DREAM FINAL WORKSHOP

In collaboration with

ADDITIVE MANUFACTURING R&I GROUP

@ formnext

Enhancing metal AM reliability through contamination control

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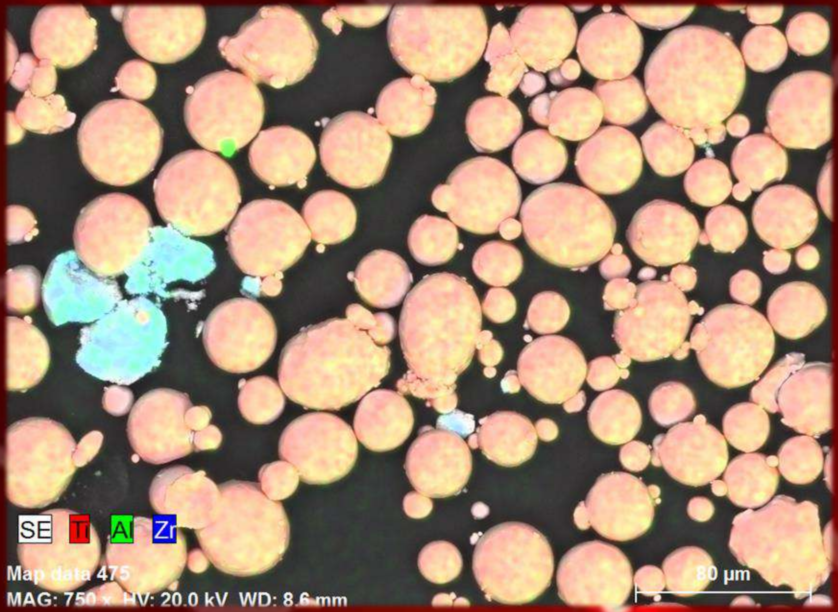
Cross-Contamination in LPBF

Accidental mixing of different raw metal powders for additive manufacturing

- ❖ Improper material handling
- ❖ Different powders used in the same equipments (AM machines, sievings…)
- ❖ Insufficient cleaning

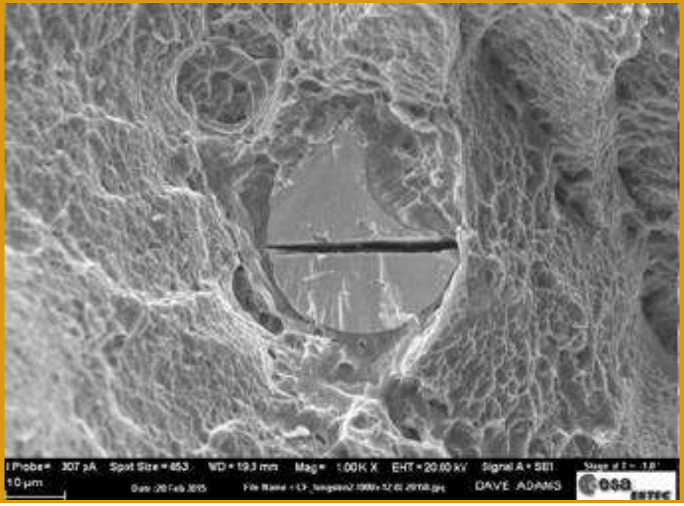


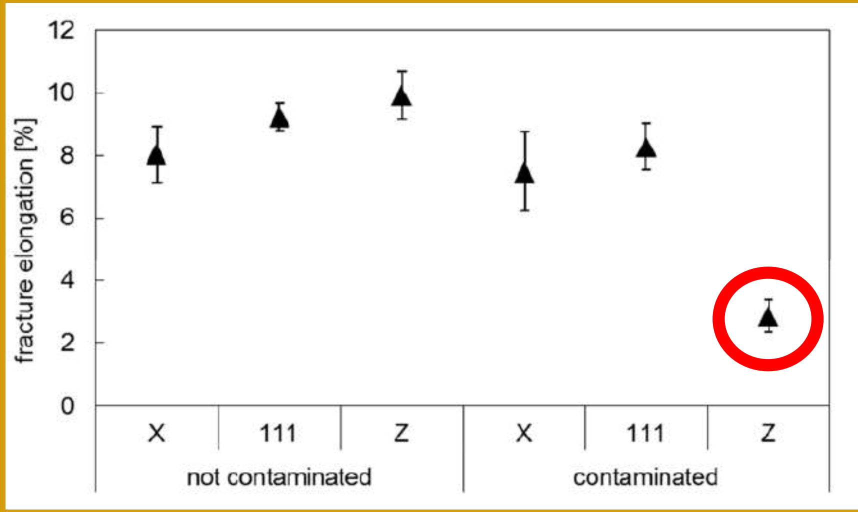
How can we enhance the LPBF process reliability?

Enhancing metal AM reliability

➢ AWARENESS

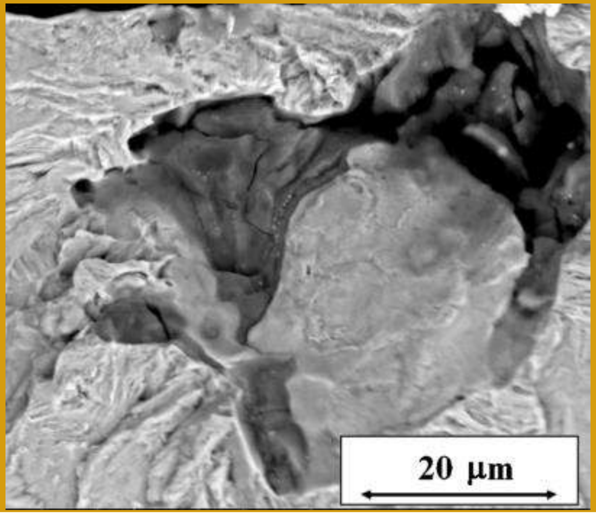
W particles in Ti64

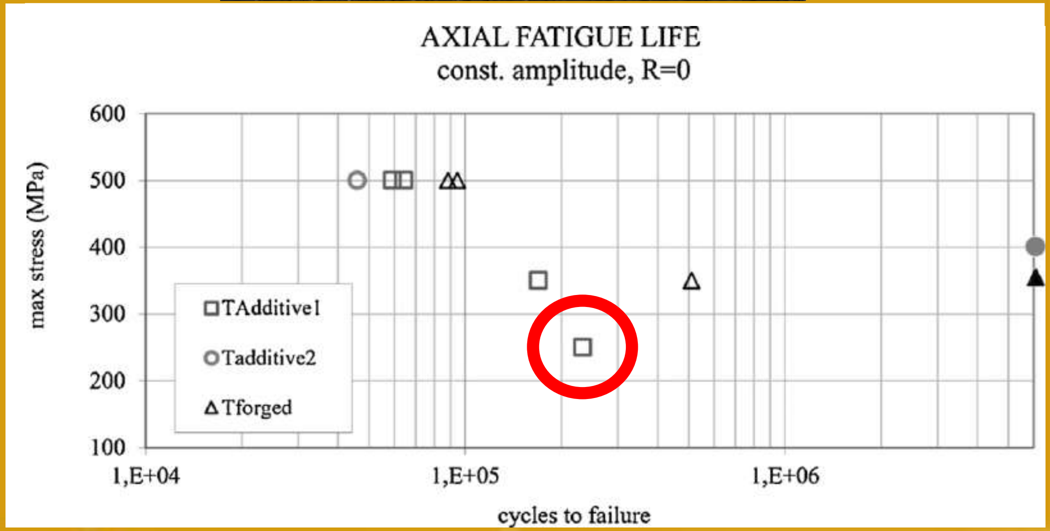




Brandão et al., Materials 19 (2017) 522

Ti- and Al- oxides in MS



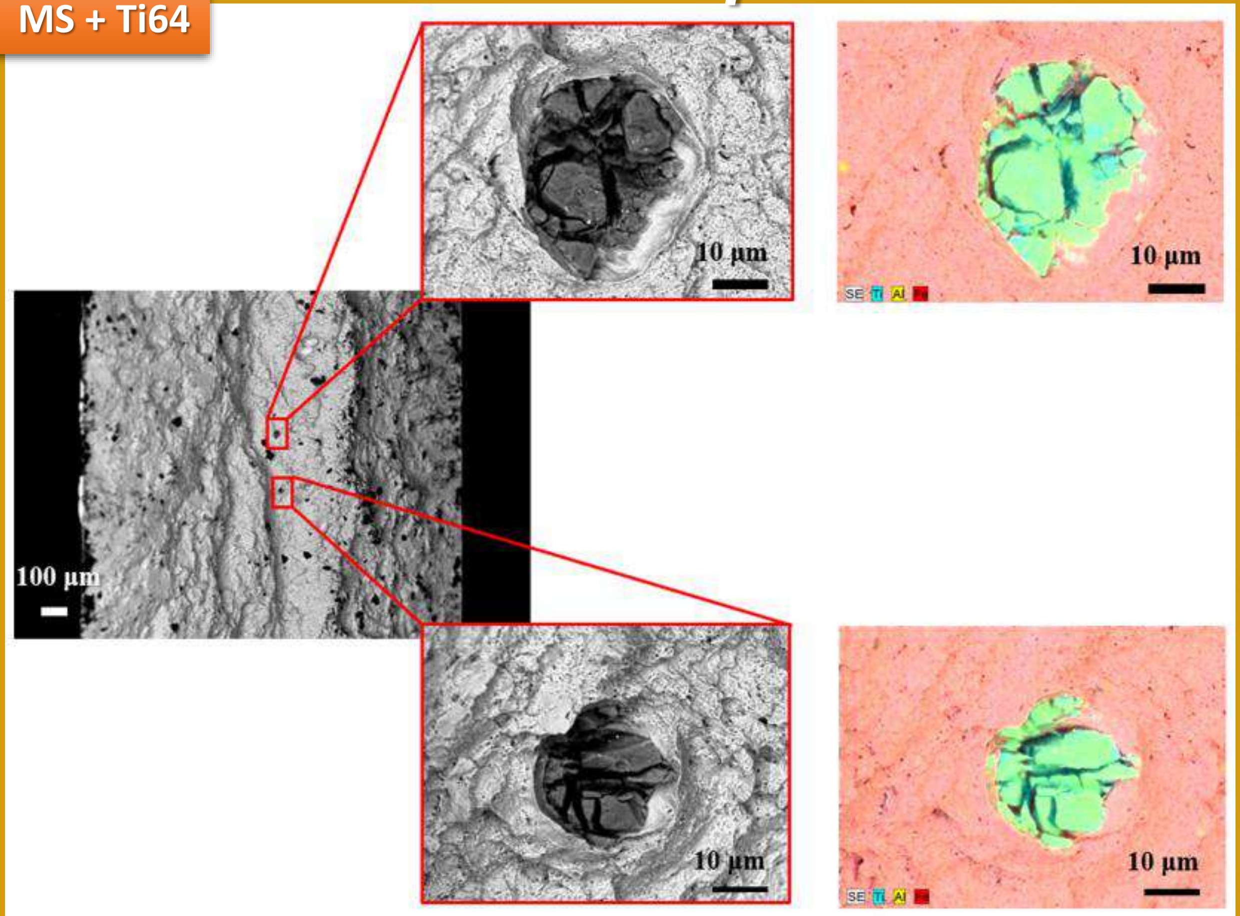


Gatto et al., Additive Manufacturing 24 (2018) 13-19

➢ AWARENESS

Solidus-Liquidus

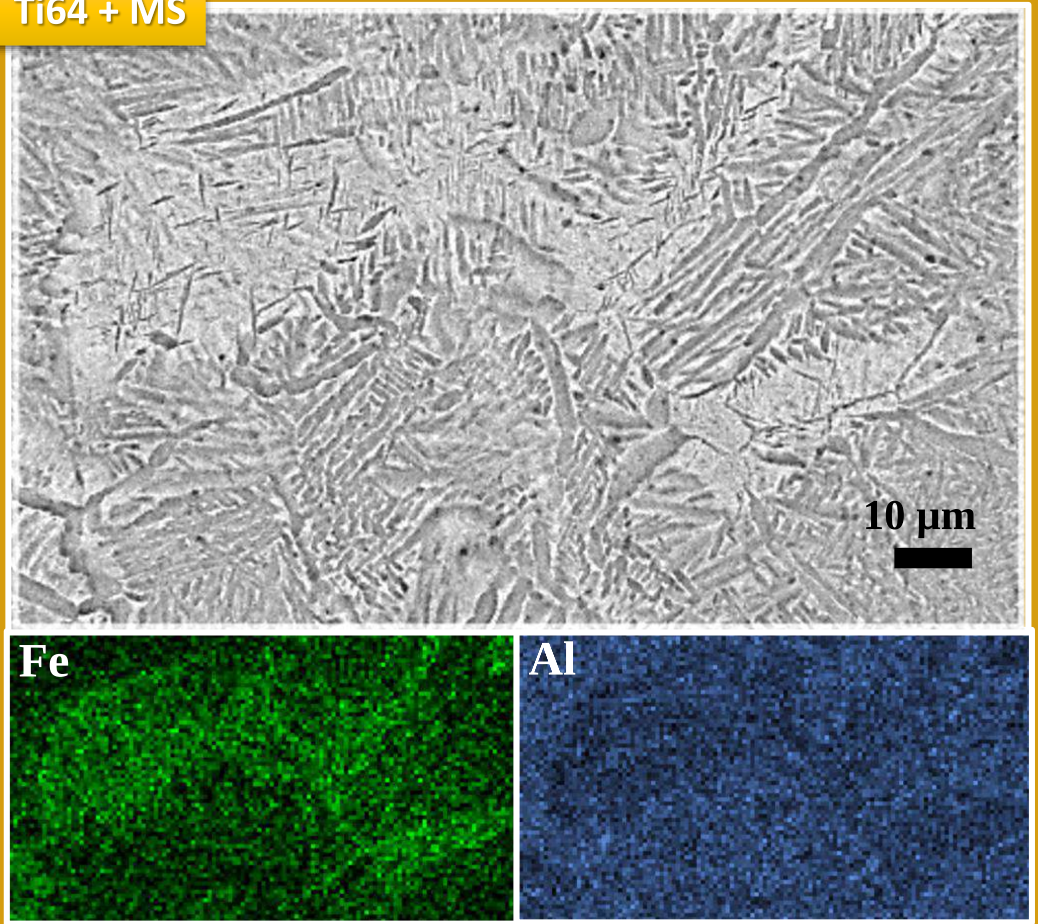
MS + Ti64



Contamination has a melting point higher than the matrix

Liquidus-Liquidus

Ti64 + MS



Contamination has a melting point lower than the matrix

Santecchia et al., Materials 12 (2019) 2342

AWARENESS

Powder batches (100 g) with controlled cross-contamination prepared by EOS GmbH

	Powder	Controlled Contamination	Possible Contamination Source	Comment
Steel	Maraging Steel	0.5 wt% Ti64	Contamination through sieving equipment, tools, gloves or AM machine that are previously used with Ti64.	-
	Maraging Steel	-	-	Production batch with Ti oxide and Al oxide inclusions.
Ti64	Ti64	0.5 wt% MS1	Breakage of the steel recoater blade or contamination from AM machine.	-
	Ti64	0.5 wt% ZrO_2	Breakage of ceramic recoater blade.	-
	Ti64	~0.2 wt% TiC	Contamination through sieving equipment, tools, gloves or AM machine that are previously used with Ti64.	-

Enhancing metal AM reliability

➢ METHODOLOGY

"The methods most commonly used in the certification of powders for AM proved to be insufficient for the detection of the cross contamination"

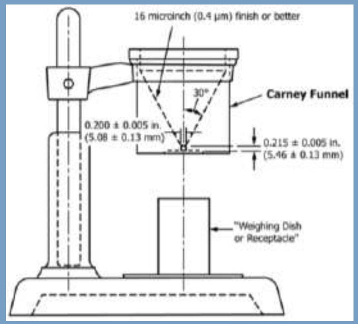
Brandão et al., Materials 19 (2017) 522

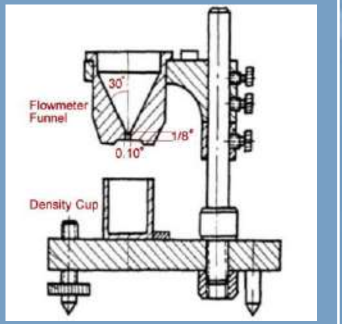
"Alternatives to conventional qualification methods must be found based upon validated models, probabilistic methods and part similarities among others"

Frazier, J Mater Eng Perform 23 (2014) 1917–1928

Standard Powder Characterization

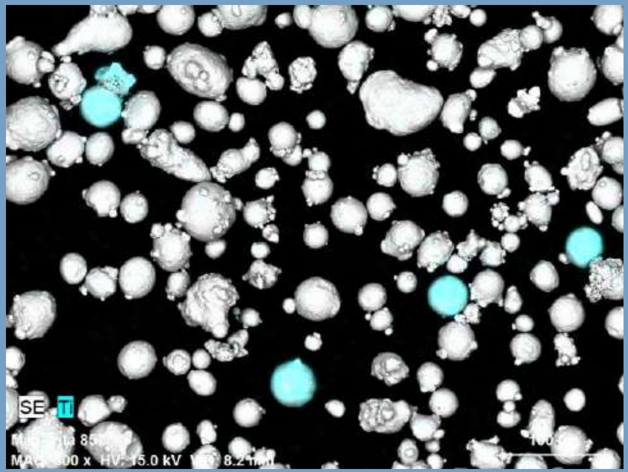
PSD





FLOW RATE

Scanning Electron Microscopy (SEM)



Energy Dispersive Spectroscopy (EDS)

Statistics

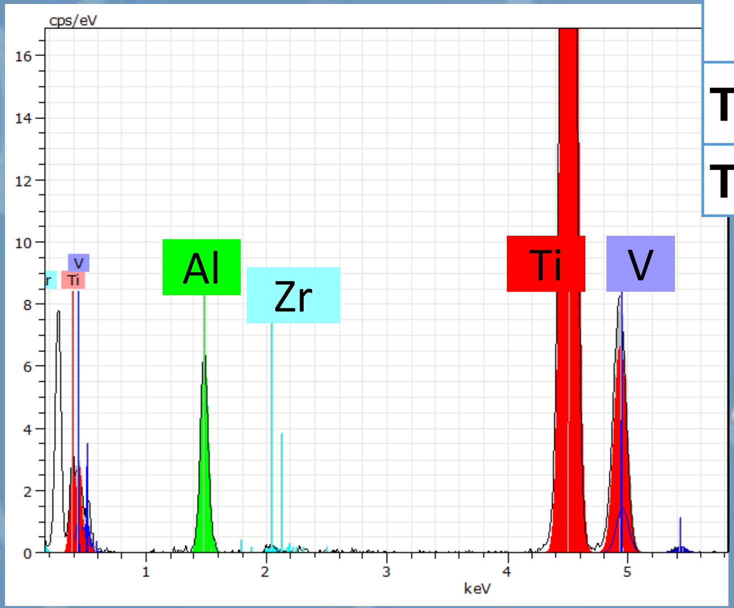
➢ METHODOLOGY

DREAM Developed protocol

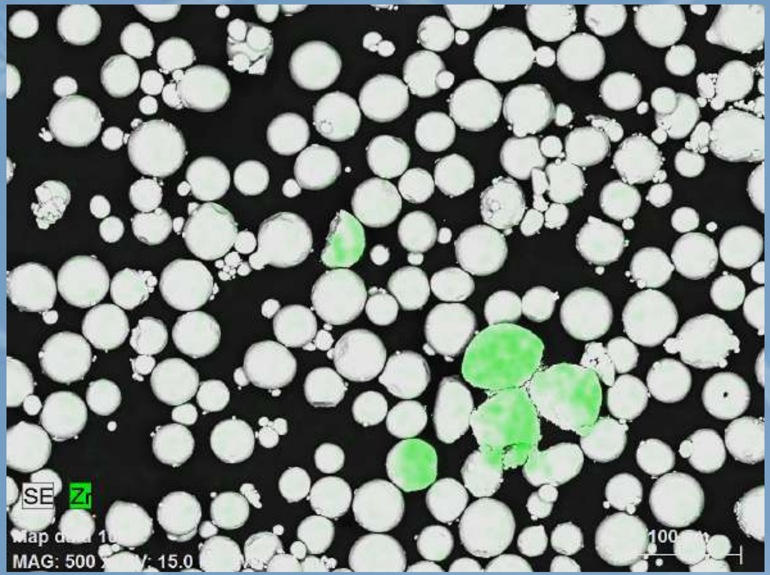
1) Sample a small amount of powder according to ASTM F3049-14 (standard B215)
2) Spread a fine layer of powder and attach it on 3 SEM stubs (graphite adhesive)

THEN FOR EACH STUB:

3) Verify the powder composition performing at least 3 EDS analysis on large areas (mag 200x)
4) SEM (BSE) signal inspection of at least 50 fields (mag 500x) of the stub area
5) If no clear composition-linked contrast is observed, perform a compositional map on each inspected area



	Al (wt.%)	V (wt.%)	Zr (wt.%)
Ti64	5.5 - 6.7	3.5 - 4.5	-
Ti64_ZrO$_2$	5.6 ± 0.3	3 ± 0.1	0.3 ± 0.1



➢ METHODOLOGY

DREAM Developed procedure

1. CALCULATED CONTAMINATION (CC) PROCEDURE

- ❑ Count the contaminant particles (n) per inspected area
- ❑ Estimate the total number of contaminant particles (TCP) per stub (stub area = 122.6 mm^2)
- ❑ Estimate the total number of particles per stub (TOT) by the ImageJ analysis software (~10^5)

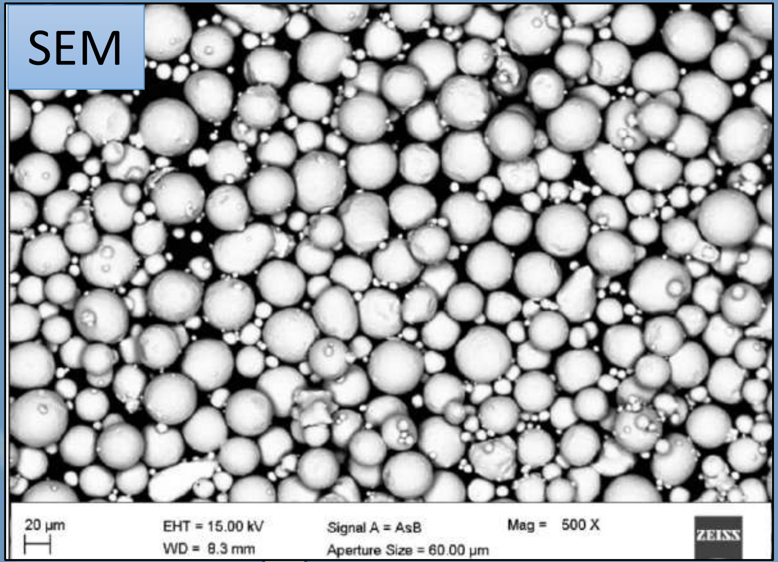
$$CC = \frac{Total\ contaminant\ particles\ (TCP)}{Total\ number\ of\ particles\ (TOT)}$$

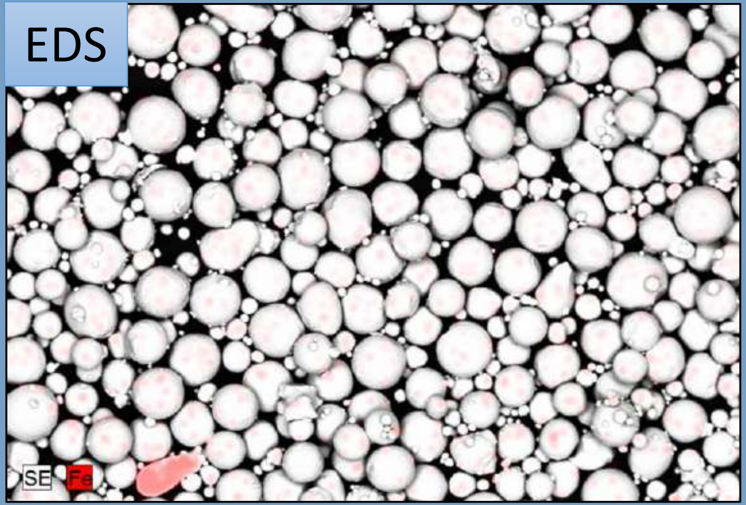
✓ Can be applied to contamination of any shape

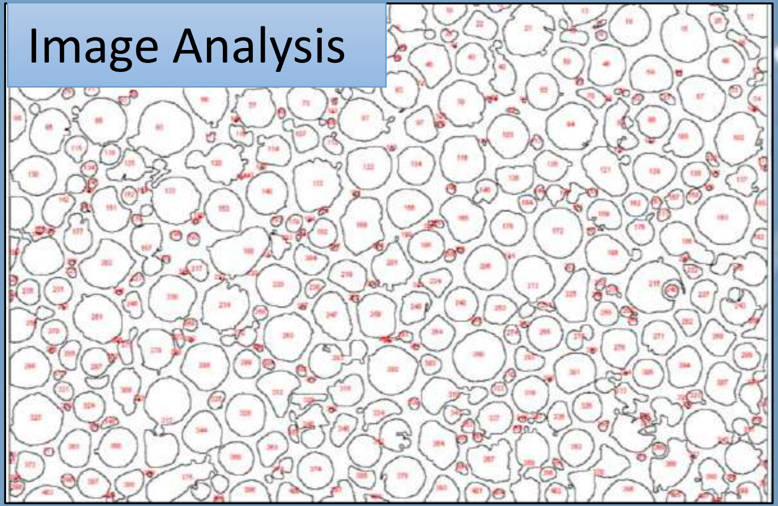
Santecchia et al., EuroPM18 Proceedings

➢ METHODOLOGY

2. IMAGE ANALYSIS PROCEDURE







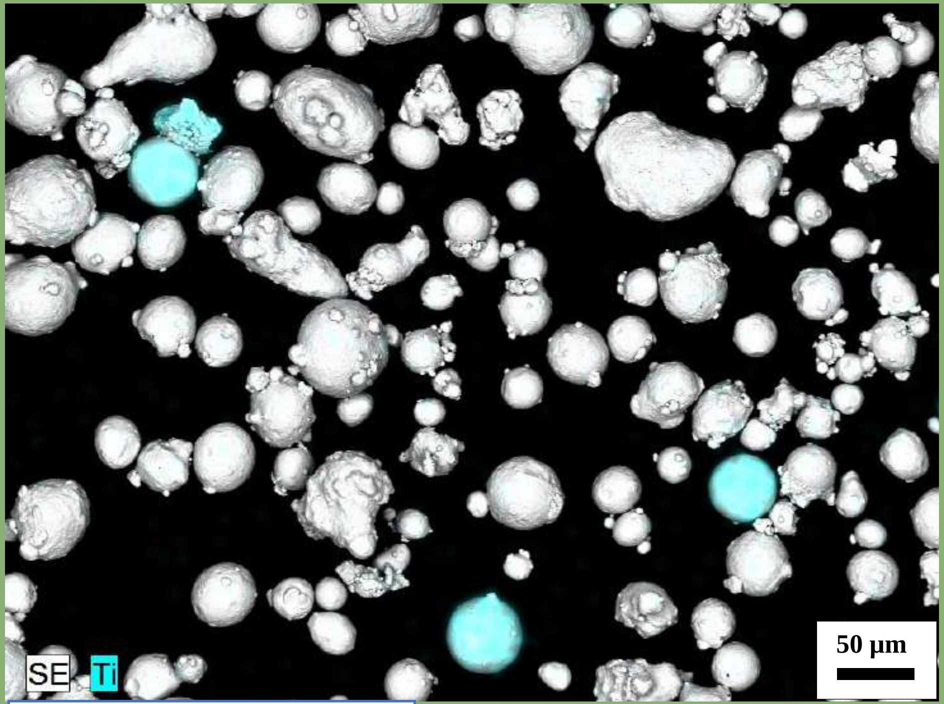
$$\textit{Area Ratio} = \frac{\textit{Area occupied by contaminants}}{\textit{Area occupied by virgin powder particles}}$$

DREAM Developed procedure

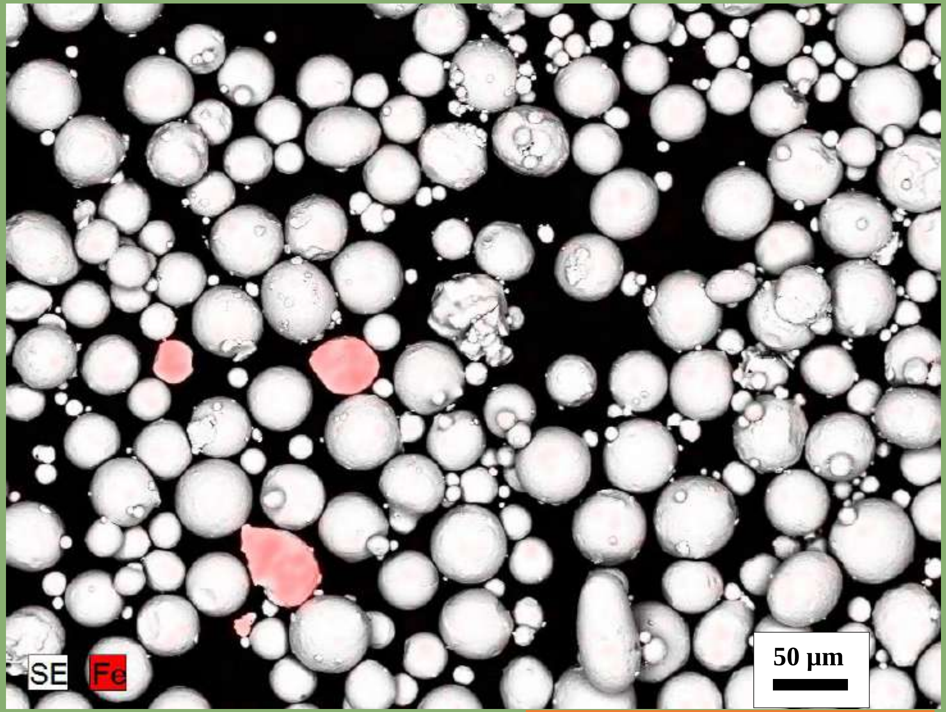
✓ Potential exploitation in Machine Learning environment

➢ CONCISTENCY

1. CALCULATED CONTAMINATION (CC) PROCEDURE



MS1+0.5wt.%Ti64



Ti64+0.5wt.%MS1

Sample	Average CC (10^{-3})
MS1+0.5wt.%Ti64	7 ± 1
Ti64+0.5wt.%MS1	2.7 ± 0.2

➢ CONCISTENCY

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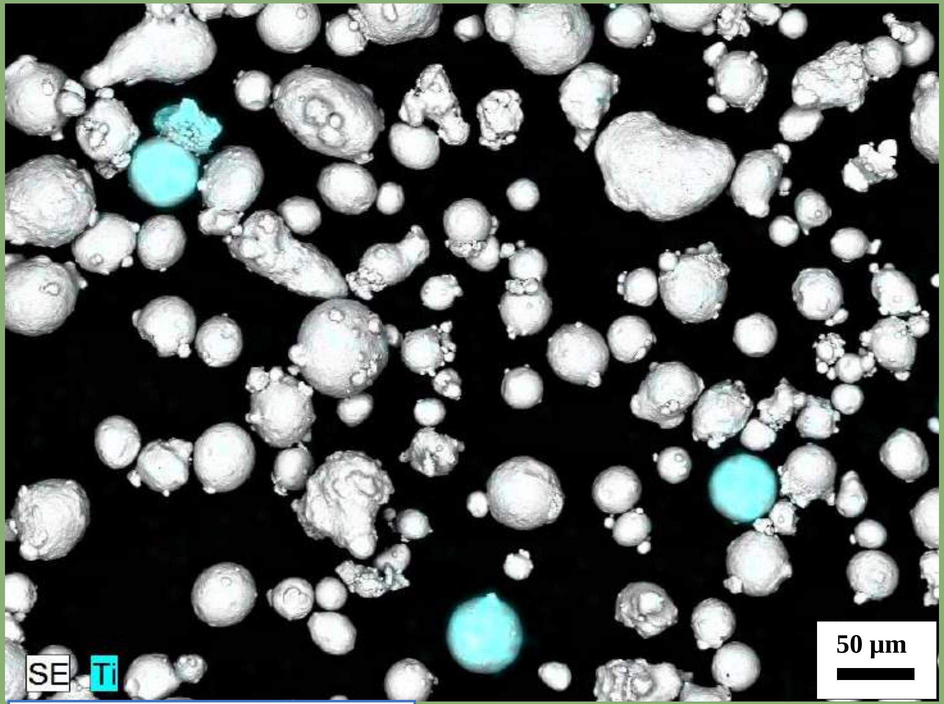

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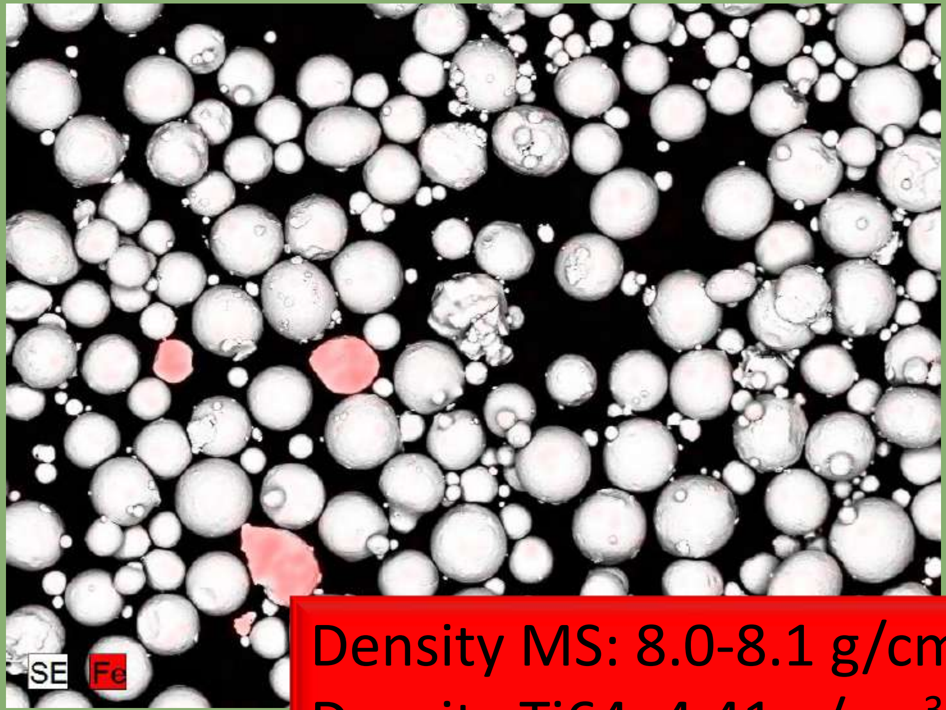


Sample	Average CC (10^{-3})
MS1+0.5wt.%Ti64	7 ± 1
Ti64+0.5wt.%MS1	2.7 ± 0.2

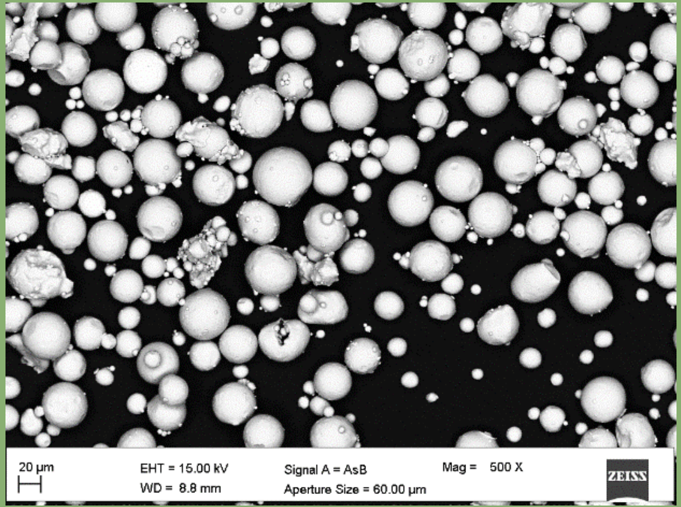
Density MS: 8.0-8.1 g/cm^3
Density Ti64: 4.41 g/cm^3

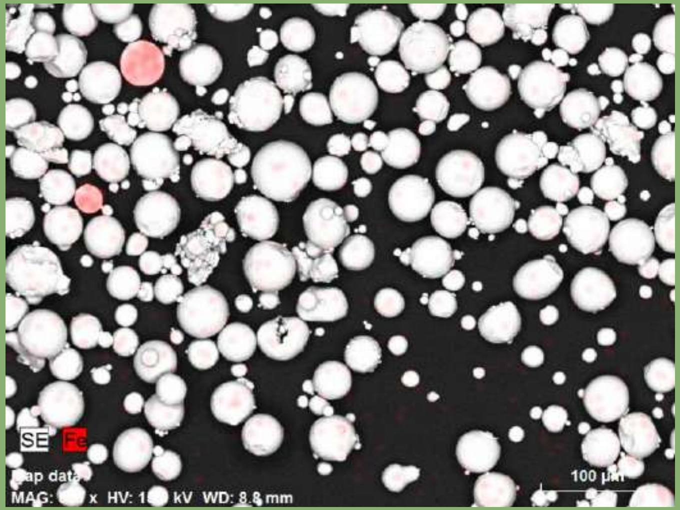
Consistent with density values!

➢ CONCISTENCY

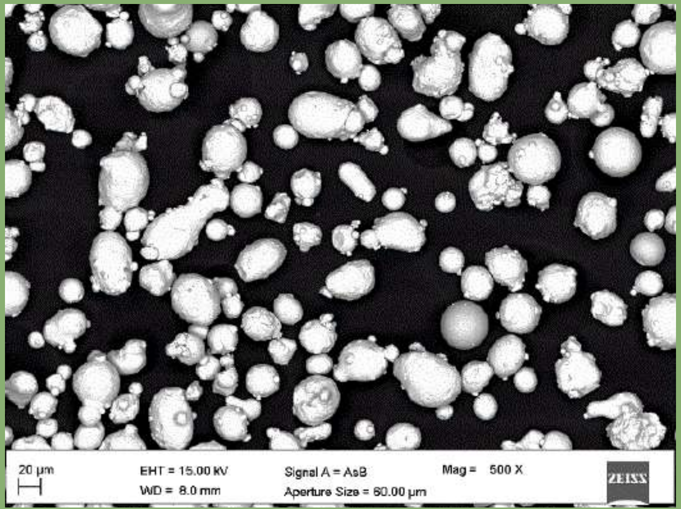
2. IMAGE ANALYSIS PROCEDURE

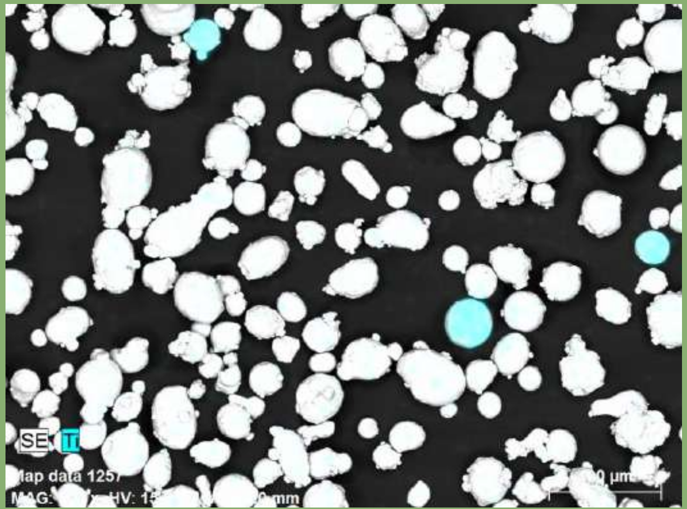
Ti64+0.5MS1





MS1+0.5Ti64





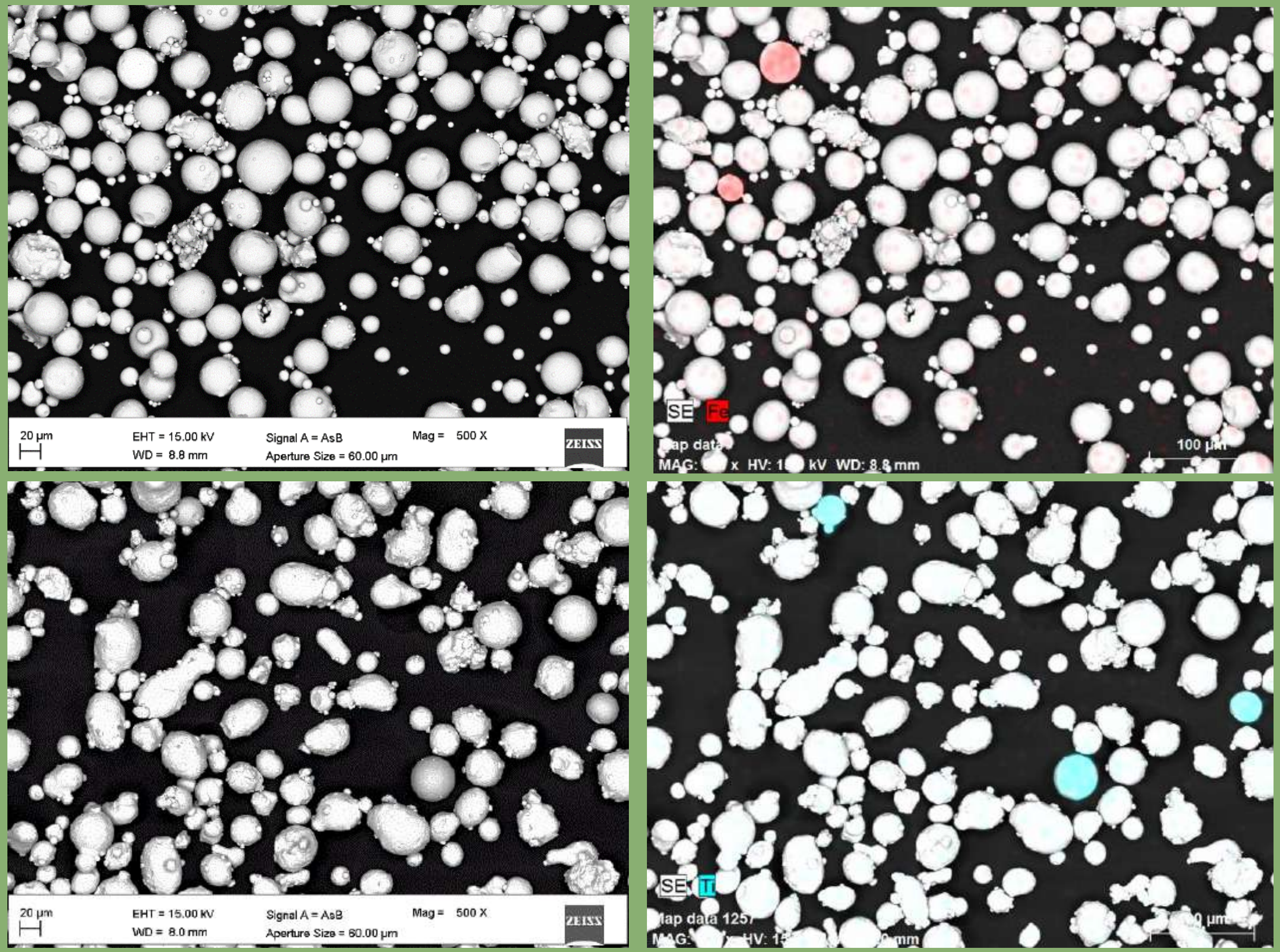
	Area Ratio
MS+0.5Ti64	2.0 ± 0.2
Ti64+0.5MS	1.1 ± 0.4

➢ CONCISTENCY

2. IMAGE ANALYSIS PROCEDURE

Ti64+0.5MS1

MS1+0.5Ti64



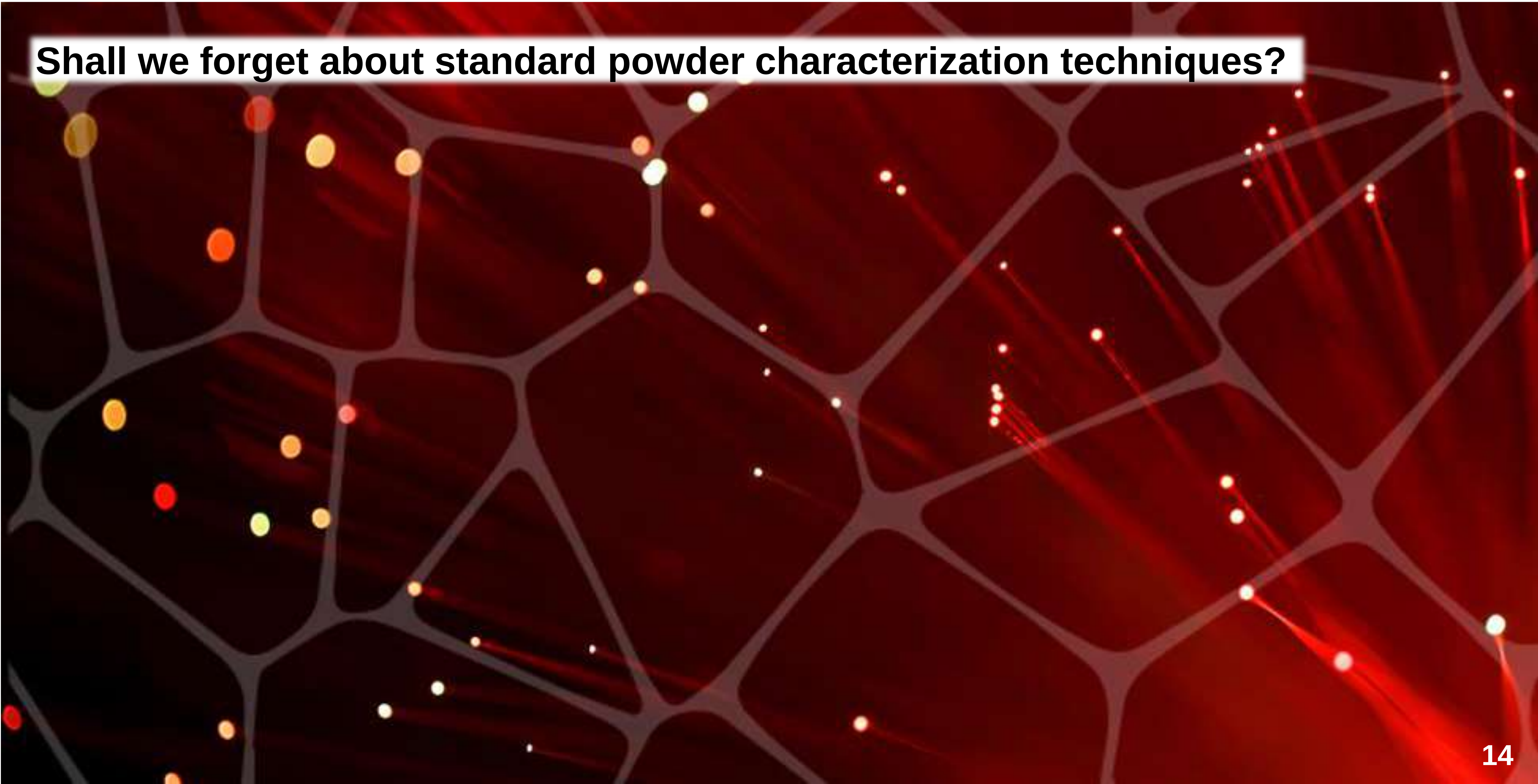
	Area Ratio
MS+0.5Ti64	2.0 ± 0.2
Ti64+0.5MS	1.1 ± 0.4

Density MS: 8.0-8.1 g/cm^3
Density Ti64: 4.41 g/cm^3

Consistent with density values!

Conclusions

PHOTONICS[21]
PHOTONICS PUBLIC PRIVATE PARTNERSHIP
FoF
Factories of the Future
Public Private Partnership
HORIZON2020



Shall we forget about standard powder characterization techniques?

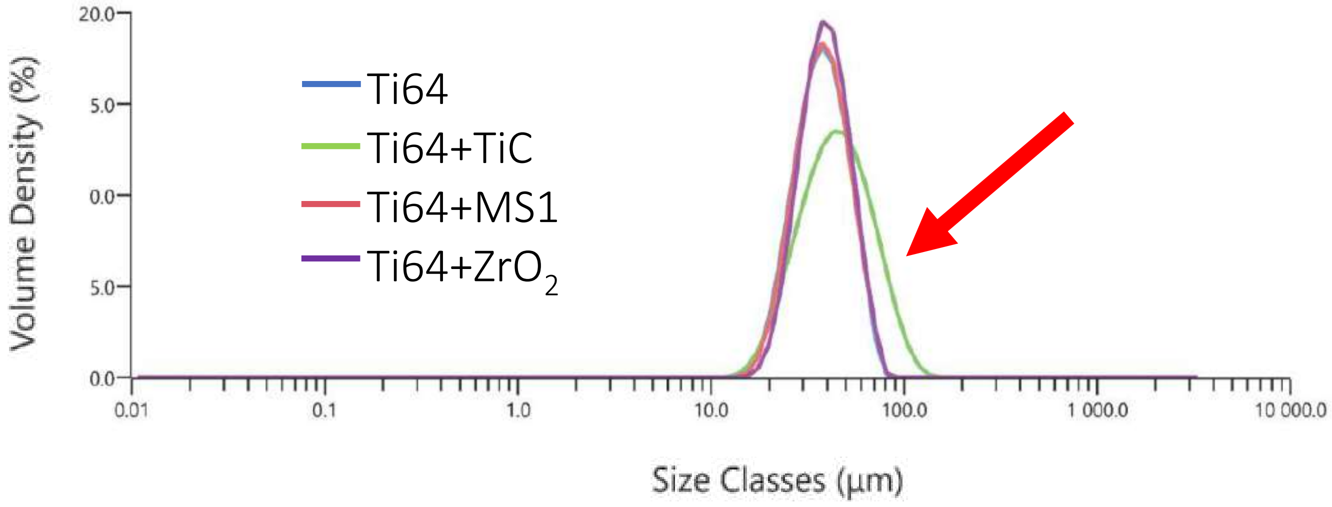
Conclusions

Shall we forget about standard powder characterization techniques? NO!

	Powder	Controlled Contamination	Possible Contamination Source
Ti64	Ti64	~0.2 wt% TiC	Contamination through sieving equipment, tools, gloves or AM machine that are previously used with Ti64.

No relevant results with the developed procedures but…

Size Distribution



Flow Rate

Sample	Carney Funnel (ASTM B964_16) [sec/90g]	Hall Funnel (ASTM B213_13) [sec/50g]
Ti64	11,78(9)	30,7(4)
Ti64+TiC	7,86(7)	27(1)
Ti64+MS1	11,5(2)	30,2(2)
Ti64+ZrO2	12,3(2)	33,7(4)

…PSD and Flow Rate allowed to identify the contamination!

Conclusions

- The combination of different characterization techniques is crucial to successfully detect any kind of cross-contamination in powders for AM, and could be suggested for standardization
- Both developed quantification procedures, calculated contamination (CC) and image analysis, allowed to quantify the cross-contamination by sampling less than 0.1 g on a 100 g batch
- Statistical treatment of the SEM-EDS data showed to be consistent with the physical properties (i.e. density) of the contaminants
- Standard powder characterization techniques showed to be essential to identify the cross-contamination events
- Potential implementation of the image analysis procedure on machine learning systems

Acknowledgements

Thank you!

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 723699.
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