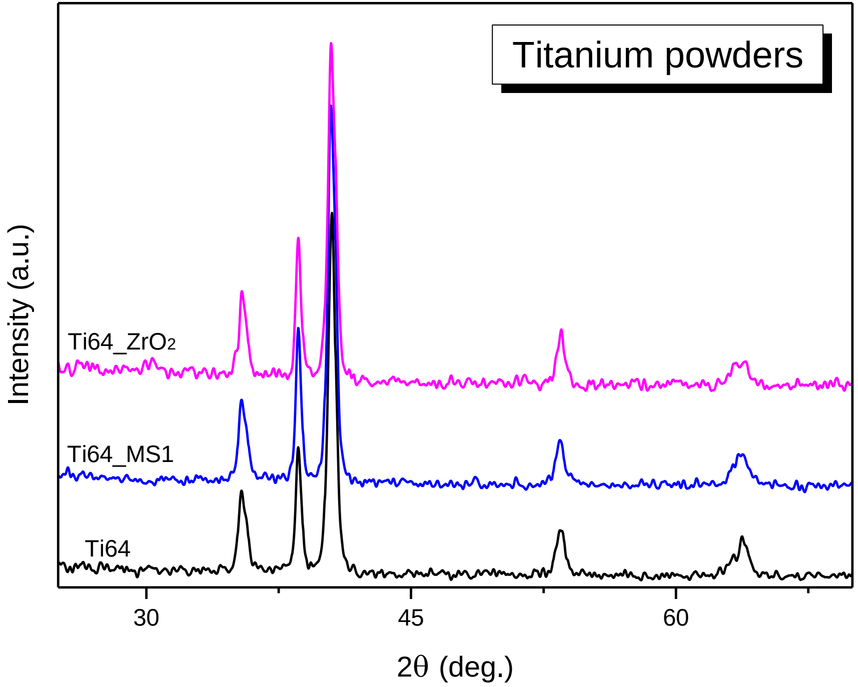
Statistical Procedure

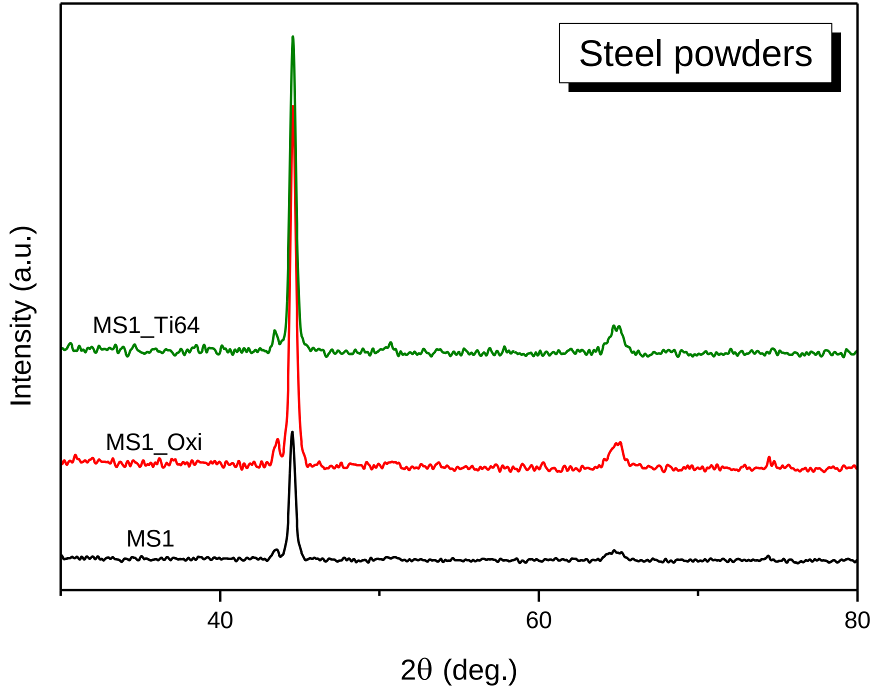
4. Count the contaminant particles (n) per inspected area
5. Estimate the total number of contaminant particles (TCP) per stub (stub area=122.6 mm^2)
6. Estimate the total number of particles per stub (TOT) by the ImageJ analysis software (average TOT≈10^5 particles per stub)
7. **Calculate contamination**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

Results | X-Ray Diffraction





Absence of reflections correlated to cross-contamination

Results | Composition Check

All the chemical compositions are in wt.%

Maraging Steel

	Ni	Co	Mo	Ti	Al	Cr	Cu	C	Mn	Si	P	S
MS1	17-19	8.5-9.5	4.5-5.2	0.6-0.8	0.05-0.15	≤ 0.5	≤ 0.5	≤ 0.03	≤ 0.1	≤ 0.1	≤ 0.01	≤ 0.01
MS1_Ti64	15.4 ± 0.3	10.8 ± 0.1	3.5 ± 0.2	1.5 ± 0.2	0.05 ± 0.01	0.15 ± 0.03	0.14 ± 0.06	-	0.08 ± 0.04	ND	ND	0.06 ± 0.04
MS1_Oxi	15.3 ± 0.2	11.2 ± 0.1	3.9 ± 0.2	0.9 ± 0.1	0.06 ± 0.03	0.25 ± 0.06	0.11 ± 0.04	-	ND	ND	ND	ND

High wt.% of Ti

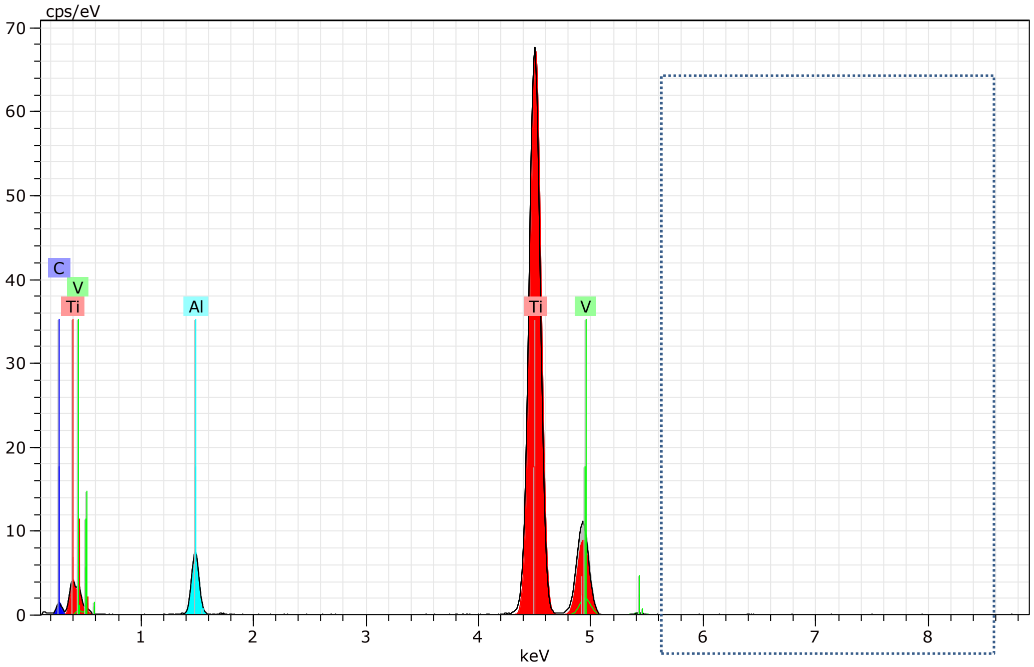
Ti6Al4V

	Al	V	Zr	O	N	C	H	Fe	Y
Ti64	5.50-6.75	3.50-4.50	-	< 0.20	< 0.05	< 0.08	< 0.015	< 0.30	< 0.005
Ti64_MS1	5.4 ± 0.1	3 ± 0.1	-	ND	ND	-	ND	ND	ND
Ti64_ZrO2	5.6 ± 0.3	3 ± 0.1	0.3 ± 0.1	ND	ND	-	ND	ND	ND

Fe not detected

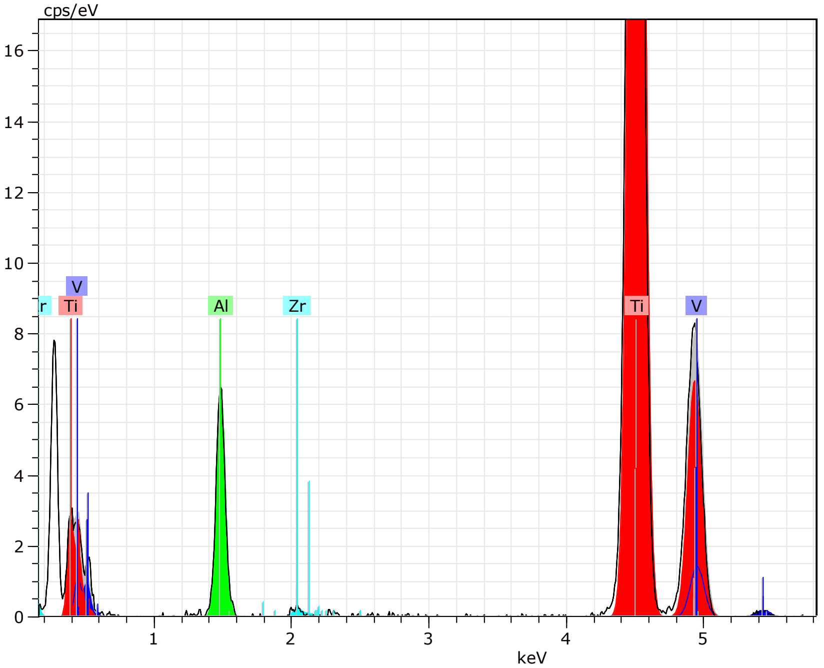
Results | Composition Check

Ti64_MS1



Fe kα 6.4 keV /Fe kβ 7.06 keV

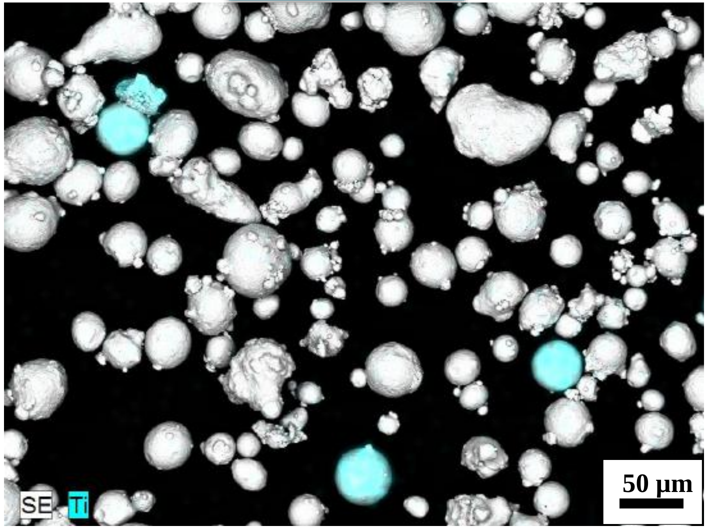
Ti64_ZrO2



Results | Cross-Contamination Quantification

Maraging Steel

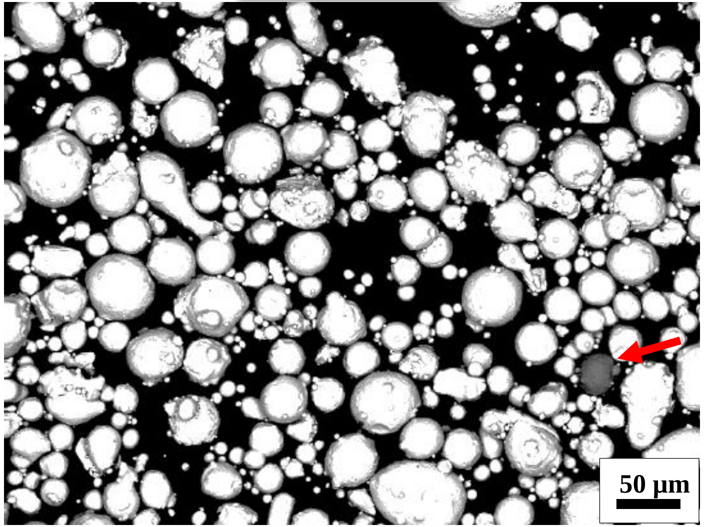
MS1_Ti64



SEM-BSE + EDS map

Ti64 highlighted although titanium is also present in the MS1 powder

MS1_Oxi

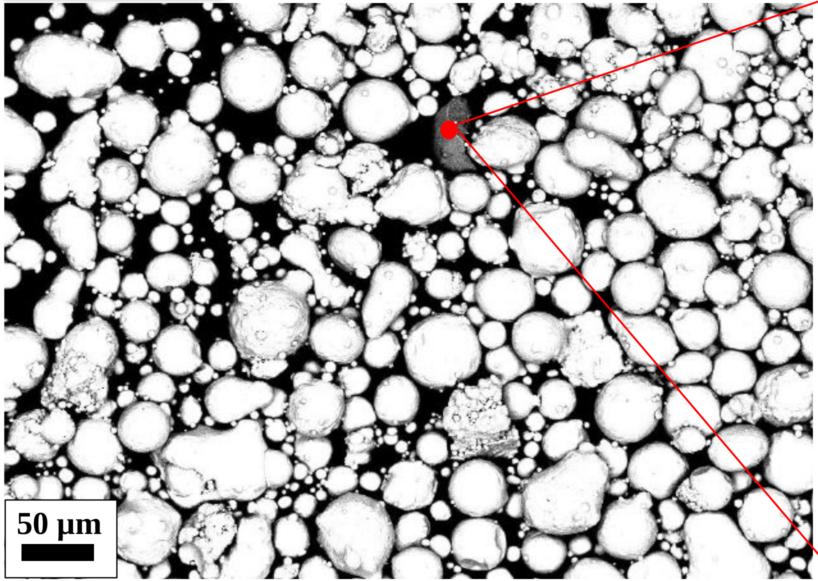


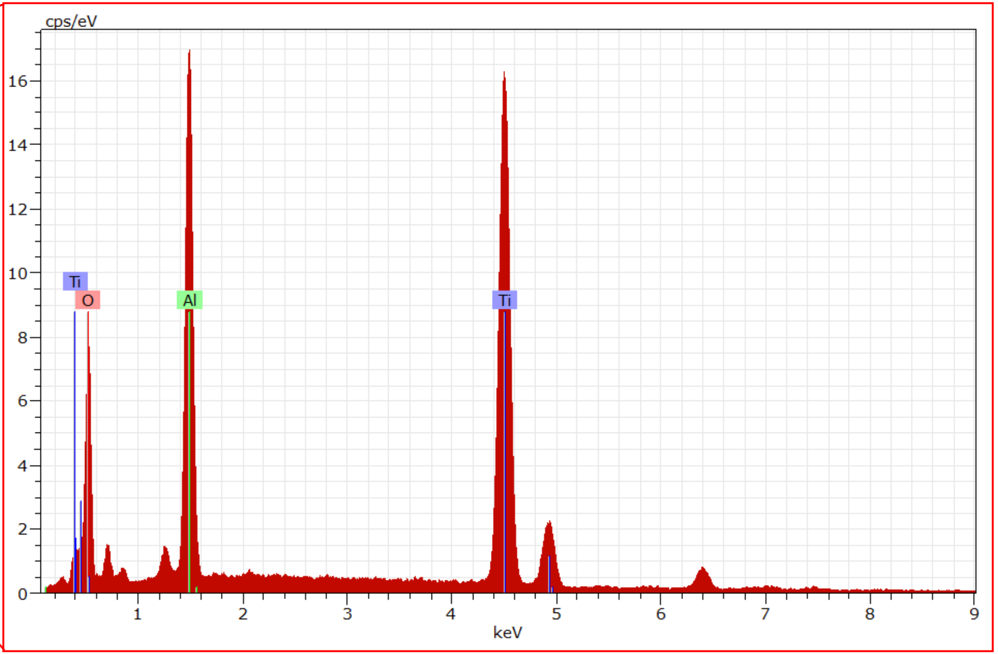
SEM-BSE

High contrast -> Non need for EDS maps

Results | Cross-Contamination Quantification

MS1_Oxi



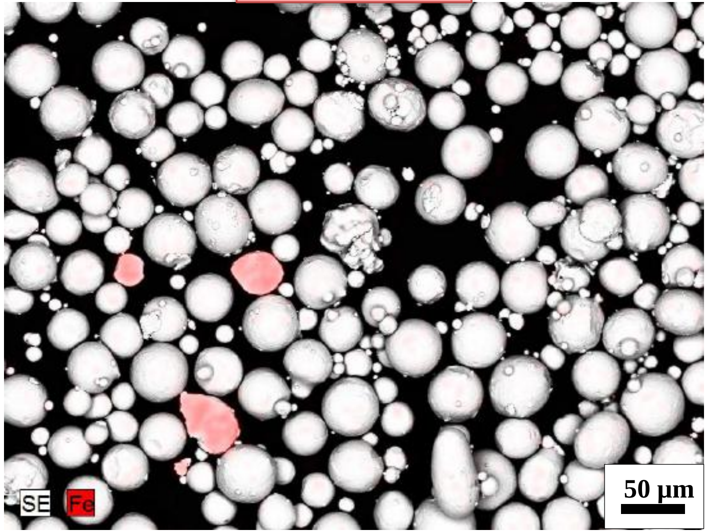


Composition verified to check the presence of other cross-contamination sources

Results | Cross-Contamination Quantification

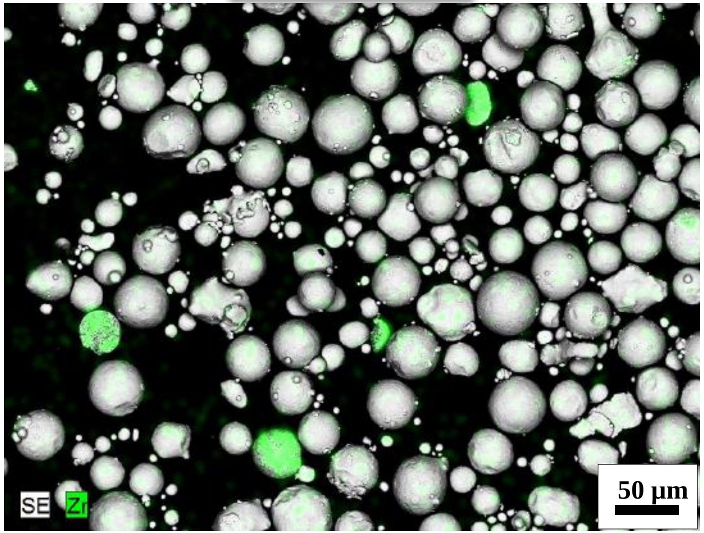
Ti6Al4V

Ti64_MS1



Particles coherent with the maraging steel raw powder

$Ti64\_ZrO_2$



Fragments of various shapes

Results | Cross-Contamination Quantification

Statistics results

Sample	Average Calculated Contamination (10^{-3})
MS1_Ti64	7 ± 1
MS1_Oxi	1.8 ± 0.5
Ti64_MS1	2.7 ± 0.2
Ti64_ZrO2	6 ± 2

Density (EOS Datasheet)

MS1 → 8.0-8.1 g/cm^3

Ti64 → 4.41 g/cm^3

To obtain the same amount of 0.5 wt% of cross-contamination, a lower number of MS1 particles is required

Conclusions

- Density is the key for cross-contamination detection via SEM-EDS
- EDS on large area fails to detect the contamination when the contaminant has a high density (low number of particles, low volume, low signal)
- Low levels of contamination are not detectable with XRD
- The statistical treatment of the collected data showed a good agreement with the physical properties of the powders

The implementation of other structural characterization techniques is crucial to improve the quality of the raw materials and allow to develop a standard practice in additive manufacturing

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HORIZON 2020

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