Development of new materials for the SLM process

a project by:

speaker: Eng. Mirko Sinico (sinico@pd.infn.it)

Partners

- **Coaching** regarding SLM process and AM software
- **Access** to SISMA's AM Lab and SISMA's facilities
- **Training on the job** at SISMA SpA for >50 days
- **Support** for metallographic examination and inspection

- **Action Research** on GD&T (Geometric Dimensioning and Tolerancing) and general engineering tolerances

sisma

OFFICINA DEI MATERIALI

ENGIN SOFT

LPW

- **Industrial** supervisor
- **Definition** of the industrial case study
- **Monitoring** of planned activity
- **Advisor** on research to business outcomes

- **Support** on SLM powder supply

- **Research** supervisor
- **Coaching** regarding CAD software and ANSYS CFX simulation
- **Support** for not-AM mechanical production and finish
- **Monitoring** of research activity
- **Advisor** on research steps

INFN Istituto Nazionale di Fisica Nucleare Sezione di Padova

Università degli Studi di Padova

Dipartimento di Ingegneria Industriale

- **Access** to the Metallurgy Group laboratories
- **Support** for SEM characterization of samples
- **Mechanical** testing of final ASTM specimens

Prof. Irene Calliari' & Prof. Giovanni Meneghetti' Groups

SLM materials

Example of SLM material developed by a specific company

BRONZE 90Cu-10Sn (sisma)

- Jewellery and fashion application
- High density and surface quality, with ease supports removal

In Use		
HastelloyX	In718	In625
Ti64	TiCP	CoCr
PH1 (15-5)	316L	
Maraging Steel (MS1)		
AlSi10Mg		

Under development	
In713	17-4
Al 7050	Copper
Niobium	Tungsten
CM247LC	
High Temp Copper Alloys	

Example of SLM material developed by a specific company

STAINLESSSTEEL CX (eos)

- Corrosion Resistant Stainless Steel for Tooling
- Can be easily machined, shot-peened and polished in as-built or heat treated status

Many other materials under development

- 2011 research, Fraunhofer ILT: Airbus Scalmalloy (**AlMgScZr**)
- 2013 research paper, L. Thijs: Pure **Tantalum** (Ta)
- 2015 research paper, S. L. Sing: **TiTa alloy** for biomedical app.
- 2017 research, A. Ivekovic: **Tungsten Heavy Alloy** ($W_5Ta_7Ni_3Fe$)
- 2017 research paper, J. Jaćimović: **NdFeB** Permanent Magnets

etc...

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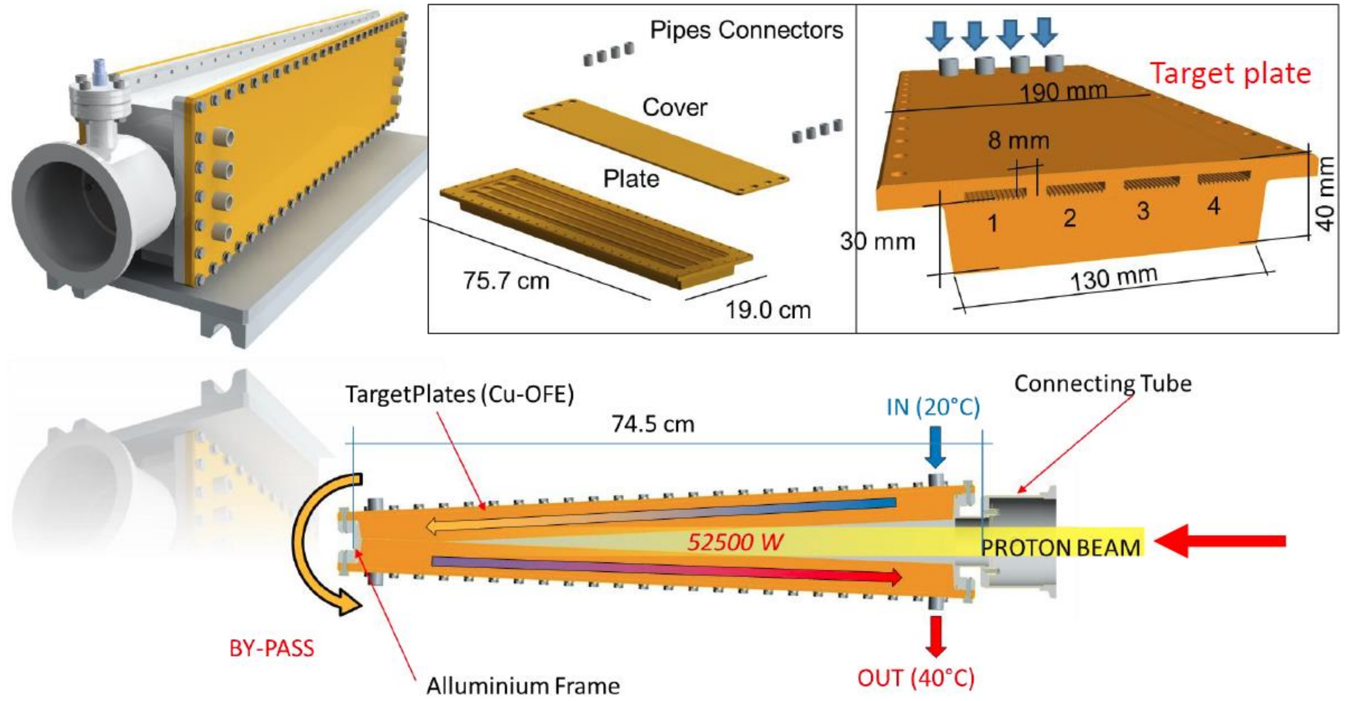
Laboratori Nazionali di Legnaro

INDUSTRIAL CASE STUDY

OLD CU BEAM DUMP



- Beam Dump **for proton H beam** (INFN SPES project)
- Pure **Cu** OFE **for heat dissipation** (max 52.5 kW !)
- **Water cooled** with 2400 l/h at 1.25 m/s

DISADVANTAGES

- **Many manufacturing steps**: production of single components, assembly by connectors, brazing…
- **Relatively simple cooling design**
- **Heavy and bulky:** 3360 kg with the Pb radiation shield

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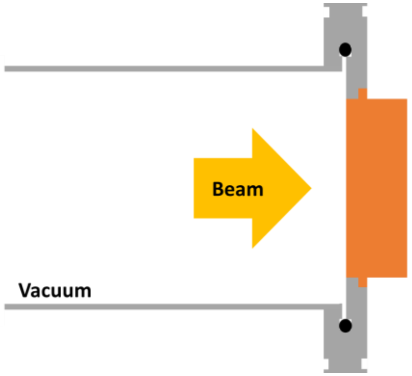

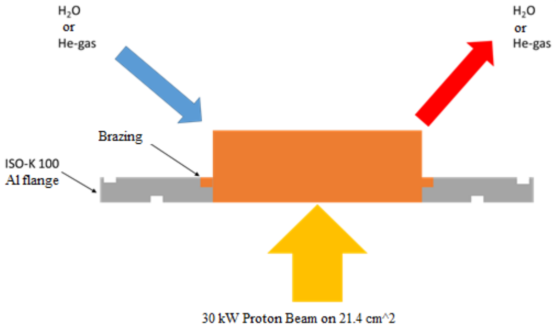

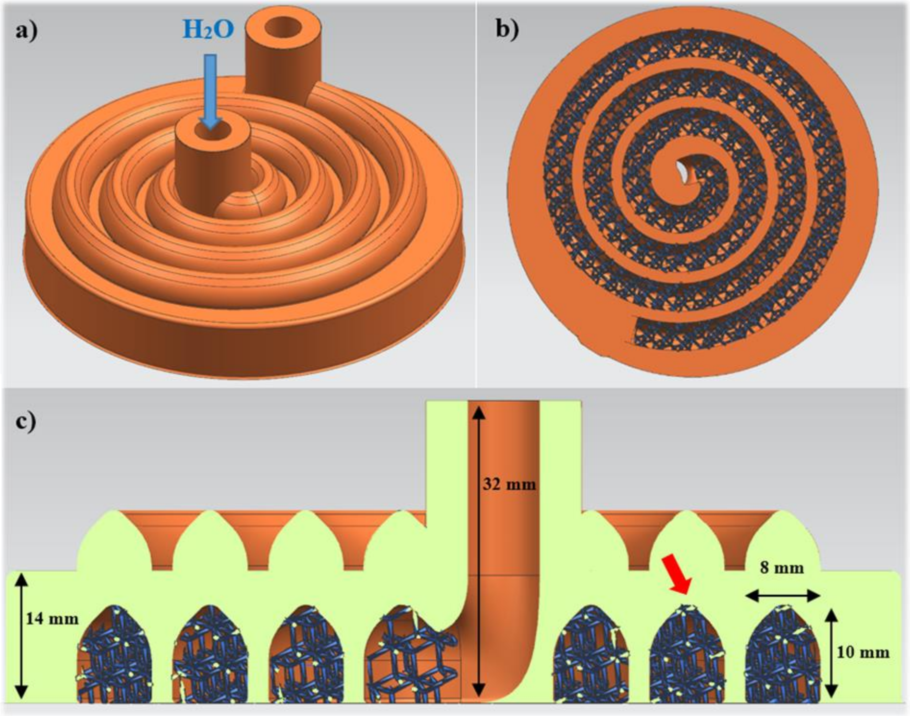

- Beam Dump **for proton H beam** (INFN SPES project)
- Pure **Cu for heat dissipation** (max <u>30 kW on 21.4 cm^2</u> !)
- **Water cooled** or He cooled

ADVANTAGES

- **Two manufacturing steps**: SLM production and brazing with the ISO K-100 flange (↓ time to product)
- **Conformal cooling design** with potential filling metallic foam
- **Lighter and smaller**

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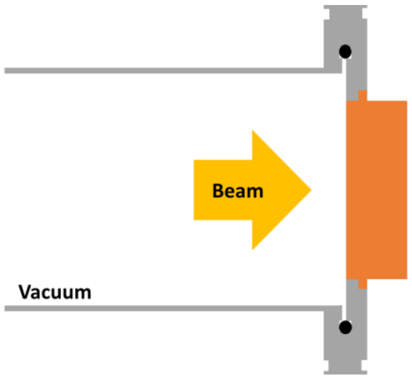
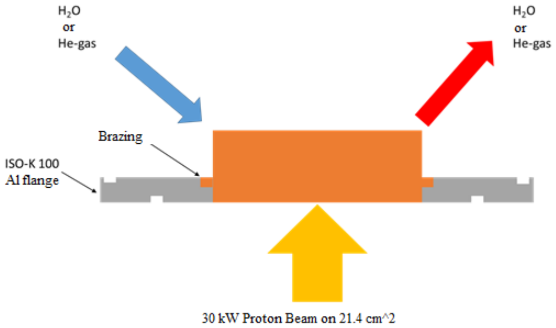
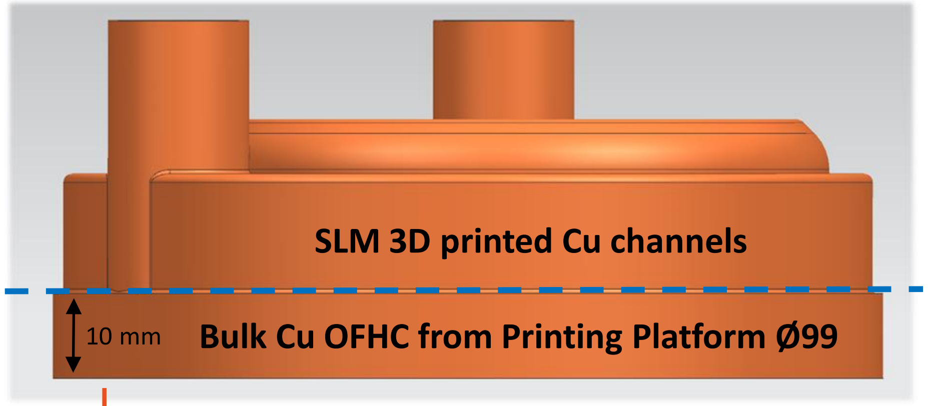
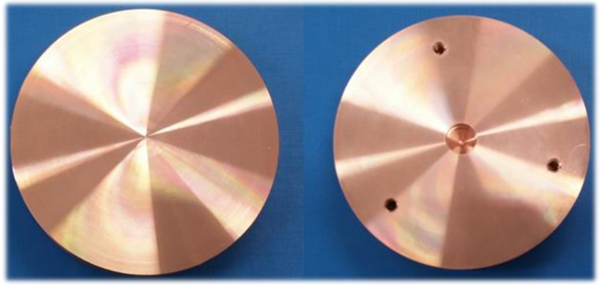
INDUSTRIAL CASE STUDY

NEW SLM CU BEAM DUMP







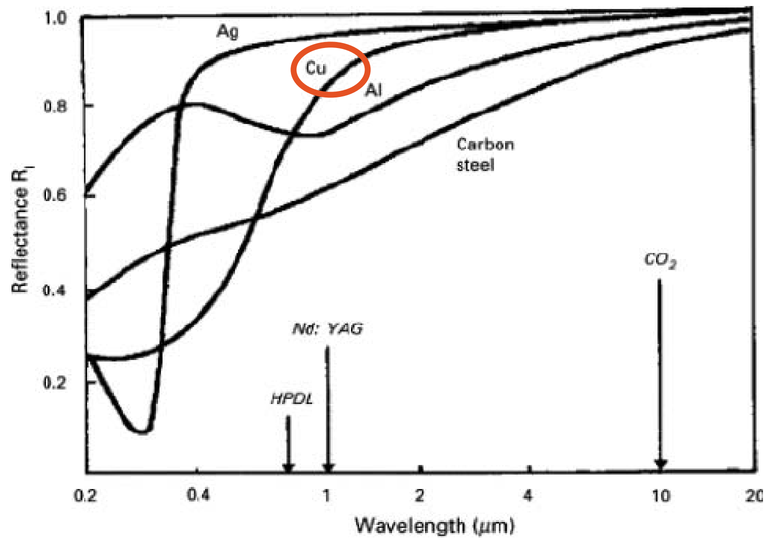


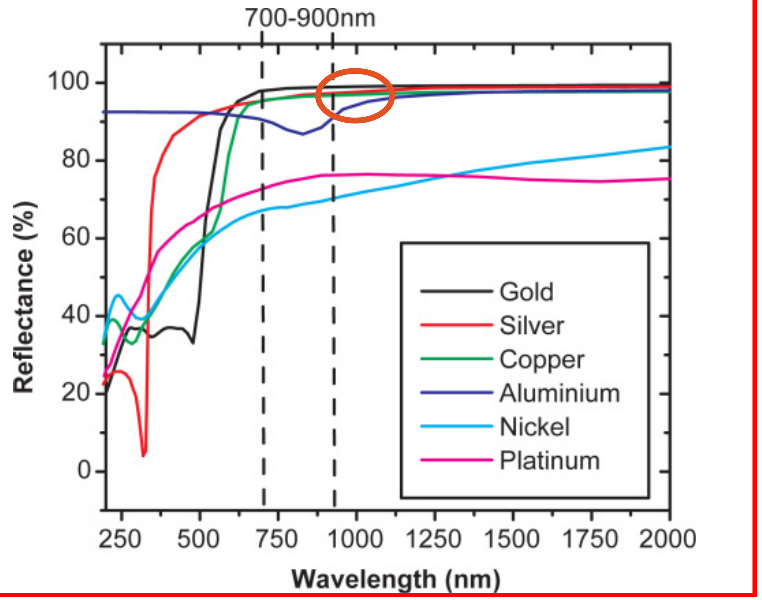
- Beam Dump **for proton H beam** (INFN SPES project)
- Pure **Cu for heat dissipation** (max 30 kW on 21.4 cm^2 !)
- **Water cooled** or He cooled

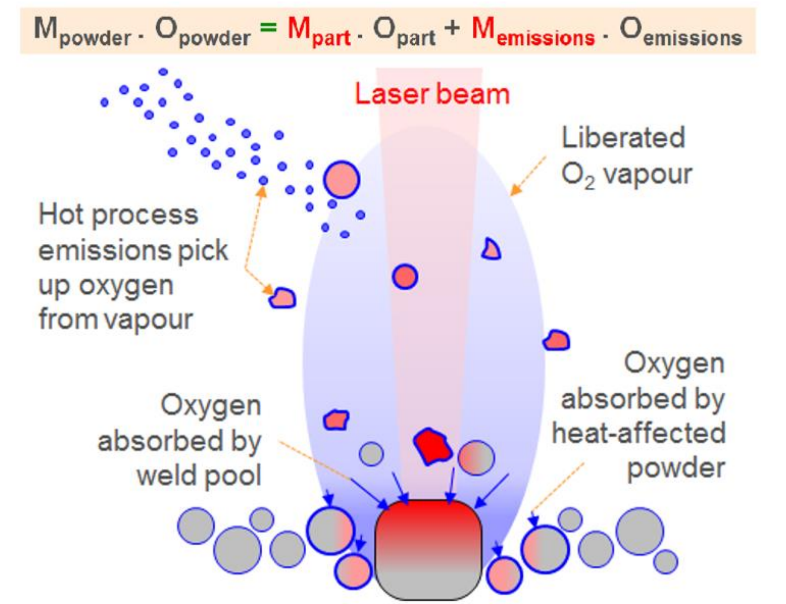
ADVANTAGES

- Eventually the SLM printing **platform** can be **part of the final object design** (↓ printing time and cost)

Design not final and introduced as an example, this project is focused on the production process





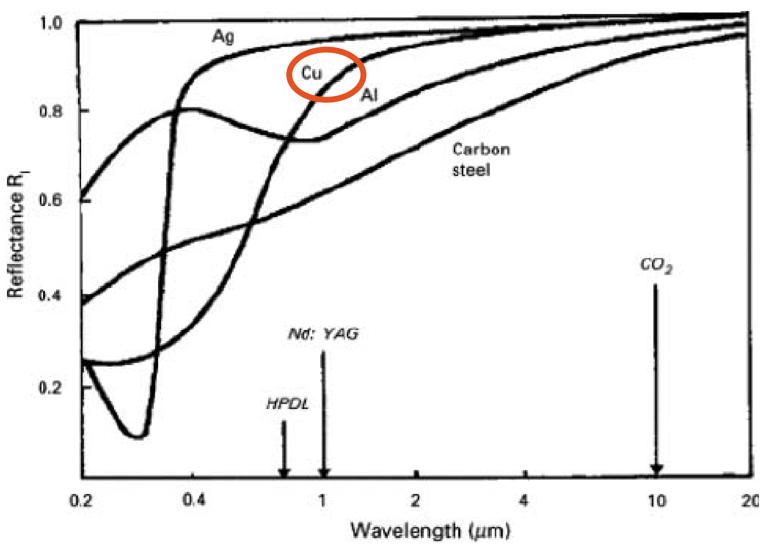


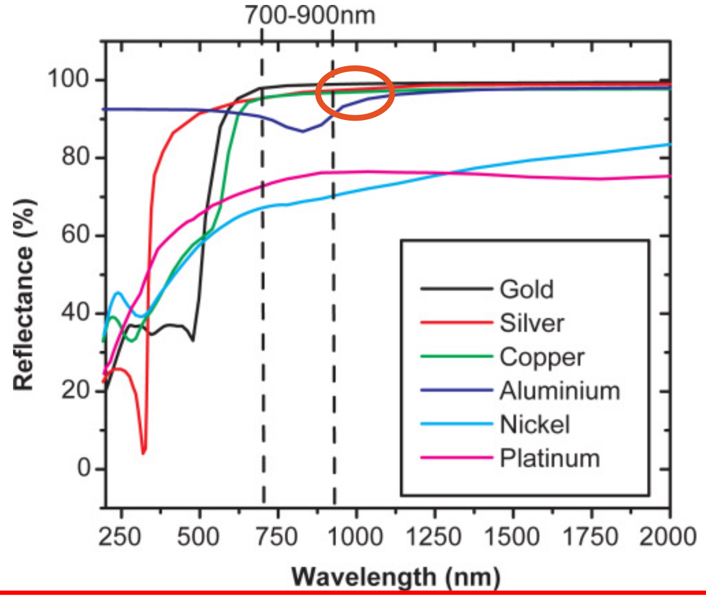
MAIN PROBLEMS

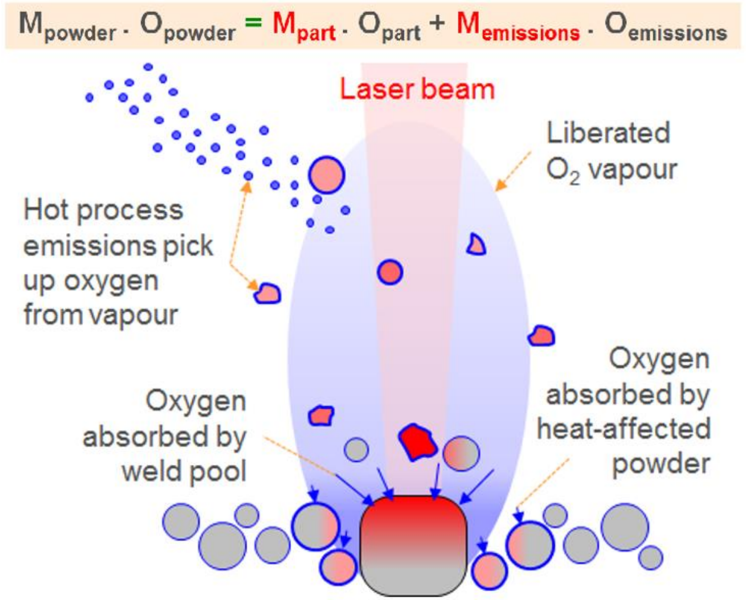
- **High reflectance** may limits power that can be put into powder to melt it
  $\rightarrow$ > 60 % reflectance on pure Cu powder, > 90 % reflectance on bulk (1070 nm, Nd:YAG)
- **High Cu thermal conductivity** (390-400 W/mK) may limits maximum local temperature that can be attained and limits successful bonding between weld lines
- **Shrinkage porosity** contributes to total porosity (CTE = ~17 10^{-6} K^{-1})
- **Oxygen** between powder particles can be adsorbed by the melt pool: CuO_x oxides will stabilise the surfaces, act as physical barriers to wetting and reduce the flowability

Cu T melting:	1083 °C
CuO_2 T melting:	1230 °C
CuO T melting:	1336 °C

SLM of Copper







SOLUTIONS

- **Higher Laser Power,** or lower laser wavelength (ex: green 515 nm)
- **Smaller Laser Spot** (~30 μm)
- **Powder mixtures** to bind together Cu particles (ex: Cu-Ni, Cu-SCuP)
- **Divert to Cu alloys:** ↓ reflectance, ↓ thermal conductivity
- **Carefully control oxygen** in the SLM machine chamber and in powder manipulation

Cu T melting:	1083 °C
CuO_2 T melting:	1230 °C
CuO T melting:	1336 °C

SLM materials

- 2014, by UK service
- Advanced Heat Exchanger, lone example of industrial pure copper SLM

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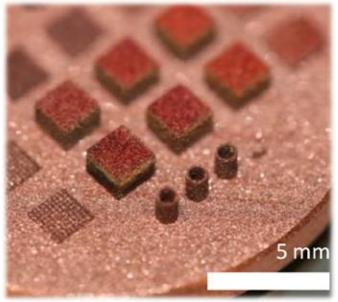
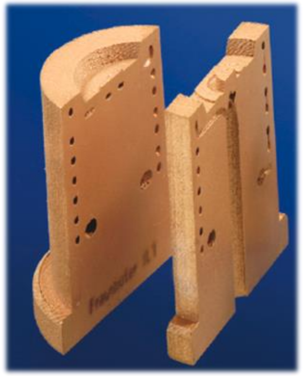
- 2016 research paper, P. A. Lykov
- 200 W CO_2 Pulsed Laser, 35 μm spot
- → 88.1 % density and 149 MPa UTS



- 2017 research paper, L. Kaden
- Ultrashort laser pulses at MHz repetition rate, 1030 nm, 35 μm spot
- → Low density and melt beads structure



- 2011 research, Fraunhofer ILT
- Copper Alloy: Hovadur K220, $CuNi_2SiCr$
- 1000 W Laser, modified SLM machine
- → 99.9 % density

A lot more research on SLM of Copper Alloys

Material	Highest relative density, %
Cu + Cu10Sn + Cu8.4P powder	84.00
Cu10Sn + Cu8.4P + Ni	95.20
CuNi15	92.00
C18400	96.74

- > 20 published research papers
- Mixtures and special Cu Alloys being developed

SLM materials

- 2014, by UK service
- Advanced Heat Exchanger, lone example of <u>industrial</u> pure copper SLM

<table>
<tr><th colspan="4">In Use</th></tr>
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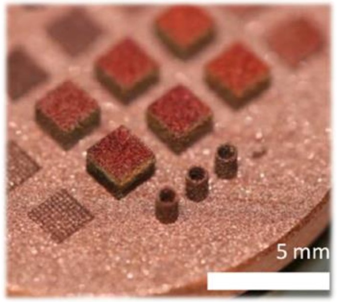

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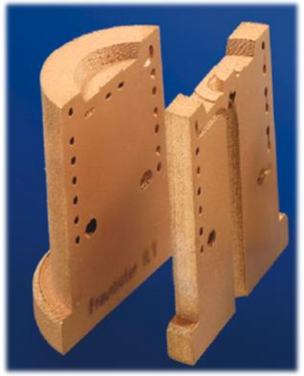

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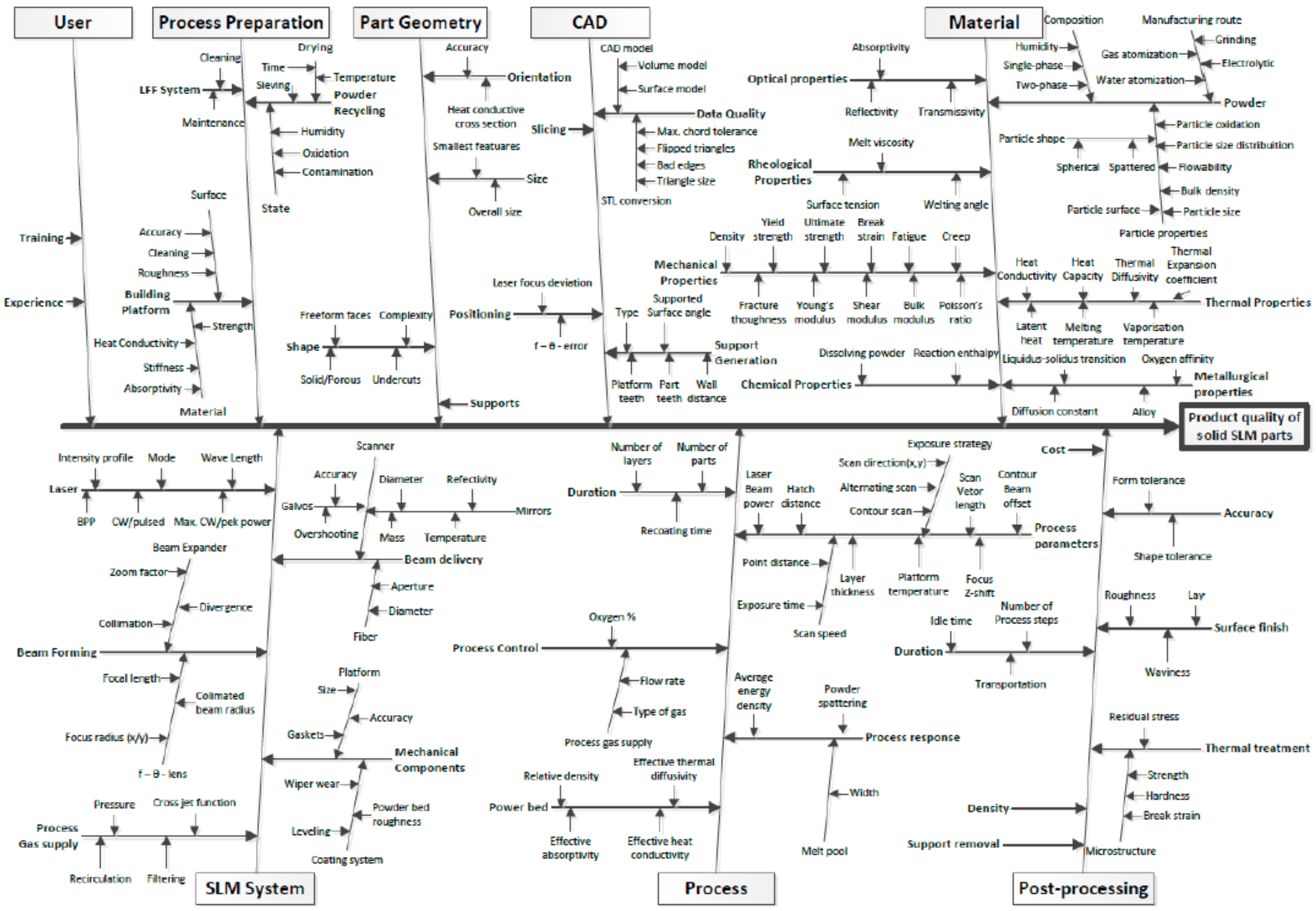
- Copper Alloy: Hovadur K220, $CuNi_2SiCr$
- 1000 W Laser, modified SLM machine
- → 99.9 % density

- 2015 <u>research</u>, NASA
- Copper Alloy: GRCop-84, Cu-Cr-Nb
- 400 W Fiber Laser, ~50-100 μm spot
- → >99.5 % density after HIP post-processing

ISHKAWA DIAGRAM FOR SLM PROCESS



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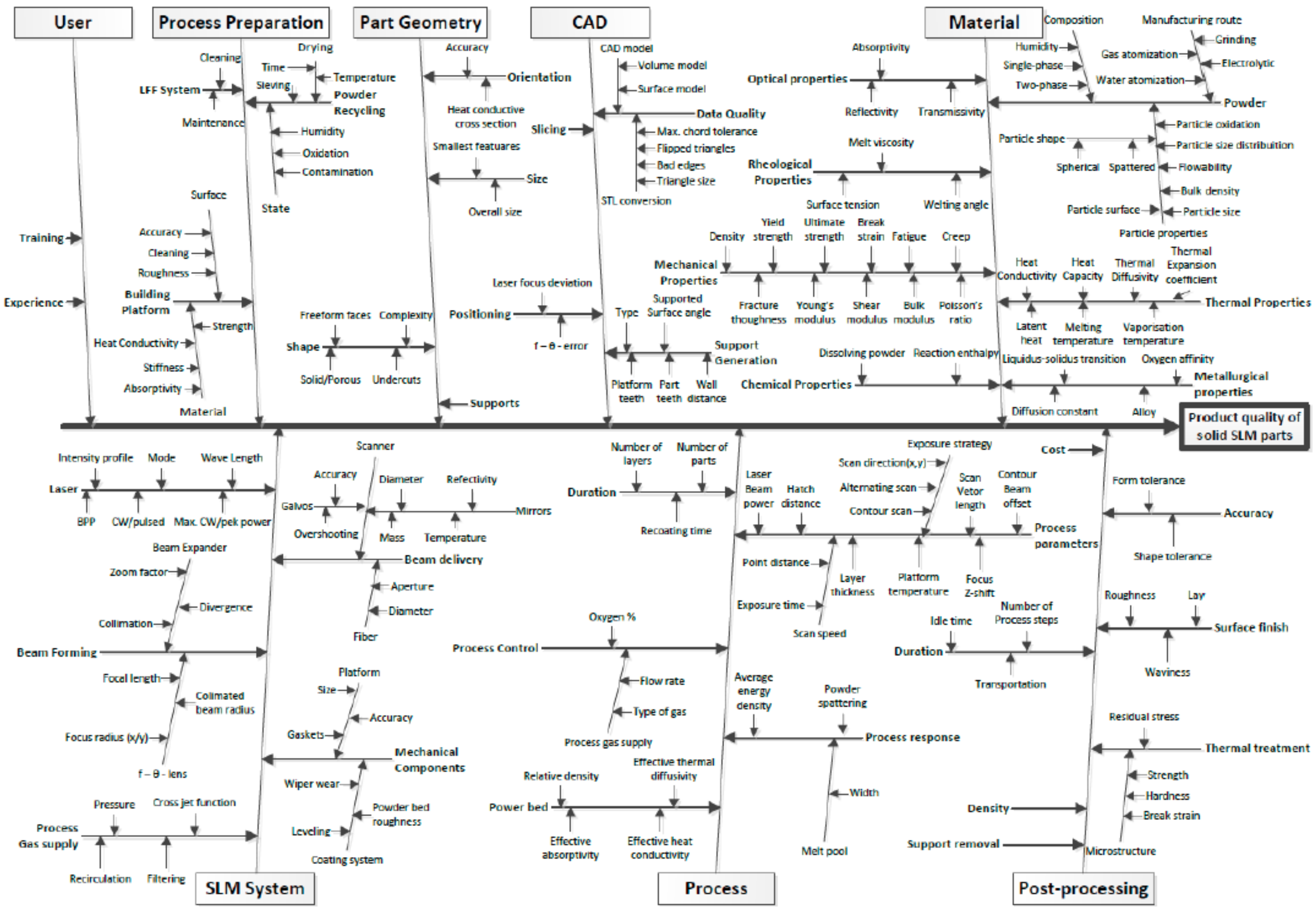
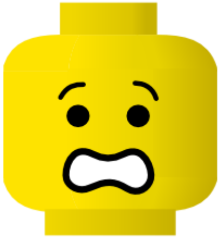


MAIN USE OF THE DIAGRAM

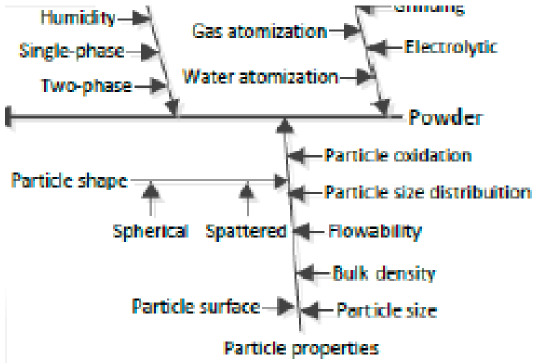


Scare People

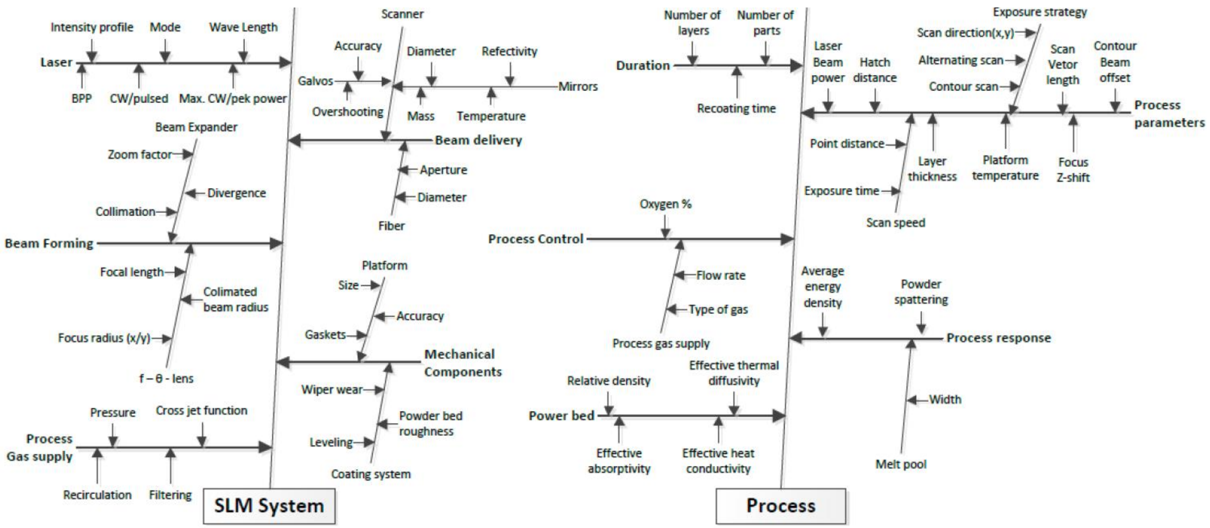
ISHKAWA DIAGRAM FOR SLM PROCESS

TWO SELECTED PURE CU POWDERS

LPW support



- “Standard” **15-45 μm distribution,** gas atomized 99.9 pure Cu powder
- Fine **selected distribution** under NDA, gas atomized 99.9 pure Cu powder
  $\rightarrow$ We call it “**Cu Fine**”



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SLM machine

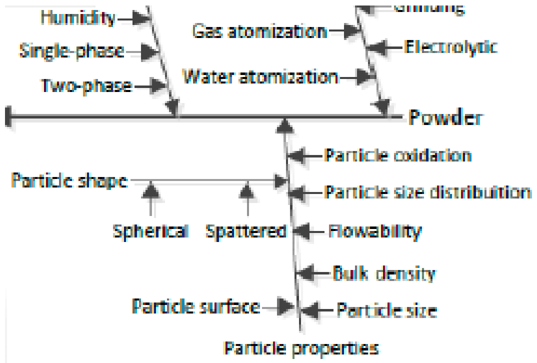
SISMA mysint100PM

ISHKAWA DIAGRAM FOR SLM PROCESS

Our SLM Cu Recipe

TWO SELECTED PURE CU POWDERS

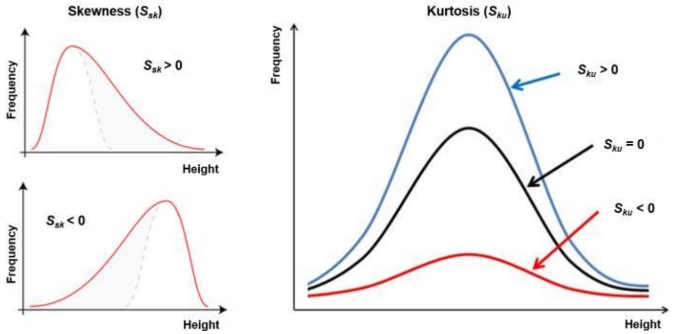
LPW support



- "Standard" **15-45 μm distribution** gas atomized 99.9 pure Cu powder
- Fine **selected distribution** under NDA

➢ **Improve flow** using high spherical and non-cohesive particles

➢ **Improve layer density** by adapting particle size distribution
→ Select proper PSD (skewness, kurtosis, size, amount of fine particles) specific for our layer thickness, laser spot, deposition system



Technical Data - Dati Tecnici	mysint100PM
Building volume - Volume di lavoro	ø 100x80 63,5x80 34,5x80 mm (interchangeable - intercambiabili)
Laser Source - Sorgente laser	Fiber Laser 200 W
Precision Optics - Ottiche di precisione	Quartz F-Theta Lens
Laser spot diameter - Diametro spot laser	30 μm
Typical layer thickness (adjustable) - Spessore tipico layer (regolabile)	20-40 μm
Inert gas - Gas inerti	Nitrogen, Argon - Azoto, Argon
Inert gas supply - Rifornimento gas inerti	ø 6 mm / 2.5 ÷ 5 bar @ 35 L/min
Inert gas consumption - Consumo gas inerti	<0,3 L/min @ 0,5% O_2
O_2 concentration - Concentrazione O_2	0,3%
Door - Porta	Standard
Filter unit - Unità filtro	Removable - Rimovibile
Power supply - Alimentazione elettrica	220-240 V 1ph - 50/60 Hz
Max power absorbed - Potenza massima assorbita	1,53 kW
Machine dimensions - Dimensioni macchina	1390x777x1600 mm (LxWxH)
Net weight - Peso netto	650 kg

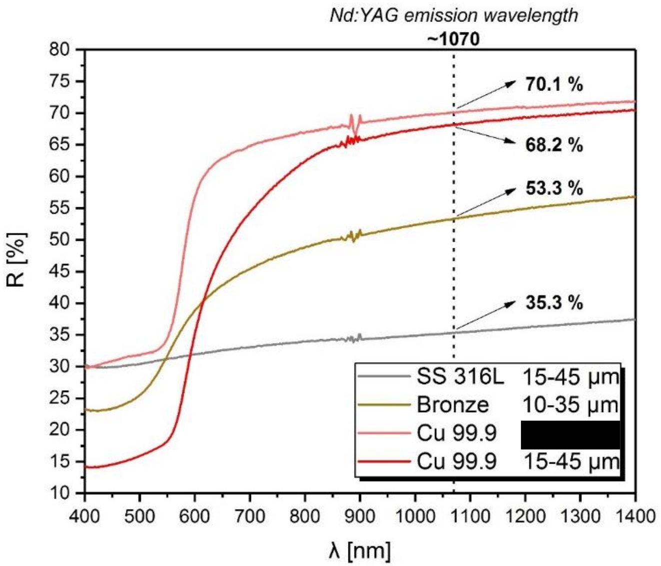
sisma **mysint100PM**

- **200 W maximum Laser Power** (1070 nm)
- **Small Laser Spot** (d = 30 μm)
- **Small Layer Thickness** (t = 20 μm)

→ Deep **research on processing parameters**

Cu powder quality

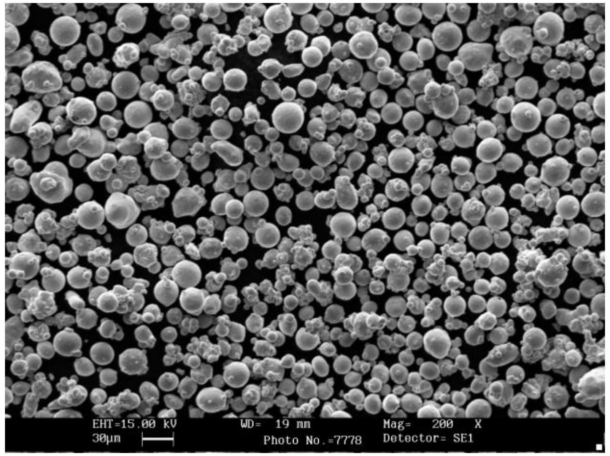
- ~70 % reflectance for both powder distributions !
- Powder packing density and distribution slightly influence total reflectance



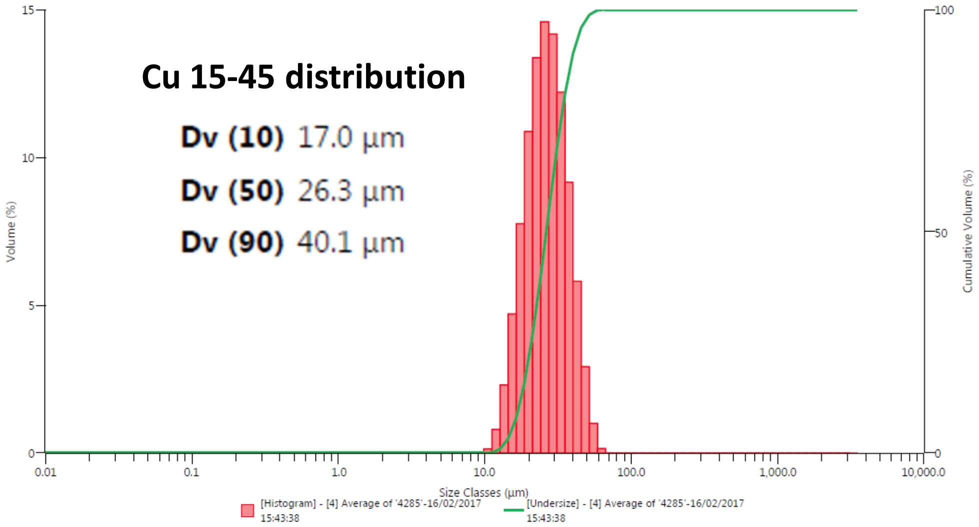
Quality control for both distributions

- **Spectrophotometer reflectance** with integrating hemisphere
- **Scanning Electron Microscope** powder analysis
- **Laser diffraction** particle size analysis

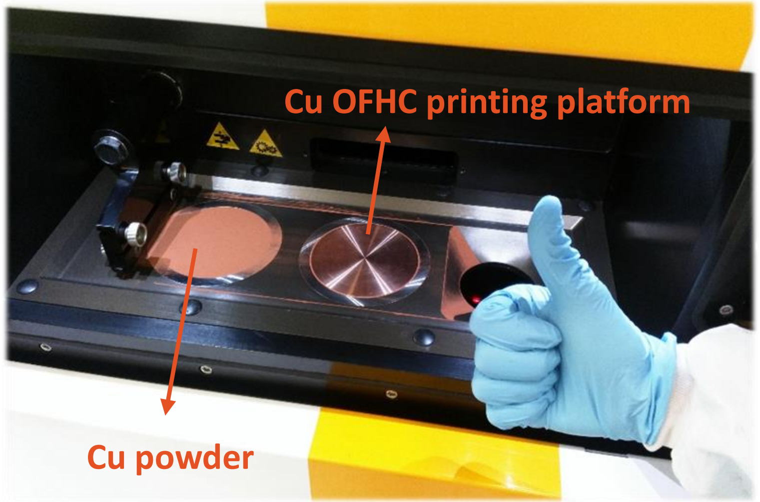
- Good overall quality for both powder distributions
- **Cu Fine** is **more spherical** and presents **less satellites** *(not shown)*



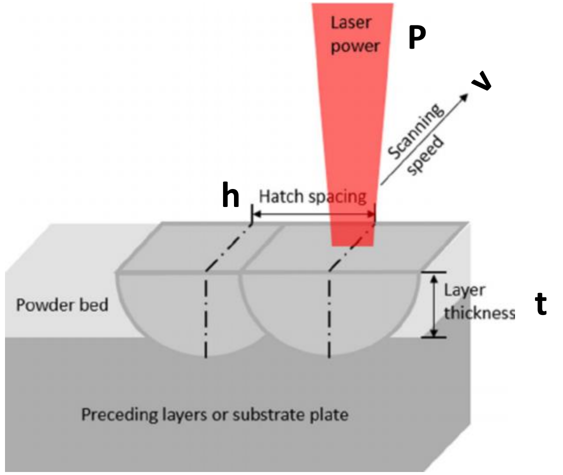
Cu 15-45 distribution

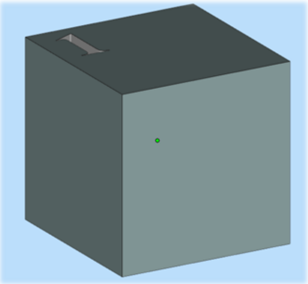


Cu 15-45 SLM test



Parameter	Value
Laser Power (P)	175 W
Inert gas	Nitrogen
Scanning mode	Parallel lines, 45° angle increment
Volumetric Energy (E_v)	From 200 J/mm^3 to over 1500 J/mm^3
Laser scan speed (v)	
Hatching space (h)	$E_V = \frac{P}{v \cdot h \cdot t}$
Layer thickness (t)	





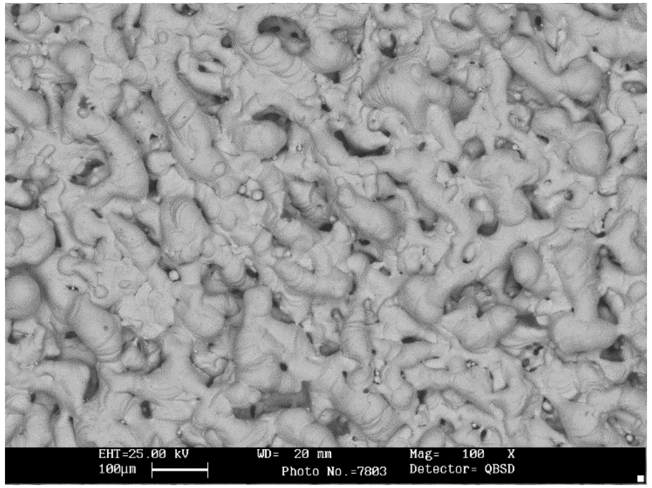
- 6x6x6 mm test cubes
- Groups of 9 cubes at different E_v
- Supports to the platform at 150 W, 600 mm/s

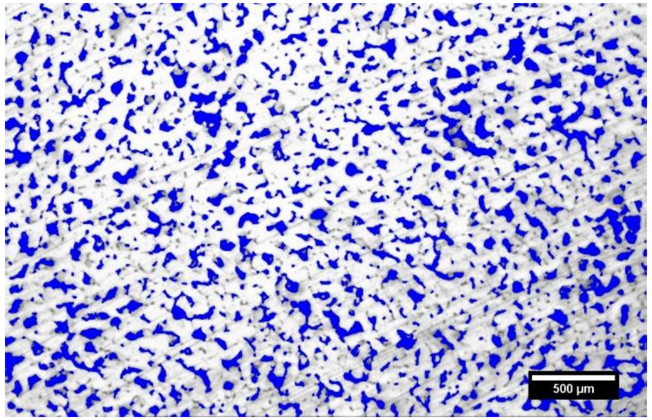
- **>70 cubes with different parameters !!**
- SEM analysis of xy top plane
- Digital image processing for porosity (optical, after metallographic polishing)

Cu 15-45 SLM parameters research

- **To validate previous Cu research literature**
- **To set a starting point and a comparison**
- **To test and train on mysint100PM machine**

Cu 15-45 SLM test





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- Digital image processing for porosity (optical, after metallographic polishing)

SEM OUTCOME (TOP SURFACE)

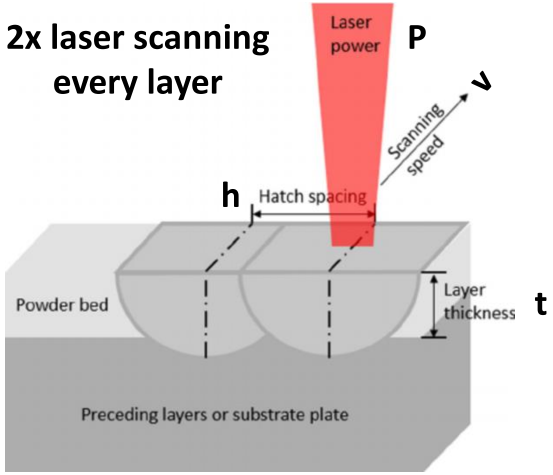
- **Melt beads structure**
- **Not continuous weld lines**
- **Lots of unfused particles**

OPTICAL OUTCOME

- **Confirmed high total porosity !**

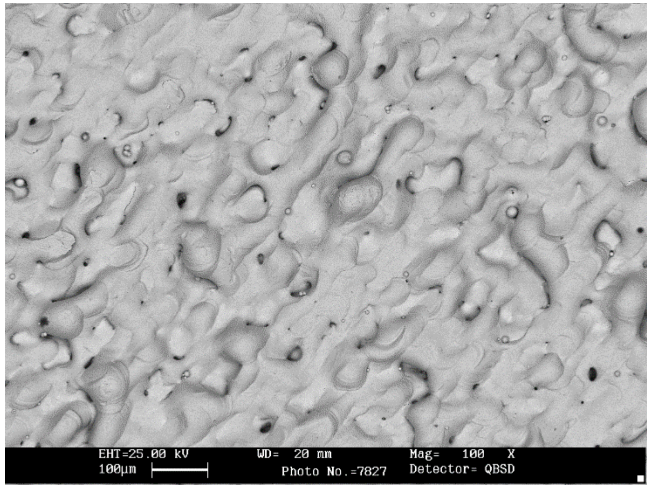
from 70 % to 88 % density
depending on parameters

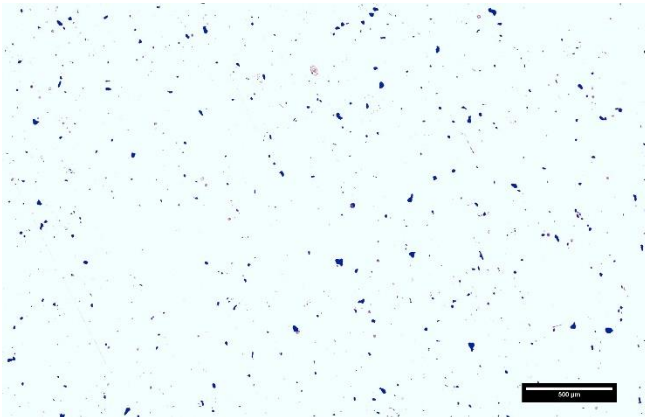
2x laser scanning every layer



Layer remelting also tested
→ no substantial improvement

Cu Fine SLM test





- **>70 cubes with different parameters !!**
- SEM analysis of xy top plane
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SEM OUTCOME (TOP SURFACE)

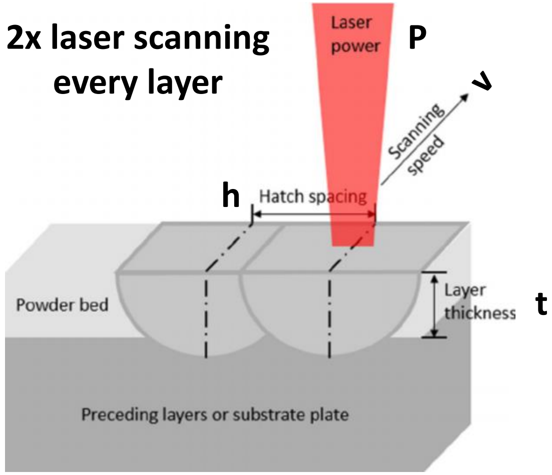
- **Better structure on surface**
- **Visible weld lines**
- **Still some unfused particles**
- **Still some beads and weld interruptions**

OPTICAL OUTCOME

- **Lower total porosity !**

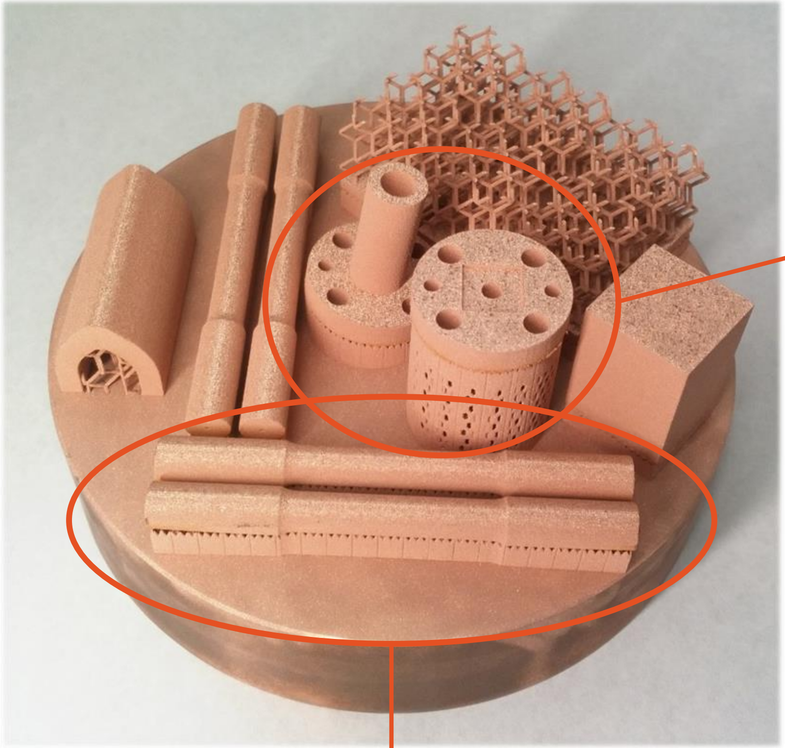
from 93 % to 99 % density
depending on parameters

2x laser scanning every layer



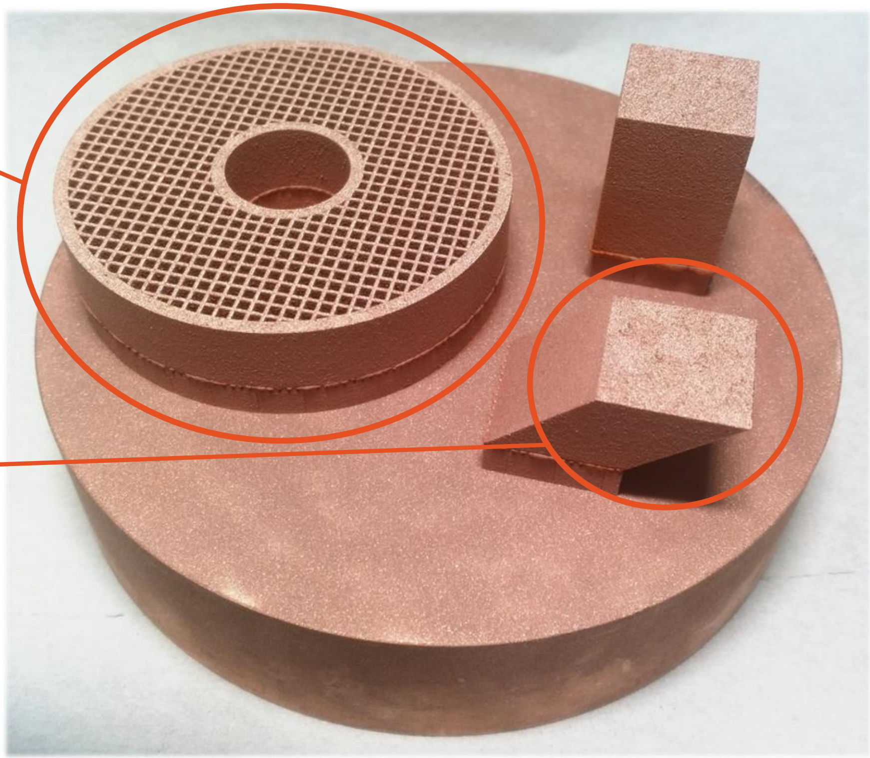
Layer remelting also tested
→ limited improvement

Cu Fine ex. prints



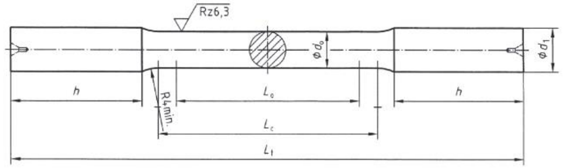
First commission for OFFICINA DEI MATERIALI

First commission for sisma

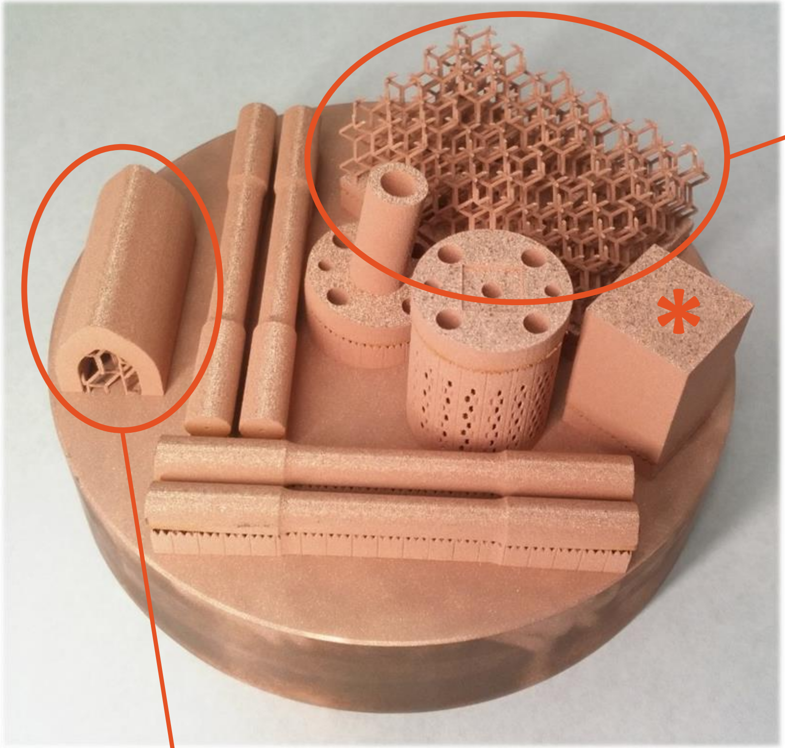


First roughness test specimen

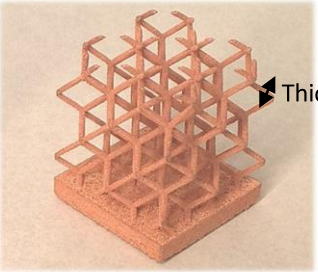
First XY ASTM mechanical test specimens



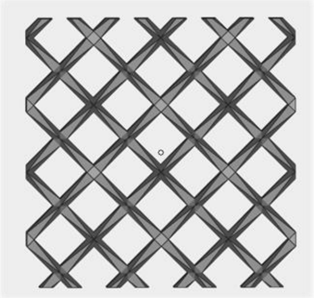
Cu Fine ex. prints



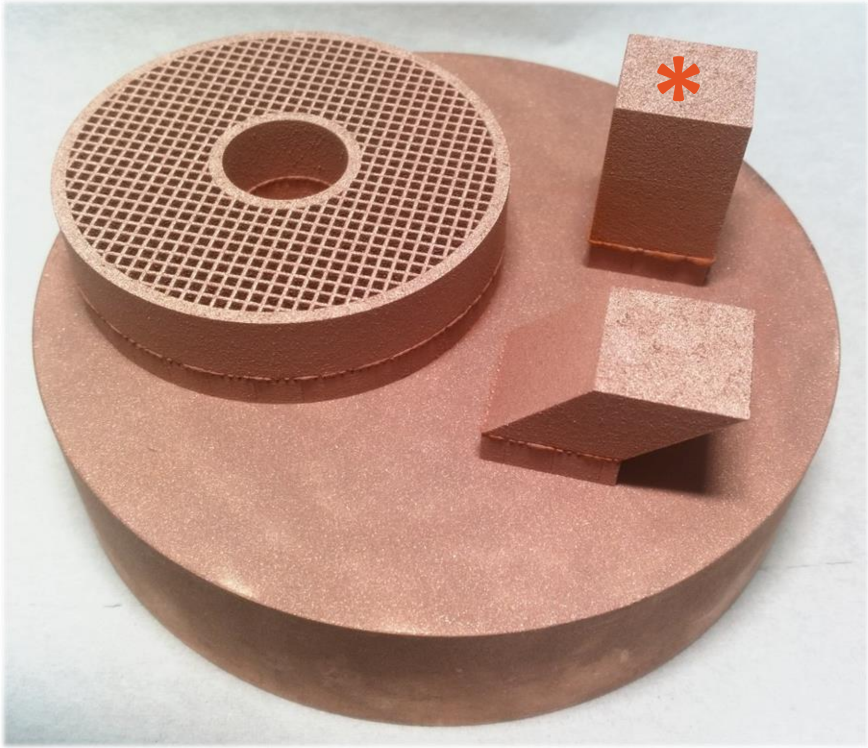
Complex structures example



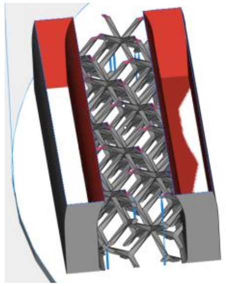
Thickness = 500 µm



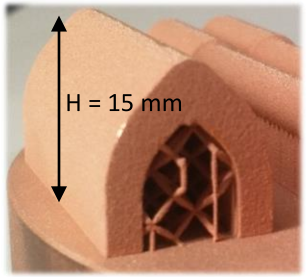
* Quality control prism



Beam dump channel example, directly welded to Cu platform



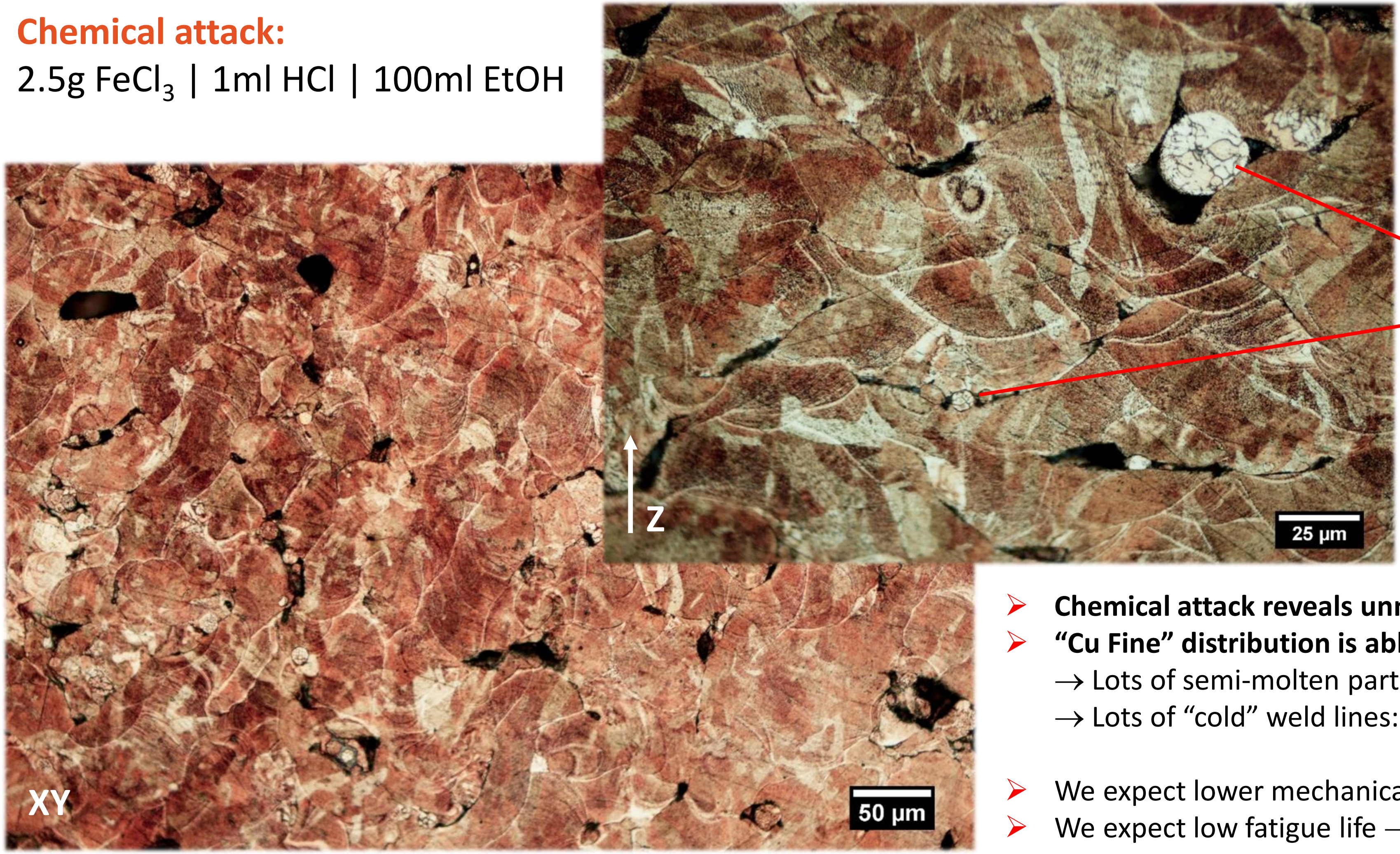
Z section and zoom
to perceive
the internal structure

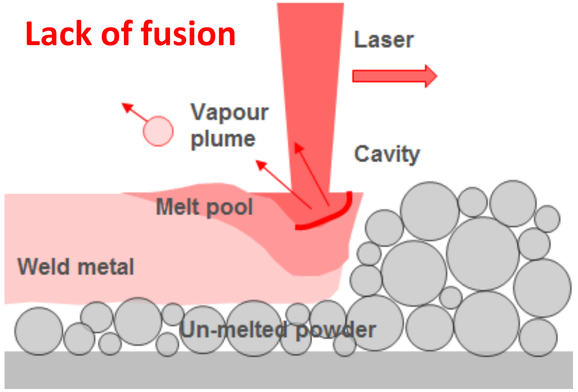


ALTHOUGH…

Chemical attack:

2.5g $FeCl_3$ | 1ml HCl | 100ml EtOH



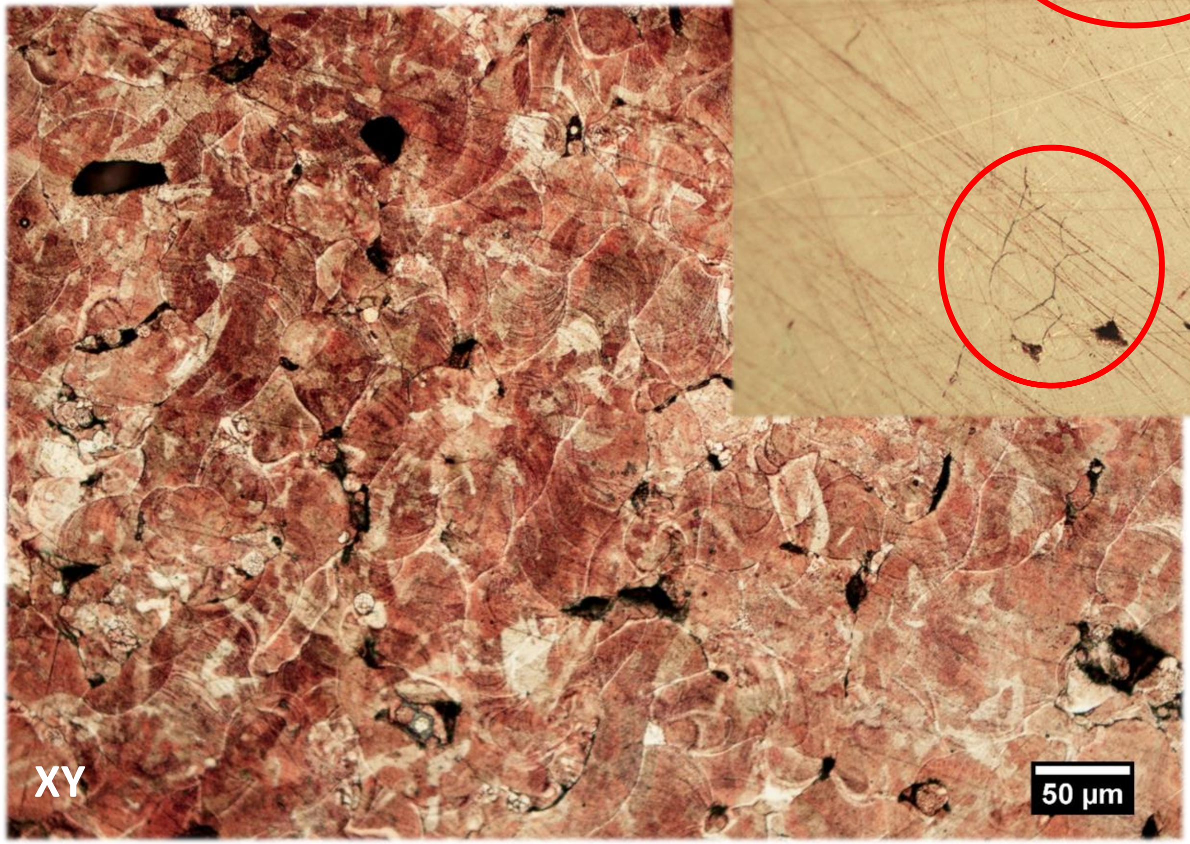


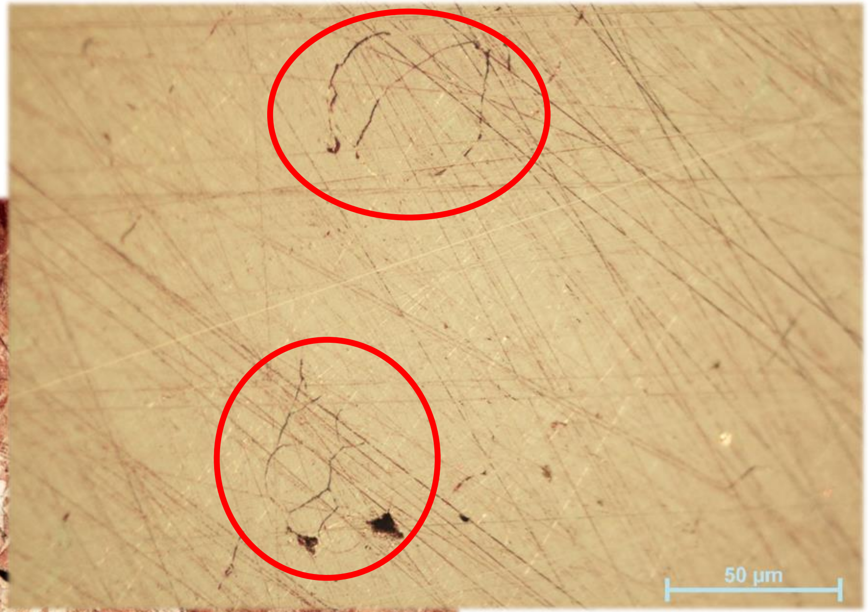
- **Chemical attack reveals unmelted powder and expands porosity**
- **“Cu Fine” distribution is able to fill voids during SLM process**
  - → Lots of semi-molten particles
  - → Lots of “cold” weld lines: two weld lines not properly fused together

- We expect lower mechanical properties in Z direction
- We expect low fatigue life → micro-cracked alike material

ALTHOUGH…

Cu Fine check





<u>OPTICAL DENSITY INSPECTION</u>

-even if properly done-

does not discriminate between bulk material and semi-molten particles

and it wouldn’t be easier with CT scan or Archimedes methods

- **Chemical attack reveals unmelted powder and expands porosity**
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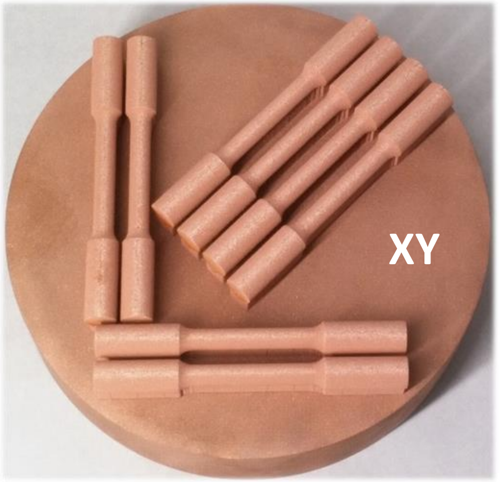
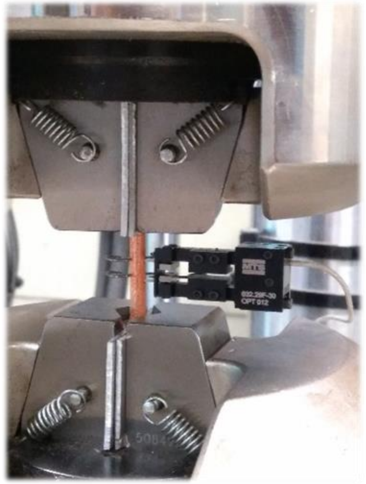
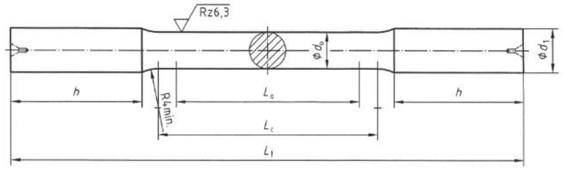
Cu Fine check

Mechanical tensile tests

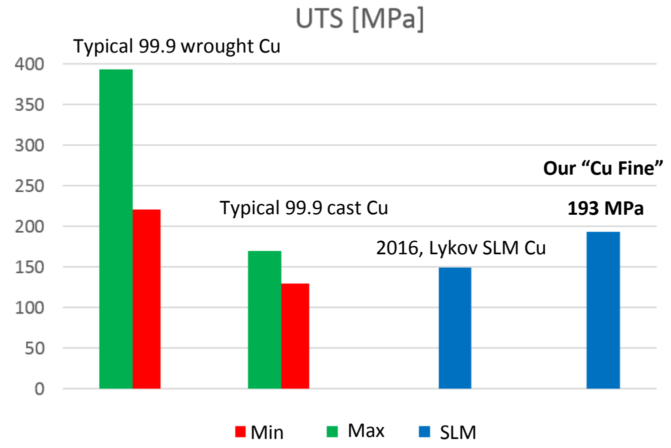


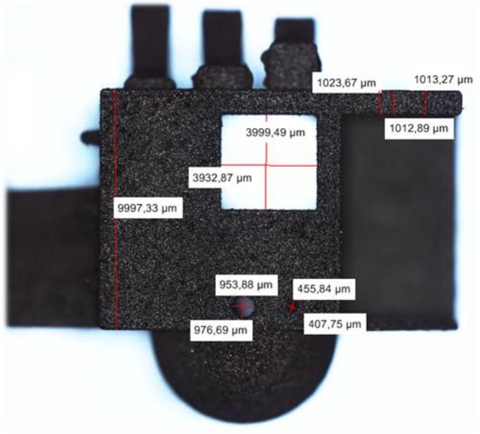


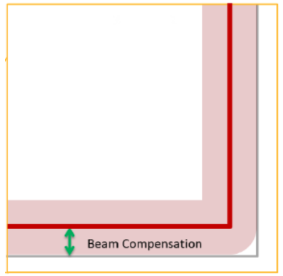


- Compliant with ASTM E8/E8M and DIN 50125
- First batch of 12 XY specimens tested
- Test on Z specimens is underway

➢ **Best UTS value so far for SLM pure copper**
➢ **Fine-grained microstructure helps strength**
➢ **Still lower than typical wrought Cu**

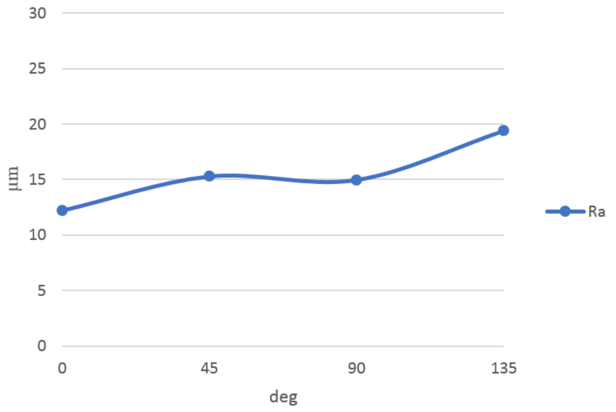






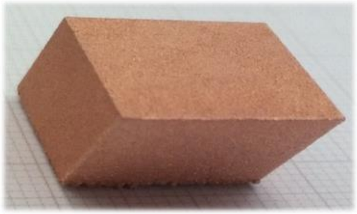
Beam compensation tuning

➢ **± ~40 µm on XY, ± ~20 µm on Z tolerances on dimensions**
→ checked with optical inspection
→ CMM measurement on the way



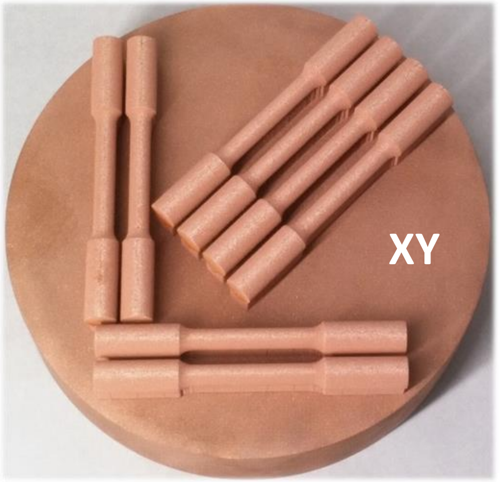
Optical profilometer test

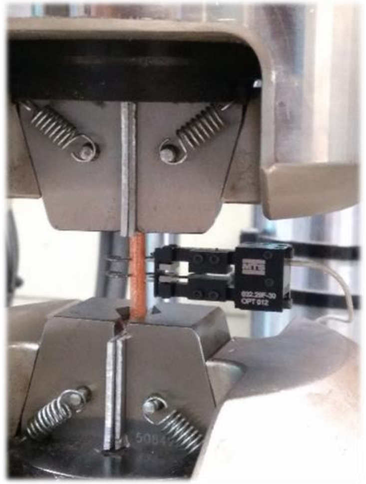
➢ **Good overall roughness**
similar to SISMA 90Cu-10Sn bronze

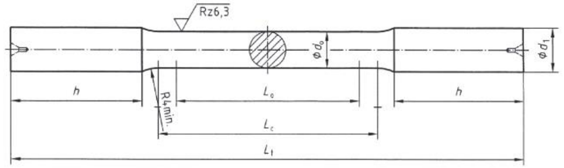


➢ Moreover, Cu prints do not require lots of supports (self-supporting till ~35° angle) and they are extremely easy to remove =D

Mechanical tensile tests







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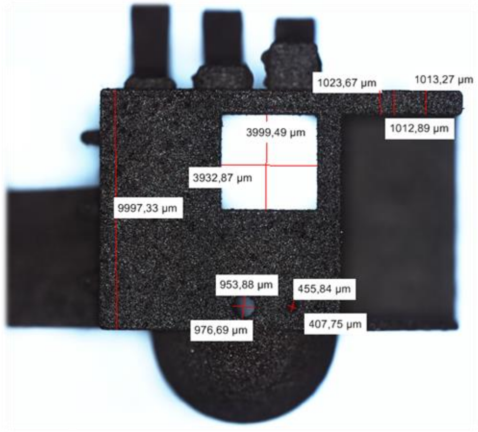
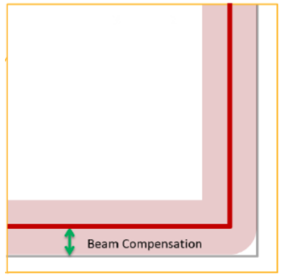
<u>*Data acquisition is supported by*</u>*:*

Eng. Valerio Pettinacci, INFN - Sez. di Roma
Eng. Donato Orlardi, INFN - LNGS

Prof. Giovanni Meneghetti, UNIPD - DII
Eng. Daniele Rigon, UNIPD - DII





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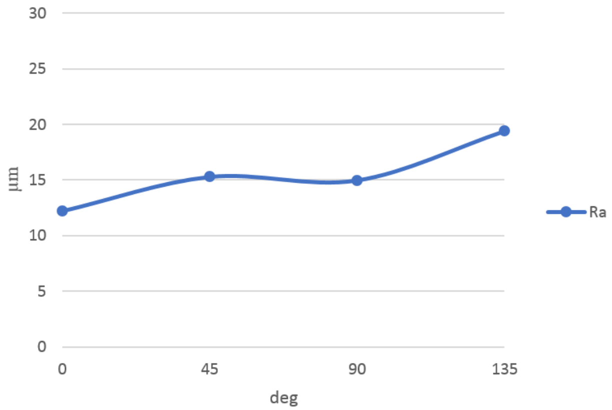

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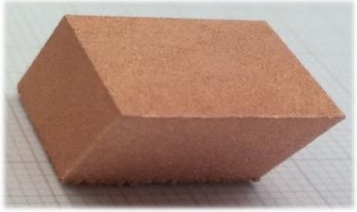

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FUTURE PLANS FOR SLM OF PURE COPPER AND CU ALLOYS WITH FINE SELECTED POWDER DISTRIBUTIONS

- The research will continue with the aim to produce copper and copper alloys **parts for high energy physics and nuclear physics experiments**

INFN Istituto Nazionale di Fisica Nucleare Sezione di Padova

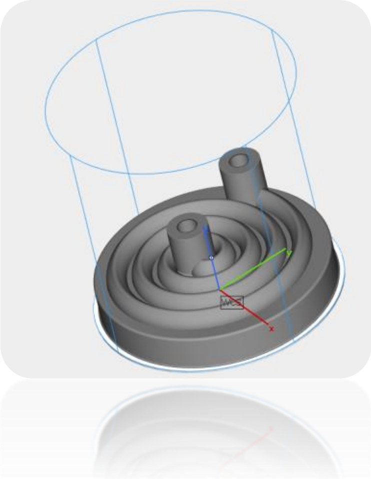
Next talk: “The New INFN Research Center on metal AM”

- OdM is currently drafting a business plan to became an **innovative startup AM oriented**, focusing on SLM of high conductive materials
- **New experimental research on Cu** SLM printed is planned regarding:
  →New selected distribution to be tested on lower layer thickness
  →New tests with different laser spot and laser scanning procedures
  →New tests on upgraded SLM machine

OFFICINA DEI MATERIALI

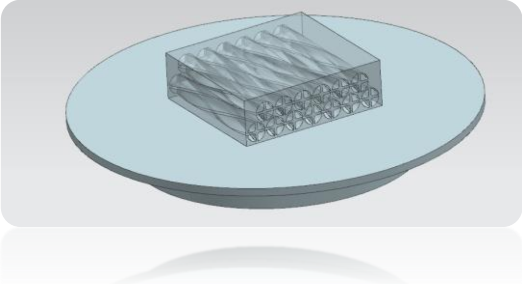
FIRST BEAM DUMP FULL PROTOTYPE PRINT

→ To evaluate potential problems on massive prints
→ To be stress-tested with ex: high pressure H_2O flow



NEW RESEARCH ON MICRO-HEAT EXCHANGING

OFFICINA DEI MATERIALI ⇄ INFN Istituto Nazionale di Fisica Nucleare Laboratori Nazionali di Legnaro



→ In collaboration with INFN - LNL (*Eng. Juan Esposito’ Group*)
→ New Cu SLM heat exchanging designs to be explored for future applications

Thank you for your kind attention

And a very special thank goes to my colleague, Giacomo, who shared with me this whole fantastic "additive" year =)