



# **KET Lab**

*THE FUTURE'S LABORATORY*

**Additive manufacturing of  
copper-based nanocomposites**

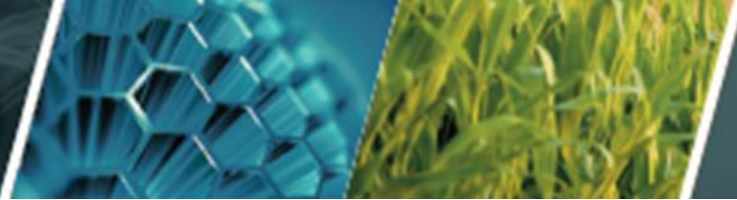
*Nanoinnovation Rome 18-09-2020*

**Strictly confidential**



**HYPATIA**

CONSORZIO DI RICERCA SULLE TECNOLOGIE PER LO SVILUPPO SOSTENIBILE



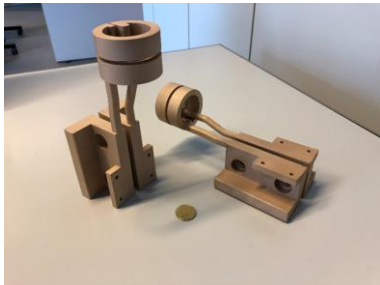
## Summary:

- Consorzio Hypatia , KetLab and Advanced manufacturing department presentation
- Pure copper development for SLM process
- Preliminary observations
- Carbon nanoparticles in pure copper matrix and analysis
- Comparison between pure copper and copper-carbon composites
- Conclusions

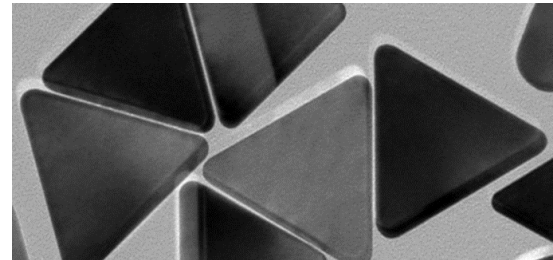
In the 2016 , Consorzio Hypatia and the Italian Space Agency (ASI) reached a partnership agreement to create the KetLab - Key Enabling Technologies LABORatory.

KetLab is now focused mostly on three main segments:

- Metal Additive manufacturing



-Life science



-Advanced Materials and thin film deposition



## Advanced Manufacturing Department of KetLab



Sisma MySint 100



Prima Additive Print Sharp 250

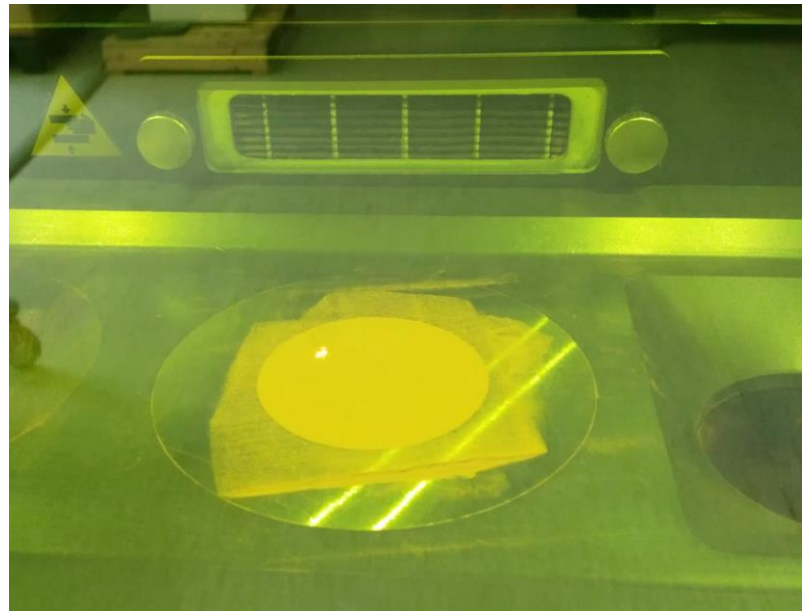




## Powder Bed Fusion

Basical requirements of PBF processability:

- High absorption of the heat source used: laser or electron beam
- Low thermal conductivity
- Low surface tension of the liquid phase



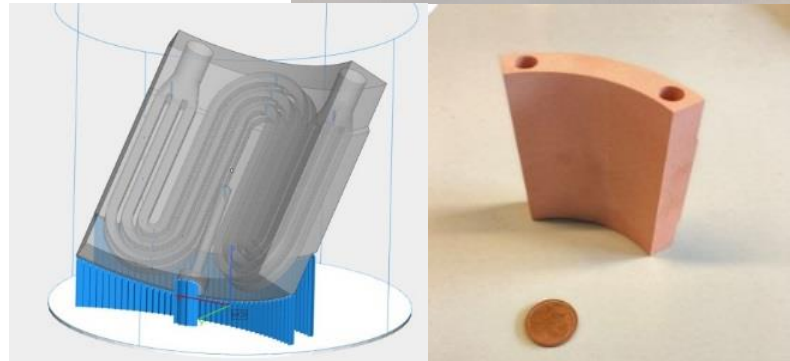
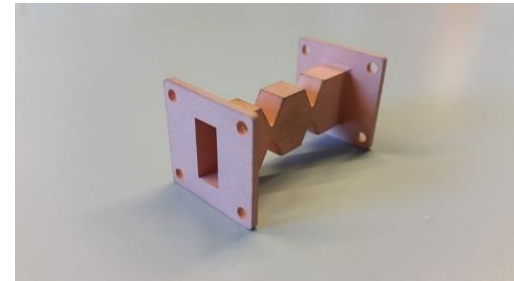
## Why Copper?

Physical characteristics of Cu-ETP electrolytic copper

- Density = 8.9 g / cm<sup>3</sup>.
- Electrical conductivity =  $5.8 \times 10^7$  S / m. Corresponding to a resistivity of 17.24 nΩ / m equivalent to 100% IACS (International Annealed Copper Standard).
- Thermal conductivity  $\approx 400$  W / (mK).
- Tensile strength = 200-250 N / mm<sup>2</sup>.

IACS of common metal alloys:

Pure Al	60% IACS
Al 5052	35% IACS
Carbon steel	8.5% IACS
AISI 304	2.3% IACS



## SLM system

SISMA MySint100

Maximum production volume = 100x80 mm

Maximum laser power = 200W

Fiber laser

Laser spot = 30  $\mu\text{m}$

Wavelength = 1070  $\pm$  10 nm

Productivity = 5  $\text{cm}^3$  / h

Gas used: Nitrogen



## Pure Copper powder

### Chemical composition

Copper min 99.9% of spherical shape produced by atomization in Nitrogen. Oxygen content less than 0.05%.

### Morphological analysis

The 95% by weight has a diameter of less than 32  $\mu\text{m}$  with the following particle size distribution: D10-8  $\mu\text{m}$ , D50-18  $\mu\text{m}$ , D90-35  $\mu\text{m}$ . Apparent density 4.8 g / cm<sup>3</sup>.

### Reflectance analysis with Ulbricht sphere

LED 940nm at 15mW with opening angle  $\pm 10^\circ$ , PIN photodiode detector with maximum absorption spectrum at 940nm and width  $\pm 150\text{nm}$ .

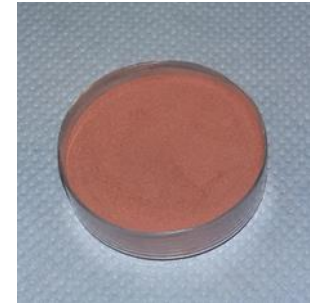
Percentage of signal reflected: 84.9%

Examples of materials commonly used in SLM and the corresponding reflected signal percentage:

AlSi10Mg 29.9%

316L 32.8%

CoCr 32.8%



## Density optimization

P=175 W; Layer=10 um fixed

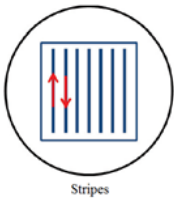
Optimal laser settings:

P=175W

V=600mm/s

HD=0.04mm

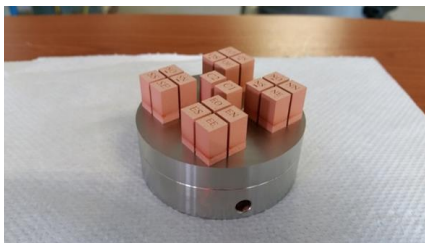
Strategy



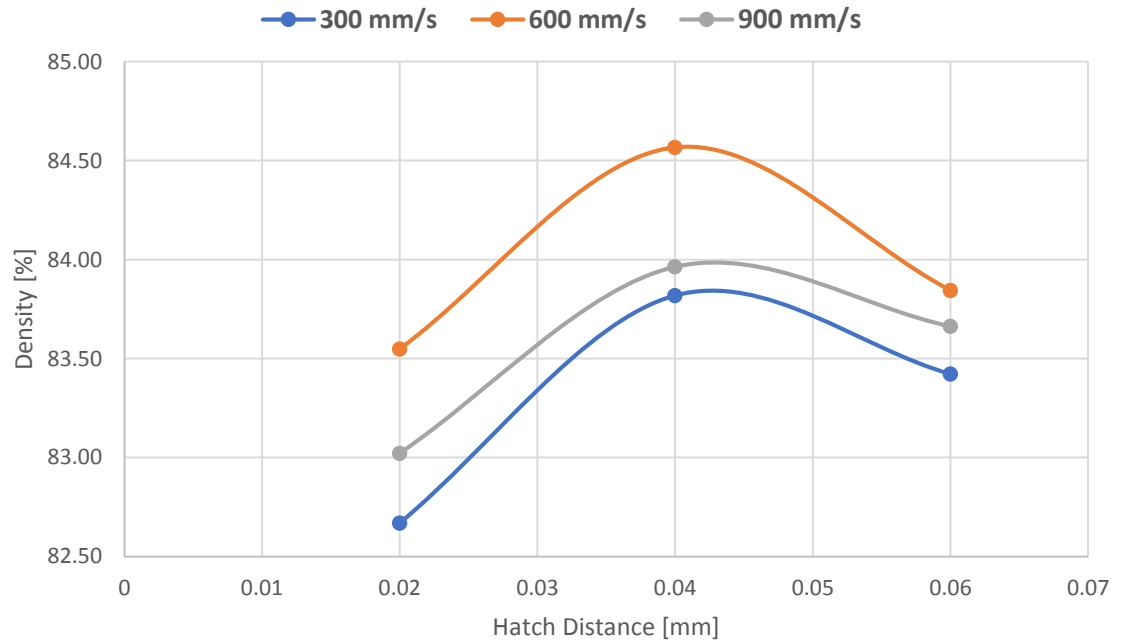
Stripes

Maximum:

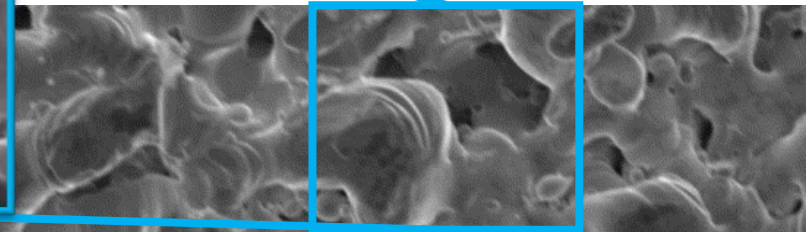
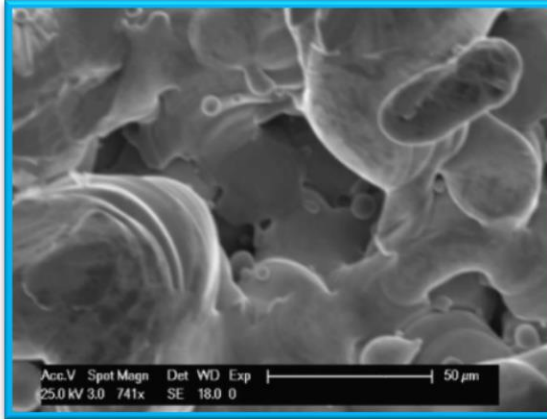
84.6%



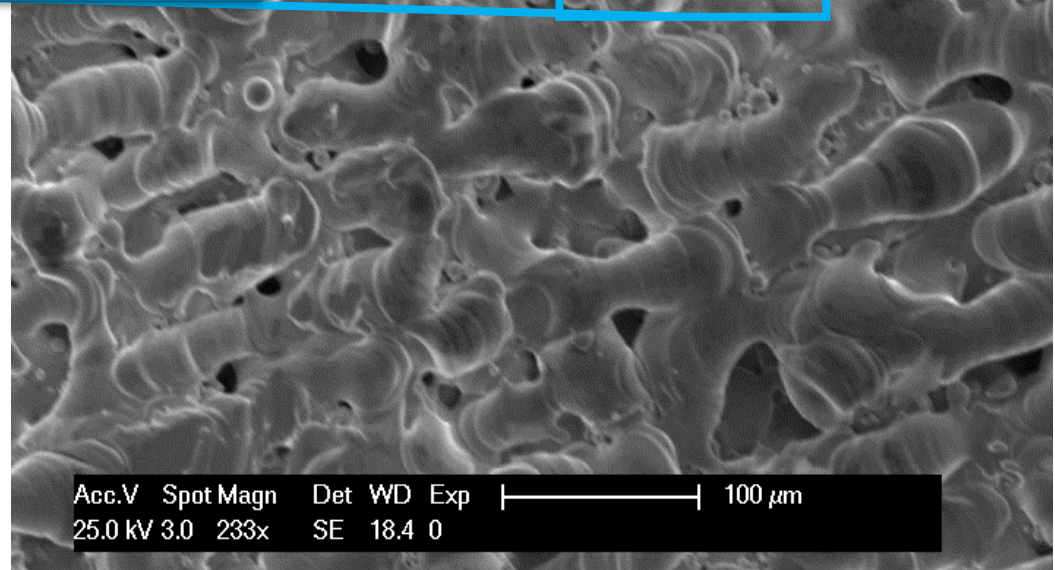
Parameters	Levels		
Speed (mm/s)	300	600	900
Hatch Distance (mm)	0.02	0.04	0.06



## SEM analysis



Not continuous laser traces  
and widespread holes



## Electrical and thermal conductivity

Wiedemann-Franz

$$\frac{K}{\sigma} = L T$$

Where L is the *Lorentz constant*

$$L = 2.44 \times 10^{-8} \text{ W}\Omega\text{K}^{-2}$$

IACS= 44.5 %

$(\sigma = 2.58 \times 10^7 \text{ [S/m]})$

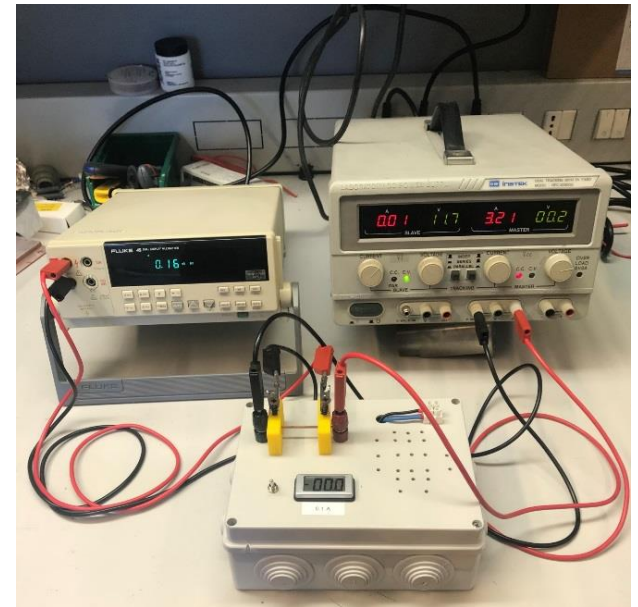
$K = 184.52 \text{ [W/(m K)]}$

$(\approx 40\% \text{ of bulk pure copper thermal conductivity})$



Specimen dimensions

$\phi = 2\text{mm}, h = 60\text{mm}$



## Pure Copper Tensile test

Comparison of mechanical behaviors of specimens produced with vertical and horizontal major axis.

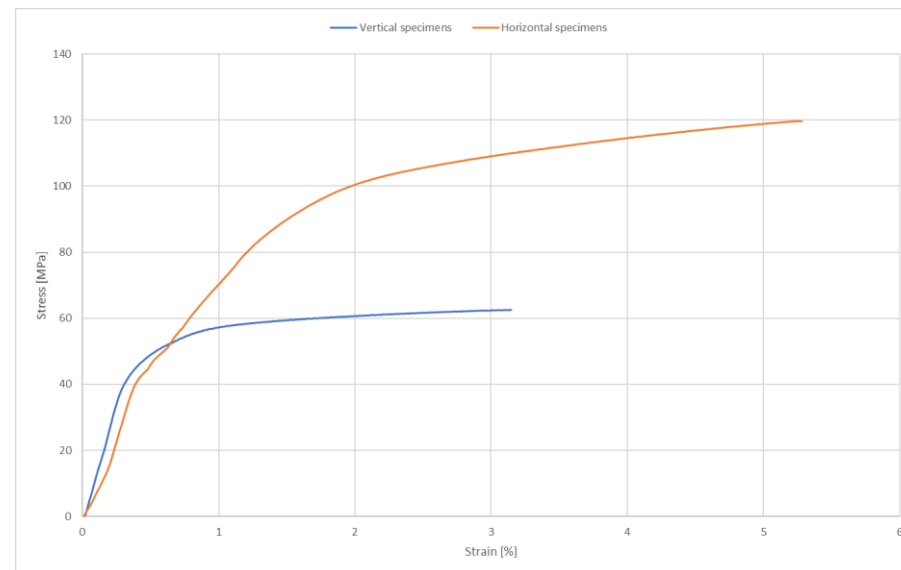
	Peak Stress [MPa]	Strain at Break [%]	Young's Modulus [GPa]
Vertical	62.62 ± 6	3.14 ± 0.1	13.2
Horizontal	119.37 ± 1.2	5.25 ± 0.1	9.7

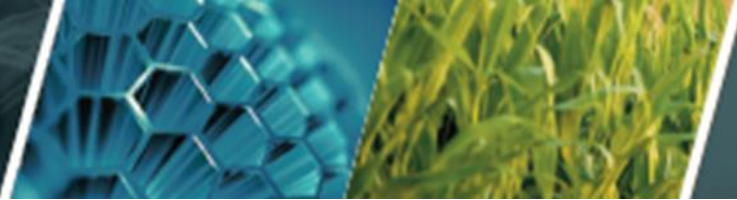
Peak stress Cu bulk:  
200/250 MPa

Young's Modulus Cu bulk:  
117 GPa



Test performed with MTS Alliance RT/50 Universal testing machine



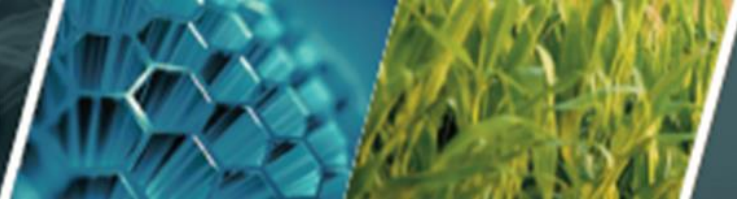


## Preliminary observations

The results shown do not represent an end point considering mainly the density and mechanical properties found in the specimens.

Two strategies have emerged:

- Change the hardware, especially the heat source
- Modify the composition of the metal powder to try to improve the mechanical properties without penalizing the electrical / thermal conductivity



## Pure Cu powder + C nanocomposites(CN)

Graphene was supplied by Sigma-Aldrich (Milan, Italy).

Form: Brown/Black powder

Bulk Density:  $\sim 1.8 \text{ g/cm}^3$

Solubility: Dispersible in water and polar solvents

0.1% w/w of CN was added to Cu powder and mechanically mixed for 12 h through a custom-made epicyclical gear system.

## Pure Cu powder + C nanocomposites (CN)

Reflectance analysis of mix by means of an Ulbricht sphere

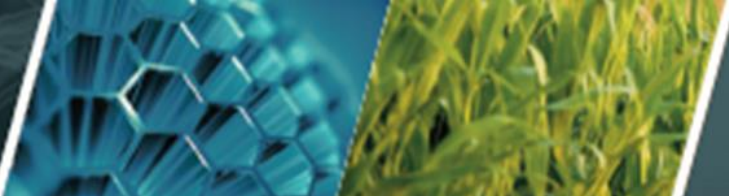
Reduction of reflectance with the addition of graphene dispersed in Copper powder.

Percentage by weight of CN: 0.1%

Copper powders analyzed and relative percentage of reflected signal:

Cu powder	84.9%
Cu powder + CN	57.1%





## Pure Copper Powder with 0.1% CN

### SEM Analysis

#### Best parameters:

P = 175W

V = 1000 mm/s

HD = 0.06mm

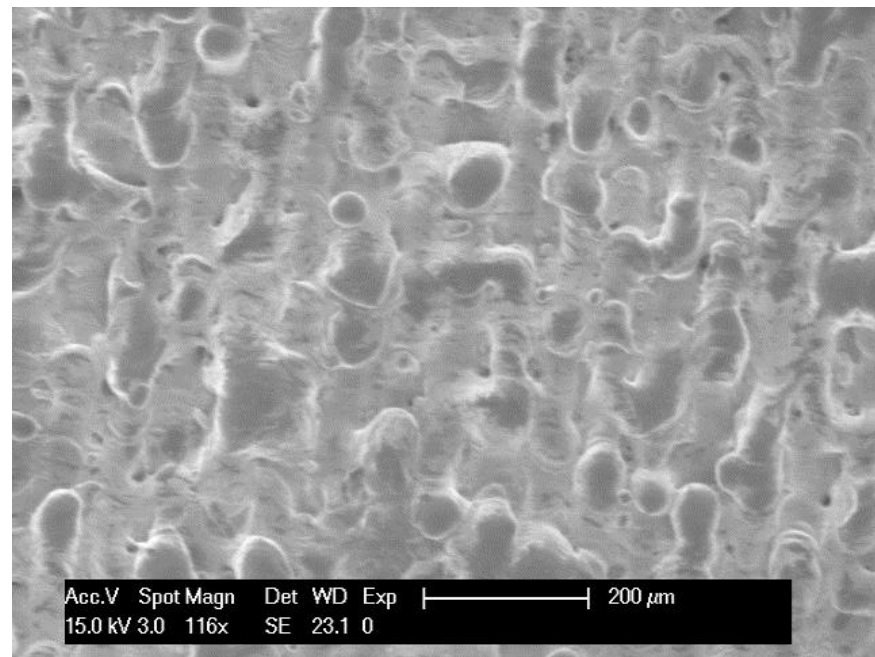
Holes reduction and  
more continuous laser traces

Maximum  
density:

**91.09%**

( reference value 84.5%)

Mean	89.893
StDev	1.065
Variance	1.133

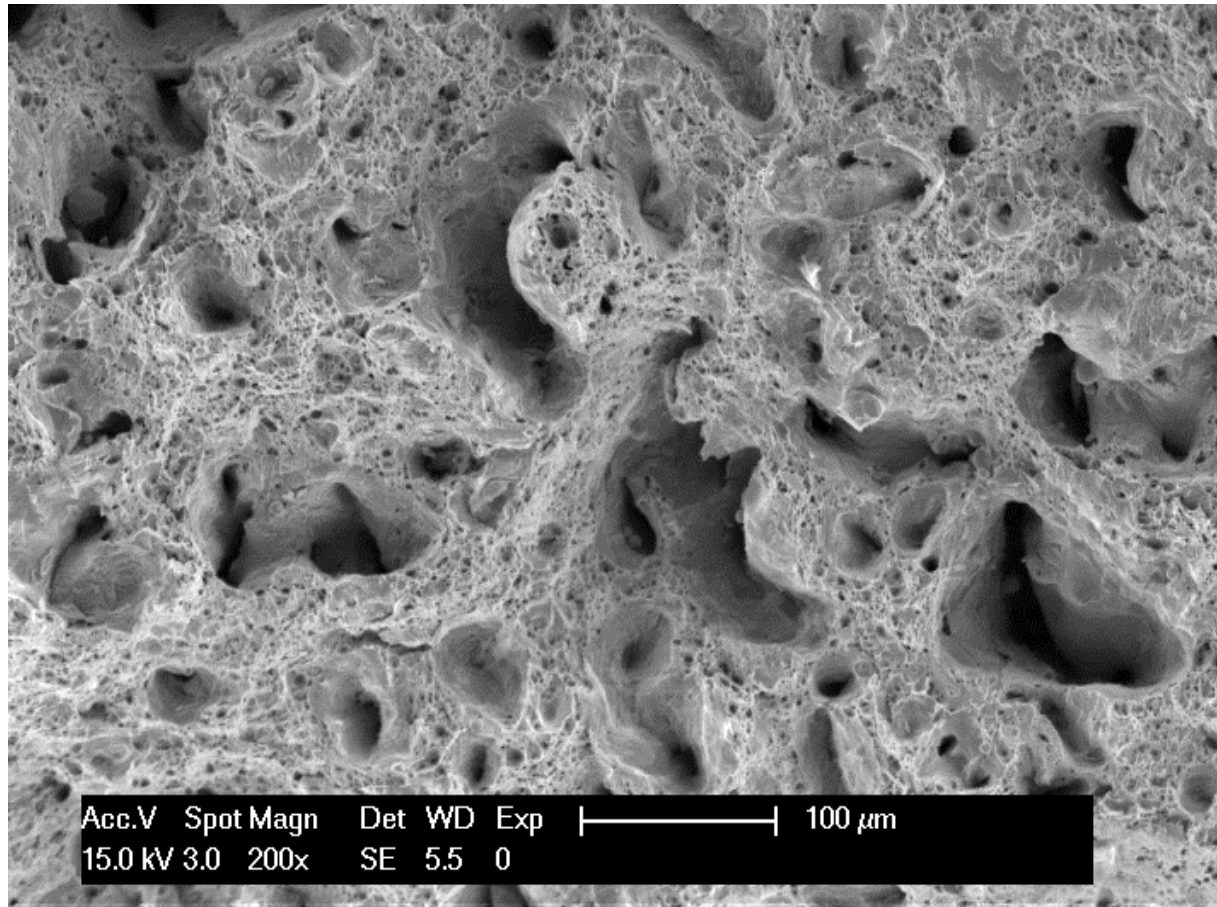




## Pure Copper Powder with 0.1% CN

### SEM Analysis

Fracture surface



## Electrical and thermal conductivity

Wiedemann-Franz

$$\frac{K}{\sigma} = L T$$

Where L is the *Lorentz constant*

$$L = 2.44 \times 10^{-8} \text{ W}\Omega\text{K}^{-2}$$

IACS= 63.53 %

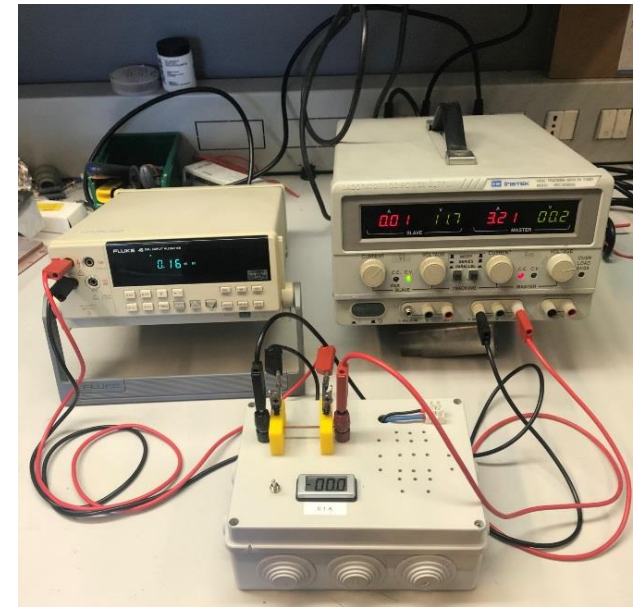
( $\sigma = 3.69 \times 10^7 \text{ [S/m]}$ )

$K = 266.67 \text{ [W/(m K)]}$

( $\approx 66\%$  of bulk pure copper thermal conductivity)



Specimen dimensions  
 $\phi = 2\text{mm}$ ,  $h = 60\text{mm}$

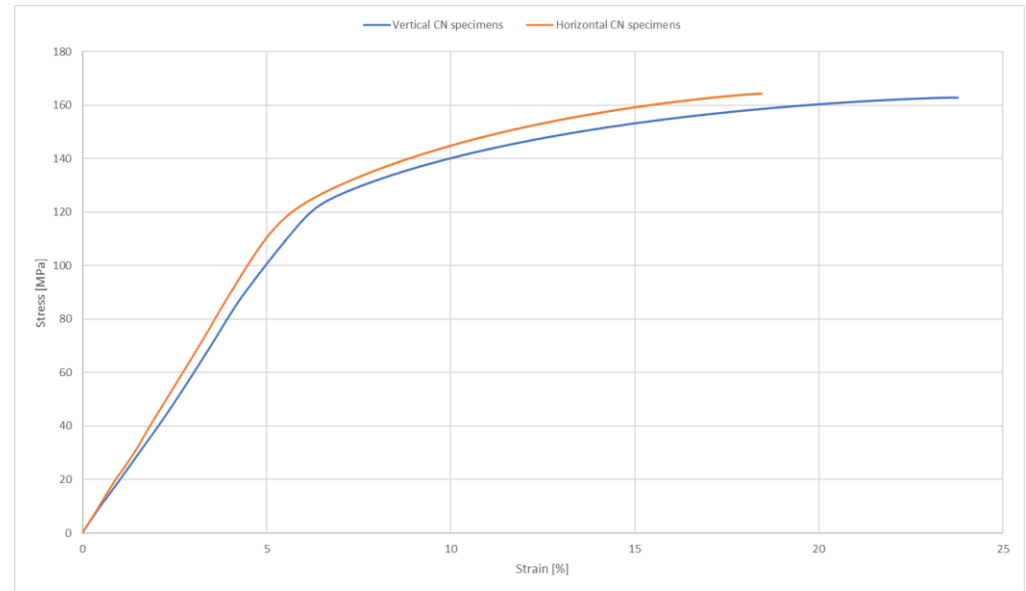


## Copper+CN Tensile test

Comparison of mechanical behaviors of specimens produced with vertical and horizontal major axis.

Peak stress Cu bulk:  
200/250 MPa

Young's Modulus Cu bulk:  
117 GPa



	Peak Stress [MPa]	Strain at Break [%]	Young's Modulus [GPa]
Vertical	162.86 ± 2	23.86 ± 0.1	2.00
Horizontal	164.37 ± 6	18.35 ± 0.1	2.12



## Comparison Pure Copper vs Copper+ CN

	Pure Copper	Copper +CN	
Density [%]	84.6	91.09	+7.7%
Vertical peak Stress [MPa]	62.62	162.86	+160.1%
Vertical Strain at break [%]	3.14	23.86	+659.9%
Vertical Young's modulus [GPa]	13.2	2	-84.8%
Horizontal peak Stress [MPa]	129.37	164.37	+27.1%
Horizontal Strain at break [%]	5.45	18.35	+236.7%
Horizontal Young's modulus[GPa]	9.7	2.12	-78.1%
Electrical Conductivity [IACS %]	44.5	63.53	+42.8%
Thermal Conductivity [W/(mK)]	184.52	266.67	+44.5%

## Conclusions

The results show an increase in all mechanical characteristics, with the exception of Young's modulus, with the addition of Carbon nanocomposites.

The decrease in reflectance causes a better absorption of the fusion energy thus creating a more homogeneous structure.

Carbon does not lack thermal and electrical conductivities.

To conclude, we can say that the addition of carbon nanocomposites proves an improvement of the fusion in SLM and offers ideas for further developments. For example we will try other quantities in copper matrices and other mixing systems.

**THANK YOU FOR THE ATTENTION !!!**

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