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Development of refractory metal powders for application in additive manufacturing processes

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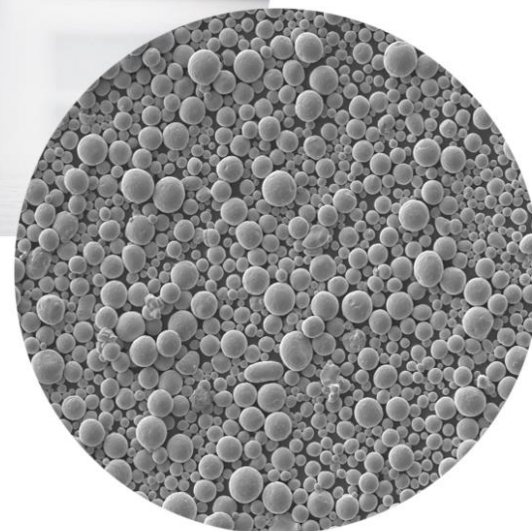
TANIOBIS at a glance

TANIOBIS is a **leading global producer** with over **60 years of experience** in processing of a broad range of **high-quality tantalum and niobium-based materials**

Since July 1st 2018, **TANIOBIS GmbH has been a part of the JX Nippon Mining & Metals Group**



*As of December 31st, 2019



AM

= Additive Manufacturing

- Laser Powder Bed Fusion (L-PBF)
- Electron Beam Fusion (EBM)
- Direct Metal Deposition (DMD)
- Laser Cladding (LC)

trinsic[®]

= intrinsic

- Intrinsic material properties
- Pre-alloyed refractory alloy powders
- Customized solutions
- Excellent processability

AMtrinsic® spherical powders

Historical

- 2015 Nb, Ta general
 - 2016 Ti/Nb biomedical
 - 2017 Ti/Nb/Ta biomedical improved
 - 2019 C-103 HT
 - 2020 FS-85 HT improved
 - 2020 RM HEAs academic
 - 2021 Metal/Ceramic Composites
-
- Since 2017 various customer-specific alloys (compositions shall not be disclosed)

Elements in AMtrinsic alloys (MP)

Refractory

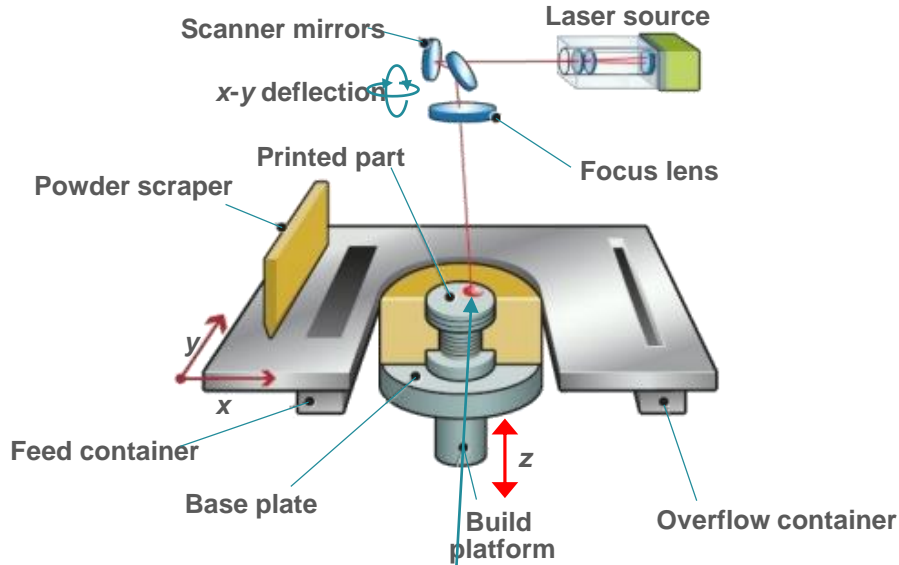
3 IIIB 3B	4 IVB 4B	5 VB 5B	6 VIB 6B
21 Sc 1545 °C	22 Ti 1668 °C	23 V 1910 °C	24 Cr 1907 °C
	40 Zr 1855 °C	41 Nb 2477 °C	42 Mo 2623 °C
	72 Hf 2227 °C	73 Ta 3020 °C	74 W 3422 °C

Main group

13 IIIA 3A	14 IVA 4A
5 B 2076 °C	6 C 3550 °C
13 Al 660 °C	14 Si 1410 °C

Printing AMtrinsic® spherical powders

Laser powder bed fusion - principles

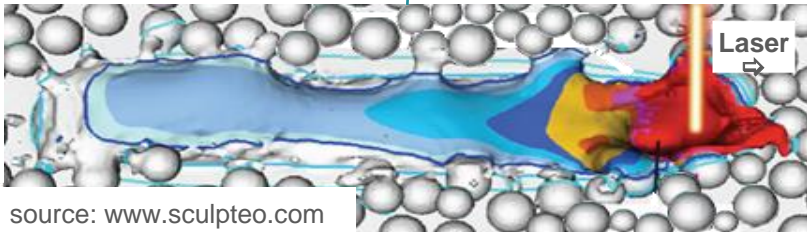


Principle: layer-wise formation of bulk parts

- substrate plate is coated with powder
- consolidation of selected areas by laser melting
- Lowering build platform
- removal of loose powder
- finishing treatments

Requirements

- pre-alloyed powder (no mixtures)
- narrow PSD with $d_{90} < 60 \mu\text{m}$
- excellent powder flow
→ spherical particles



source: www.sculpteo.com



TANiOBIS
inspiring metal evolution

AMtrinsic® spherical powder production

Challenges

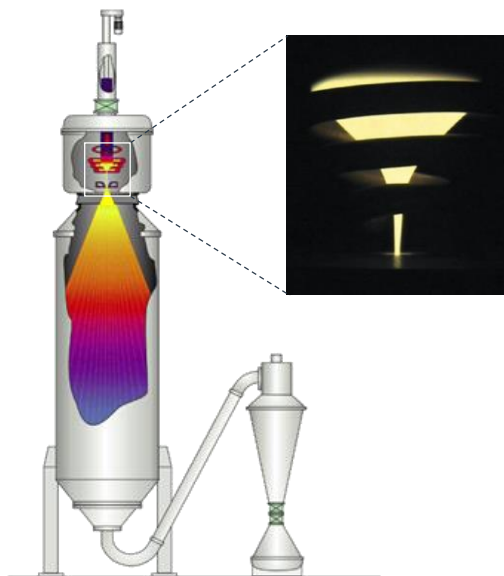
- Atomization using conventional crucible-based processes almost impossible
 - lack of crucible materials for UH temperature
 - contamination of target material with crucible material
 - oxidation
- Atomization using wire limited to very simple and ductile systems
- Spheroidization requires pre-alloyed powders

Solution

- Electrode induction-melting gas atomization (EIGA)
 - contact-free inductive melting of pre-alloyed or PM rods
 - in-situ alloying of PM rods during inductive melting
 - almost no limitation in material composition: from elemental to multinary (e.g. HEA)
 - adjustable particle size distribution
 - dense, spherical particles with good processability

Electrode Induction-melting Gas Atomization

Production setup

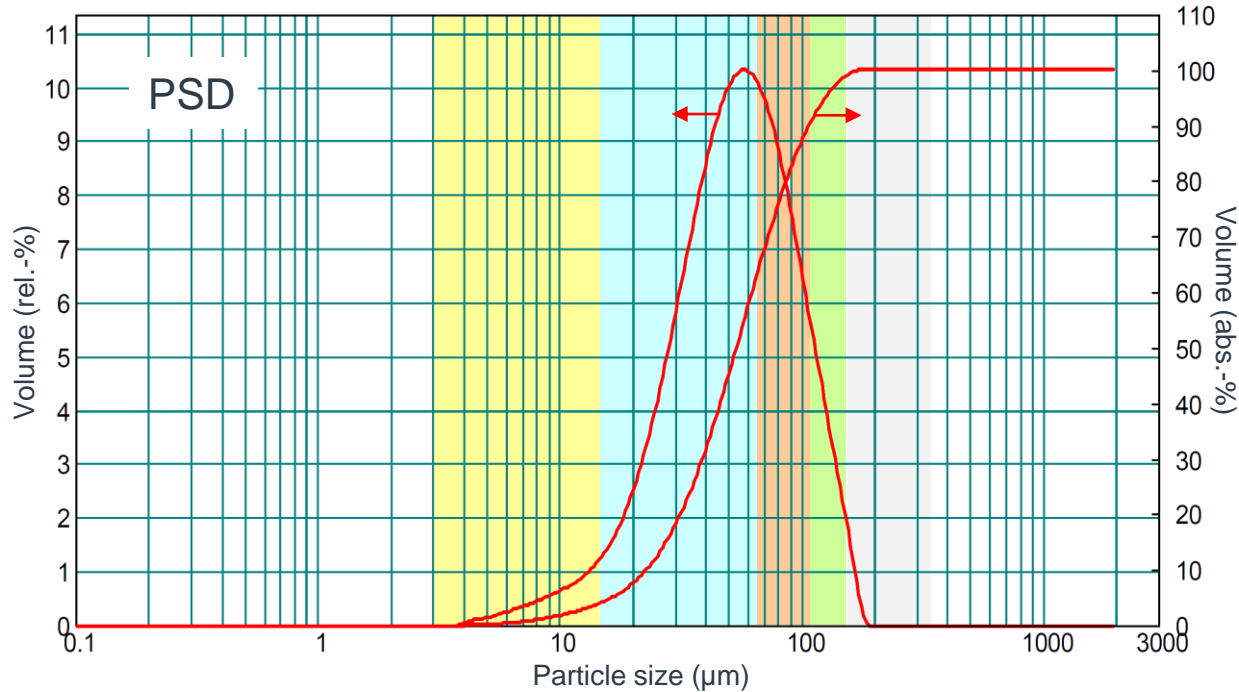


Source: ALD



AMtrinsic® spherical powders

Post-atomization processing: classification required

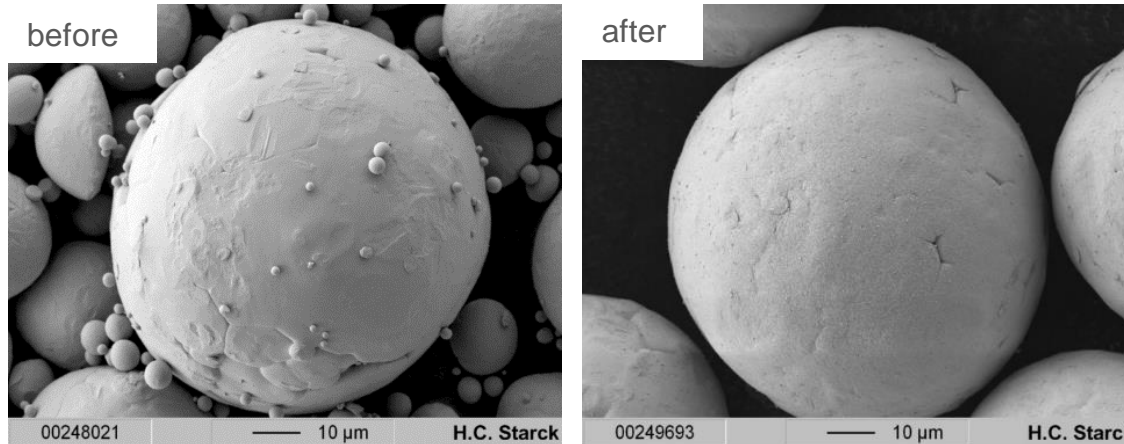


< 15 μm :	dispose
15 – 63 μm :	L-PBF
63 – 105 μm :	SEBM
105 – 150 μm :	LC, LMD
> 150 μm :	dispose

AMtrinsic® spherical powders

Post-atomization processing: de-oxidation by metal vapor reduction

- Example: Nb de-oxidation by Mg vapor



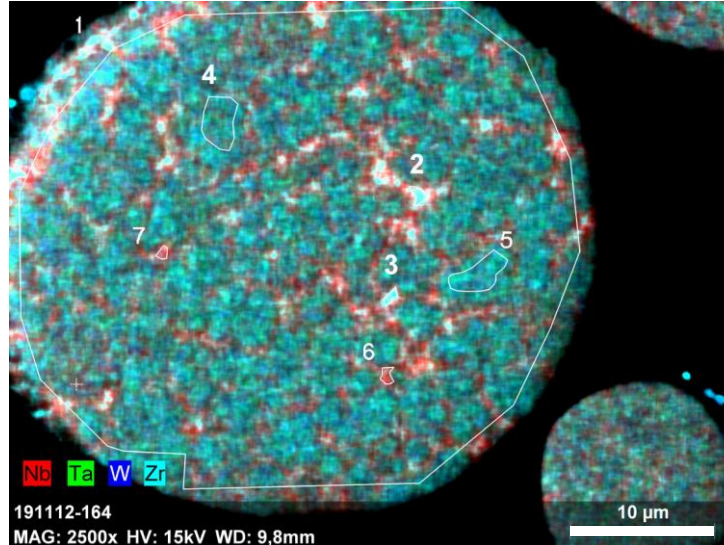
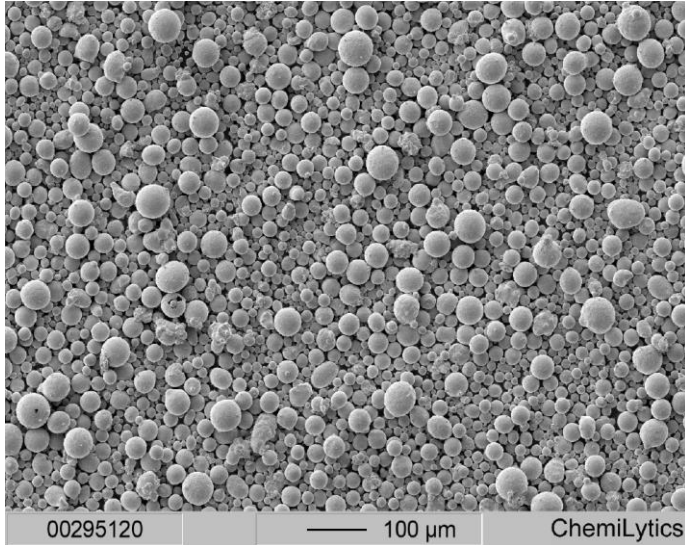
Fraction (µm)	O as obtained (ppm)	O after Deox (ppm)
105-150	1512	171
63-105	1716	268
<63	2038	247

- Extremely low O content in AMtrinsic® Nb → unique capabilities @TANIOBIS
- Removal of fine fraction after leaching
- No satellites, no agglomeration → very good flow
- Deoxidation of Ti-containing alloys under investigation

AMtrinsic® spherical powders

FS-85 powder morphology: alloys for HT application (Nb-28Ta-10W-1Zr)

22 Ti 1900 °C	23 V 1900 °C	24 Cr 1907 °C
40 Zr 1850 °C	41 Nb 2477 °C	42 Mo 2623 °C
72 Hf 2397 °C	73 Ta 3000 °C	74 W 3422 °C



wt-%	1	5	6
Nb	63,3	54,6	66,5
Ta	26,1	31,3	22,6
W	9,2	13,7	9,3
Zr	1,3	0,4	1,6

↑ average ↑ inter-dendrite

↓ dendrite

SEM: spherical, no satellites or agglomerates

EDX: dendrite-type, though less pronounced.

- Ta/W-enriched dendrite vs. Nb/Zr-enriched inter-dendrite phase

Printing AMtrinsic® spherical powders: Niobium

Parts for superconductor application

22 Ti 1063 °C	23 V 1910 °C	24 Cr 1907 °C
40 Zr 1855 °C	41 Nb 2477 °C	42 Mo 2623 °C
72 Hf 2205 °C	73 Ta 3022 °C	74 W 3422 °C



printed @CERN

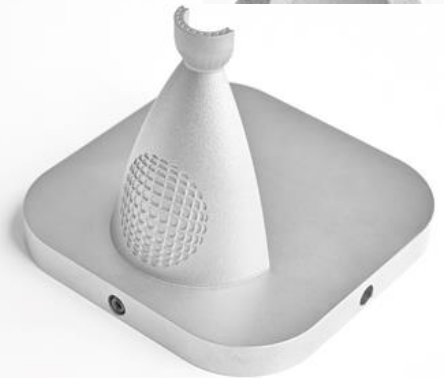
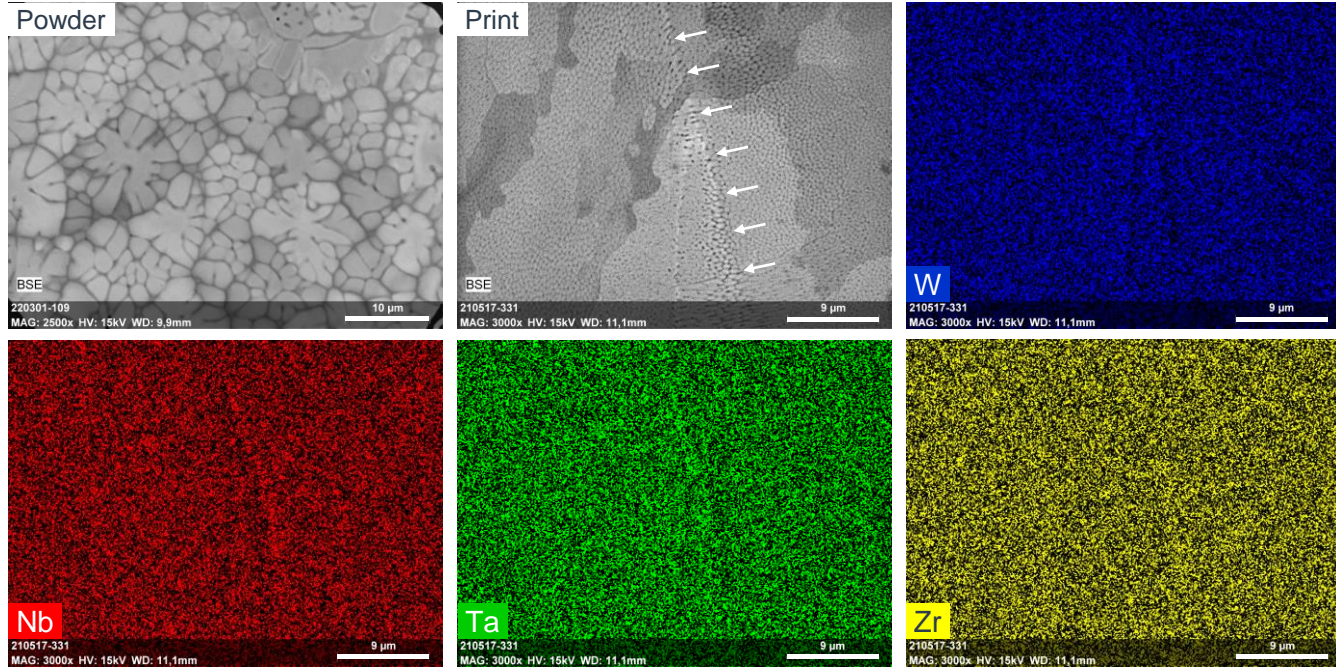


HOM coupler Crab
cavity DQW
printed @CERN

Printing AMtrinsic® spherical powders: FS-85

Parts for high-temperature application (Nb-28Ta-10W-1Zr)

22 Ti 1922 °C	23 V 1910 °C	24 Cr 1907 °C
40 Zr 1855 °C	41 Nb 2477 °C	42 Mo 2623 °C
72 Hf 2205 °C	73 Ta 3009 °C	74 W 3422 °C



Satellite thruster ©Alloyed

- Crack- and pore-free, homogeneous element distribution without dendrites
- Melt tracks and grain boundaries visible
- Mechanically fully investigated, superior to C-103

Acknowledgements

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