





Development of new materials for the SLM process

a project by:



sisma



Istituto Nazionale di Fisica Nucleare Sezione di Padova

speaker:

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



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

ENGIN

SOF



Coaching regarding SLM process and AM software

Support for metallographic examination and inspection

Access to SISMA's AM Lab and SISMA's facilities

Training on the job at SISMA SpA for >50 days



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

 Support on SLM powder supply



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- 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





- 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



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Example of SLM material

developed by a specific company



- 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		

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Example of SLM material

developed by a specific company

STAINLESSSTEEL CX

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



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SLM materials

Many other materials under development

- 2011 <u>research</u>, Fraunhofer ILT: Airbus Scalmalloy (**AlMgScZr**)
- 2013 research paper, L. Thijs: Pure Tantalum (Ta)
- 2015 <u>research</u> paper, S. L. Sing: **TiTa alloy** for biomedical app.
- 2017 <u>research</u>, A. Ivekovic: **Tungsten Heavy Alloy** (W₅Ta₇Ni₃Fe)
- 2017 research paper, J. Jaćimović: NdFeB Permanent Magnets

etc...

Example of SLM material

developed by a specific company



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

In Use		Under development				
HastelloyX		In718 In625			In713	17-4
Ti64	TiCP	CoCr			Al 7050	Copper
PH1 ((15-5)	31	6L		Niobium	Tungsten
Maraging Steel (MS1)		CM247LC				
AlSi10Mg		High Temp Copper Alloys				



Example of SLM material

developed by a specific company

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- Corrosion Resistant Stainless Steel for Tooling
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etc...





INDUSTRIAL CASE STUDY

OLD CU BEAM DUMP





- Beam Dump for proton H beam (INFN SPES project)
- Pure Cu OFE for heat dissipation (max <u>52.5 kW</u> !)
- 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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INDUSTRIAL CASE STUDY

NEW SLM CU BEAM DUMP



- Beam Dump for proton H beam (INFN SPES project
- Pure Cu for heat dissipation (max <u>30 kW on 21.4 cm²</u> !)
- Water cooled or He cooled

ADVANTAGES

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



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ADVANTAGES

Eventually the SLM printing platform can be part of the final object design (\$\frac{1}{2}\$ printing time and cost)

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



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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:** \downarrow reflectance, \downarrow thermal conductivity
- > Carefully control oxygen in the SLM machine chamber and in powder manipulation







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Advanced Heat Exchanger, lone example of <u>industrial</u> pure copper SLM

Under development

CM247LC

High Temp Copper Alloys

17-4

Copper

Tungsten



SLM materials

- 2016 <u>research</u> paper, P. A. Lykov
- 200 W CO₂ Pulsed Laser, 35 μ m spot
- ightarrow 88.1 % density and 149 MPa UTS
- 2017 <u>research</u> paper, L. Kaden
- Ultrashort laser pulses at MHz repetition rate, 1030 nm, 35 μm spot
- ightarrow ightarrow Low density and melt beads structure





- 2011 <u>research</u>, Fraunhofer ILT
- Copper Alloy: Hovadur K220, CuNi₂SiCr

In713

AI 7050

Niobium

- 1000 W Laser, modified SLM machine
- \rightarrow 99.9 % density

A lot more research on SLM of <u>–</u> <u>Copper Alloys</u>

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 <u>research</u> papers
- Mixtures and special Cu Alloys being developed



INFN

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- Ultrashort laser pulses at MHz repetition rate, 1030 nm, 35 μm spot
- \rightarrow Low density and melt beads structure
- 2015 <u>research</u>, NASA
- Copper Alloy: GRCop-84, Cu-Cr-Nb
- 400 W Fiber Laser, ~50-100 μm spot
- \rightarrow >99.5 % density after HIP post-processing

In Use				
HastelloyX		In718	In625	
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CAD Manufacturing route User **Process Preparation** Part Geometry Material Composition Grinding Absorptivity Drving Accuracy CAD model Humidity-h Gas atomization-Cleani -Electrolyt Volume model Single-phase-Time --Orientation Temperature Optical properties Water atomizatio Two-phase-Sleving -Surface model LFF System Powder Heat conductive Recycling Reflectivity Transmissiv Data Quality Maintenance cross section -Particle ovidation - Humidity Slicing --Max. chord tolerance Particle shape Melt viscosity Smallest featuares Flipped triangles Particle size distribution Oxidation Bad edges Rheological Spherical Spattered - Flowability - Contamination Triangle size Properties Bulk densit Surface STI conversion Surface tension Welting angle State Particle surface -> -> Particle size Overall size Ultimate Break Yield Accuracy — Particle properties Training-Density strength strength strain Fatigue Creep Therma Heat Heat Thermal Mechanica Expansion onductivity Capacity Diffusivity coefficient Properties Laser focus deviation Building Supported Experience -Thermal Properties Fracture Young's Shear Bulk Polsson's Platform Freeform faces Complexity Positioning Surface angle thoughness modulus modulus modulus ratio Strengt Latent Melting Vaporisation Heat Conductivity heat temperature temperature 1-0-error Shape -Dissolving powder Reaction enthalpy Liquidus-solidus transition Generatio Oxygen affinity Stiffness -Metallurgical Solid/Porous Undercuts Platform Part Wall Chemical Propertie Absorptivity -> properties teeth teeth distance Supports Diffusion constant Material Alloy Product quality of solid SLM parts Exposure strategy Scanner Number of Number of Cost -Intensity profile Mode Wave Length lavers parts Scan direction(x,y)=>> Accuracy Laser Contou Diameter Refectivity Scan Form tolerance Alternating scan-Beam Hatch Beam Laser Duration Vetor power distance offset Galvos length Contour scan-Accuracy BPP CW/pulsed Max. CW/pek powe Process Recoating time Overshooting Mass Temperature parameters Beam Expander Shape tolerance Beam delivery Point distance -Zoom factor-Platform Laver Focus Aperture thickness temperature Z-shift Roughness Lav Exposure time --) Number of Divergence Diameter Oxygen % Idle time Process steps Collimation-I urface finish Scan speed Fibe Process Control Duratio Beam Forming Platform Waviness Focal length-Average Flow rate Transportation Powde Size ->/ energy Colimated density spattering beam radius Type of gas Residual stress - Accuracy Gaskets-Focus radius (x/y)-Process response Process gas supply Thermal treatment Mechanical Effective therma Components f – O - lens **Relative density** diffusivity Strength Wiper wear--Width -Hardness Cross jet function Pressure Powder bed Power bed Density Break strain roughness Process Leveline-Effective Effective heat Gas supply Support removal Melt pool Microstructure conductivity absorptivity Coating system Recirculation Filtering SLM System Process Post-processing

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Our SLM Cu Recipe





MAIN USE OF THE DIAGRAM

Our SLM Cu Recipe



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Our SLM Cu Recipe



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SISMA mysint100PM

Eng. Mirko Sinico

TWO SELECTED PURE CU POWDERS

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



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





Our SLM Cu Recipe

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% 0 ₂
0_2 concentration - Concentrazione 0_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

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- ~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)







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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)	Р
Hatching space (h)	$E_{\rm V} = -\frac{1}{L}$
Layer thickness (t)	$v \cdot h \cdot t$

Cu 15-45 SLM test



- 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



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SEM OUTCOME (TOP SURFACE)

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

Cu 15-45 SLM test



OPTICAL OUTCOME

Confirmed high total porosity !

from 70 % to 88 % density depending on parameters



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

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or set

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

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

Cy Fine SLM test



Layer remelting also tested \rightarrow limited improvement



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Complex structures example





Beam dump channel example, directly welded to Cu platform



Z section and zoom to perceive the internal structure



Cy Fine ex. prints

***** Quality control prism





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ALTHOUGH...

Cu Fine check





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ALTHOUGH...





OPTICAL DENSITY INSPECTION -even if properly donedoes 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
- "Cu Fine" distribution is able to fill voids during SLM process
 → Lots of semi-molten particles
 - \rightarrow 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 \rightarrow micro-cracked alike material



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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

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Cu Fine check



1023,67 µm 1013,27 µm 1012,89 µm



Beam compensation tuning

- \pm ~40 μm on XY, \pm ~20 μm on Z tolerances on dimensions
 - \rightarrow checked with optical inspection
 - \rightarrow CMM measurement on the way



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



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Optical profilometer test

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



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Cu Fine check

Data acquisition is supported by:

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

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





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

What's next

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



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
 - \rightarrow New tests on upgraded SLM machine



FIRST BEAM DUMP FULL PROTOTYPE PRINT

 \rightarrow To evaluate potential problems on massive prints \rightarrow To be stress-tested with ex: high pressure H₂O flow

NEW RESEARCH ON MICRO-HEAT EXCHANGING

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



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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 =)



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15 september 2017