Additive manufacturing for rare events searches

Work Package 5.6

Development and tests of Ultrapure copper structures made through 3D printers and innovative electroforming techniques

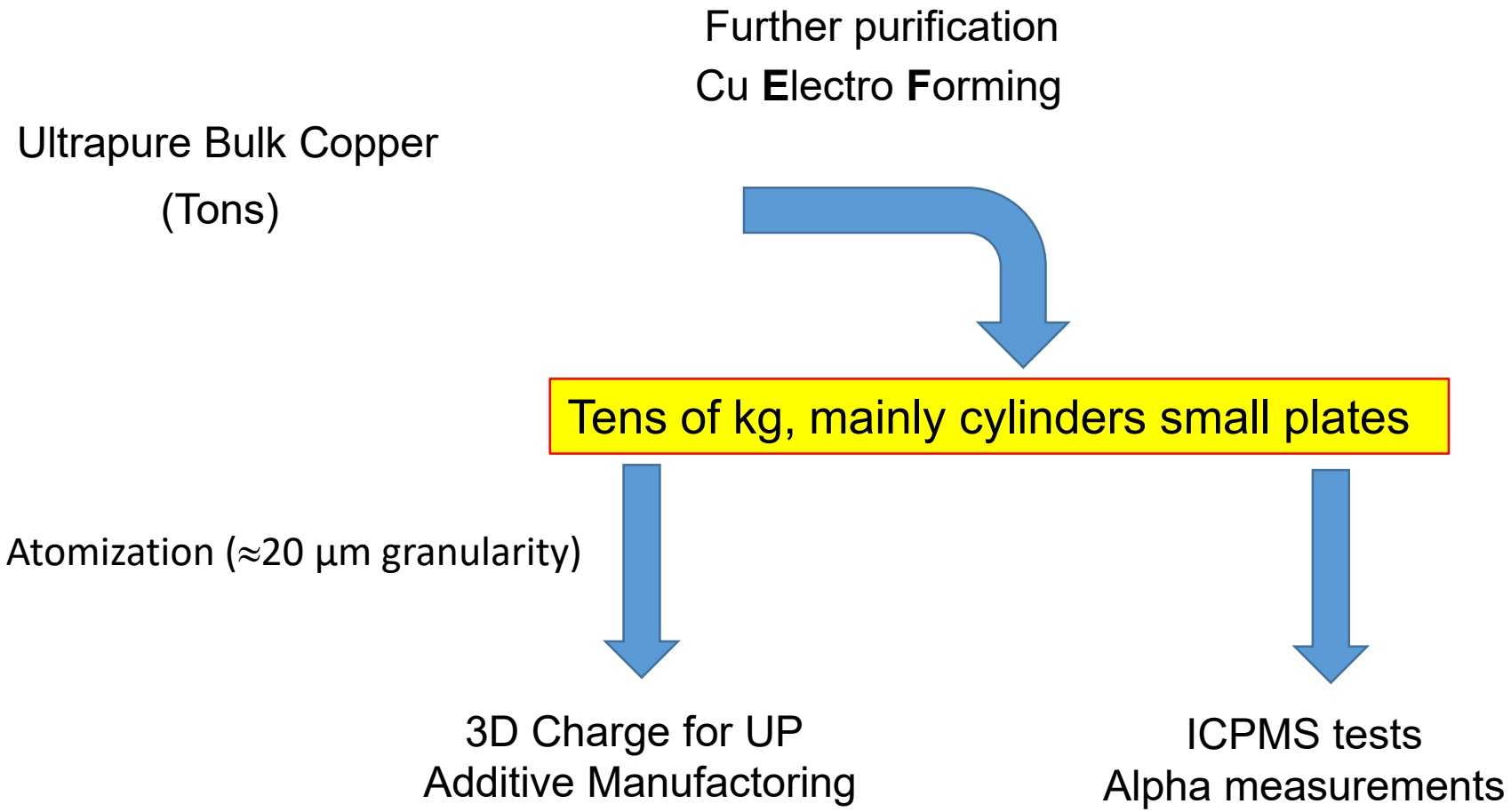
LNGS - Stefano Pirro

LSC – Silvia Borjabad Sánchez

LSM- Ali DastGheibi Fard

Apogeia Meeting, LNGS 28-04-2022

Copper plays a fundamental role in most of detectors components used for ultra-low radiopurity background experiments, ranging from Double Beta Decay (CUORE, GERDA, EXO…) to Dark Matter experiments (CRESST, CDMS, ANAIS…)

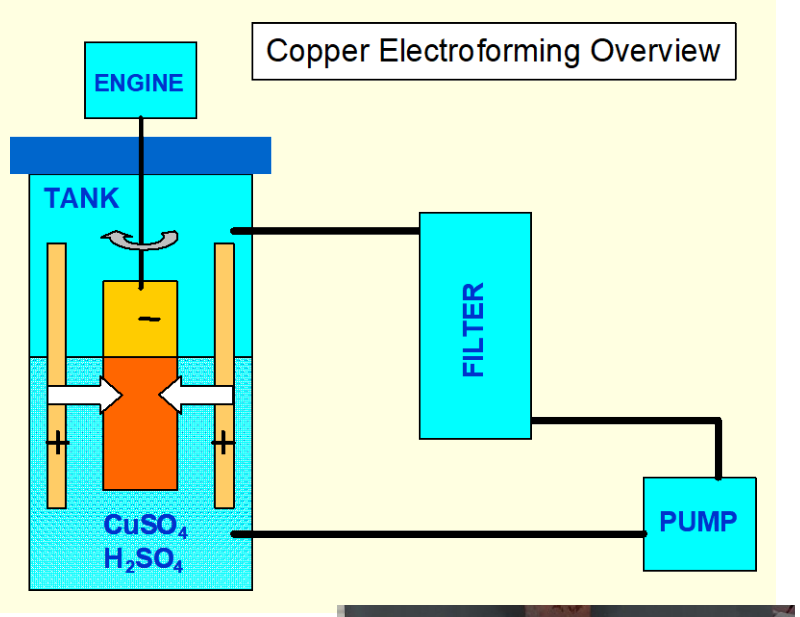


LSC CES (Copper Electroforming Service) Technique and setup

Silvia Borjabad - LSC

Electroforming is a method of producing pieces by the deposition of a metal onto a mold, which is subsequently removed.

- Pulse-reverse electroplating technique
- High-purity commercial chemicals & ultra-pure water
- OFHC copper (anodes) & stainless steel 316L (cathode)



1 set-up operative underground at the LSC Clean Room

Mandrels in different geometry

LSC CES (Copper Electroforming Service)
ICPMS assay and Gamma spectrometry measurement

ICP-MS assays carried out to analyze the Th and U contamination at the surface and bulk in OFHC-Cu (anodes, raw material) and EF-Cu at LNGS and PNNL.

Gamma spectrometry measurement performed using a HPGe detector (GeOroel, 2Kg Ge coaxial p-type, ~100 % relative efficiency) in EF-Cu at LSC.

OFHC Copper	ICP-MS assay					
	LNGS surface [mBq kg^{-1}]	LNGS surface [ppt]	LNGS bulk [mBq kg^{-1}]	LNGS bulk [ppt]	PNNL [mBq kg^{-1}]	PNNL [ppt]
U	0.6049	**49**	0.01234	**<1**	0.00242	**0.196**
Th	1.1382	**280**	0.01869	**4.6**	0.00393	**0.967**

EF Copper	Gamma spectrometry HPGe		ICP-MS assay					
	LSC GeOroel [mBq kg^{-1}]	LSC GeOroel [ppt]	LNGS surface [mBq kg^{-1}]	LNGS surface [ppt]	LNGS bulk [mBq kg^{-1}]	LNGS bulk [ppt]	PNNL [mBq kg^{-1}]	PNNL [ppt]
U	**< 11.2 (^{234}Th)**	< 907.2	0.2716	**22**	< 0.012	**<1**	< 0.000614	**<0.0498**
Th	**< 2.23 (^{228}Ra)**	< 548.5	0.1992	**49**	< 0.0406	**<1**	0.000147	**0.0363**

AIP Conference Proceedings 1921, 020001 (2018); https://doi.org/10.1063/1.5018987

LSC CES (Copper Electroforming Service)

WP5. 6

The copper powder used to feed the 3D printer has to be produced in a totally controlled ultraclean way. One of the aim of this WP is therefore the production of ultrapure copper powder starting from EF copper.

LSC available structures

EF-Cu equipment + materials (1ton Cu Grade A+EF chemicals) + ISO6/ISO7 Clean Room

LSC Deliverables

- Studies and selection of surface cleaning protocols in copper electroformed pieces suitable for future common applications across network laboratories
- Design and construction of enlarged electrolytic bath to increase the production capacity and to obtain different geometries in the copper electroforming facility

LNGS-3D printing service

This WP will take huge advantage from Hammer

HAMMER is focused on design and Hi-Res complex devices manufacturing for both nuclear & astro-particle physics research and industrial technology transfer and services.

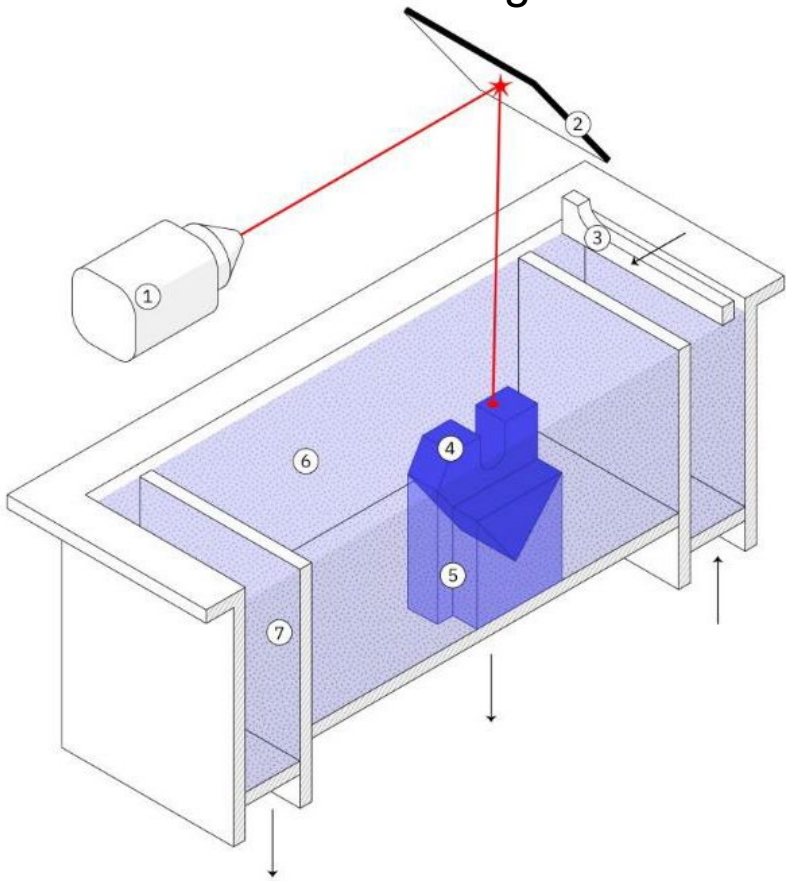
HTTPS://HAMMER.LNGS.INFN.IT/

Contact: Donato Orlandi

Additive Manufactoring for dummies

How does Metallic Additive Manufacturing work? Similar to all other 3D printing processes, metal 3D printers produce objects by adding material ***one layer at a time.***
In this way it is possible to build objects with geometries that are impossible to produce with "traditional" subtractive (CNC machining) or training (Metal Casting) technologies, without the need for specialized equipment (for example a mold).
The process that any metal 3D printer goes through to produce an object varies greatly by technology:

Selective **L**aser **M**elting



Main Printing parameters

- **P – Laser Power (100 – 1000 W)**
- **H – single layer Height (20 – 50 µm)**
- **V – Scan Velocity (100 – 3000 mm/s)**
- **D – Hatch-Distance (20 – 100 µm)**

- Inhert gas (Argon, Nitrogen)
- Heating of the base (50 – 200 °C)

Energy density

$$E = P / (H\ V\ D)$$

≈25000 J/mm^3

Additive Manufactoring for dummies (SLM)

The dimensions of the build platform usually range from **100 x 100 x 100 mm** (for R&D machines), up to 300 x 300 x 300 mm (for production machines), but larger machines are also available (up to 500 mm). The dimensional accuracy that a metal 3D printer can achieve is approximately ± 0.1 mm, depending on the manufacturer.

When the construction process is finished, the parts are ***completely covered with the metal powder***. The parts are attached to the build platform via support structures. The support in metal 3D printing is constructed **using the same material** as the part and is always needed to mitigate the warping and distortion that can occur due to high processing temperatures.

As the printed object cools to room temperature, excess powder is **manually removed** and parts are typically heat treated while still attached to the build platform **to relieve any residual stress**. The components are then detached from the build plate by cutting, machining or wire EDM and are ready for use or for further post-treatment.

Metal powder is highly recyclable - typically less than 5% is wasted. After each print, the unused powder is collected, sieved and then refilled with new material to the level required for the next build.

Additive Manufactoring for dummies

Description of the production process :

1) The build chamber is first filled with **inert gas** (e.g. nitrogen or argon) to minimize oxidation of the metal powder and then heated to the optimum build temperature.

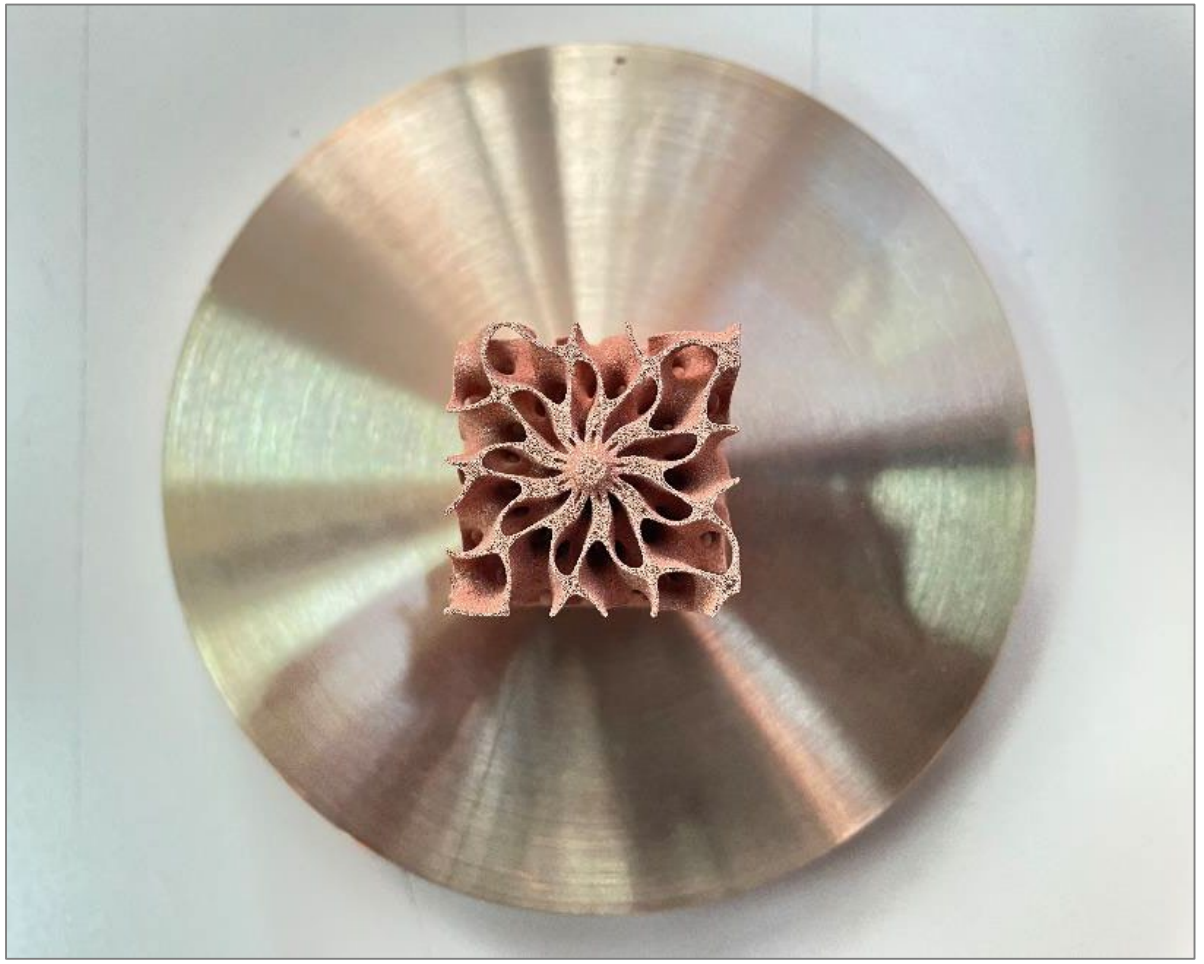
2) A thin layer of metal powder is spread over the build platform and a high-powered laser scans the cross section of the component, fusing the metal particles together and creating the next layer.

3) When the scanning process is complete, the build platform moves down according to the thickness of the layer and the spreads another thin layer of metal powder. The process is repeated until the entire part is completed.

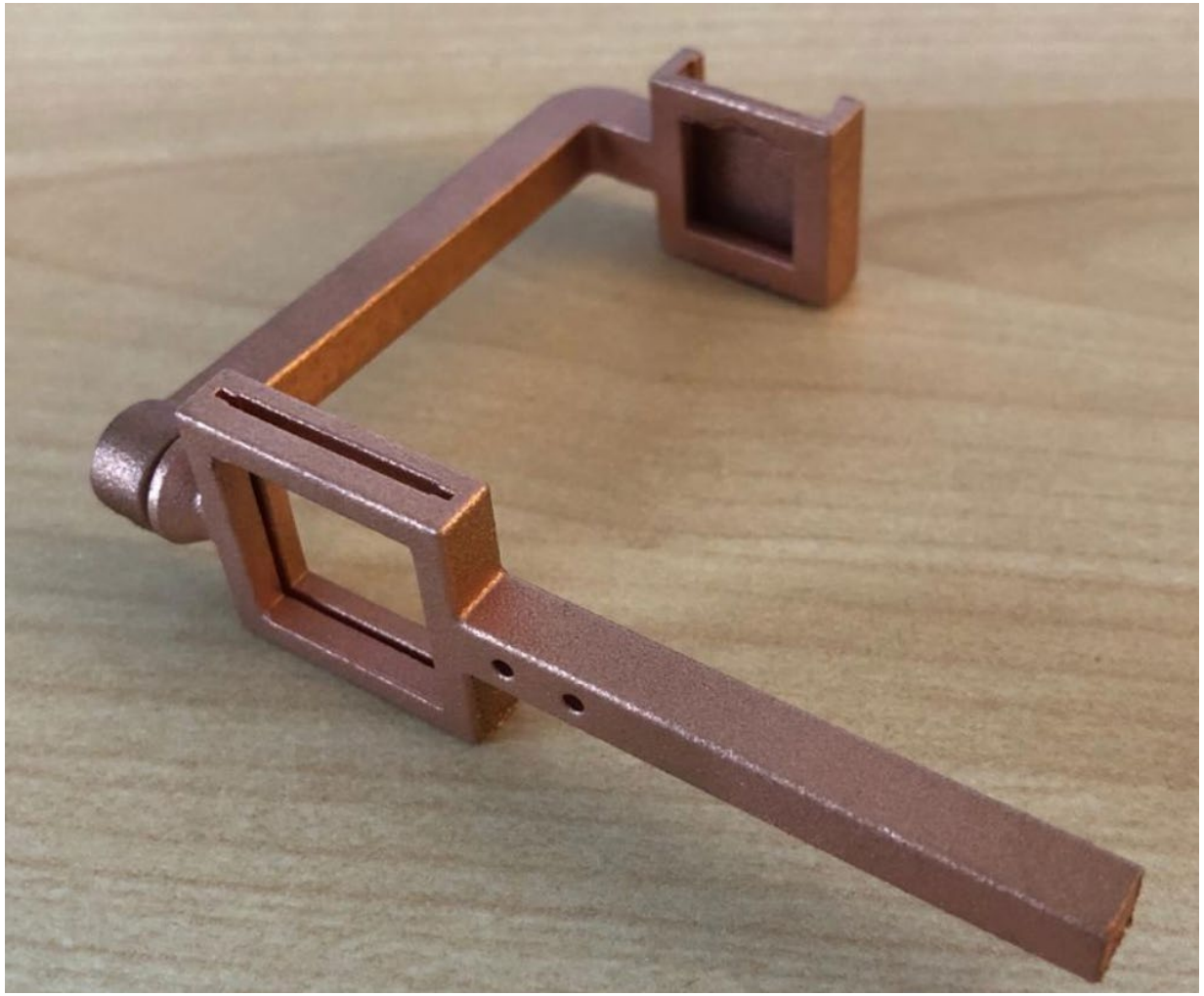
Additive Manufactoring for dummies

https://www.youtube.com/watch?v=yiUUZxp7bLQ

SLM Cu-printed objects @ HAMMER (LNGS)



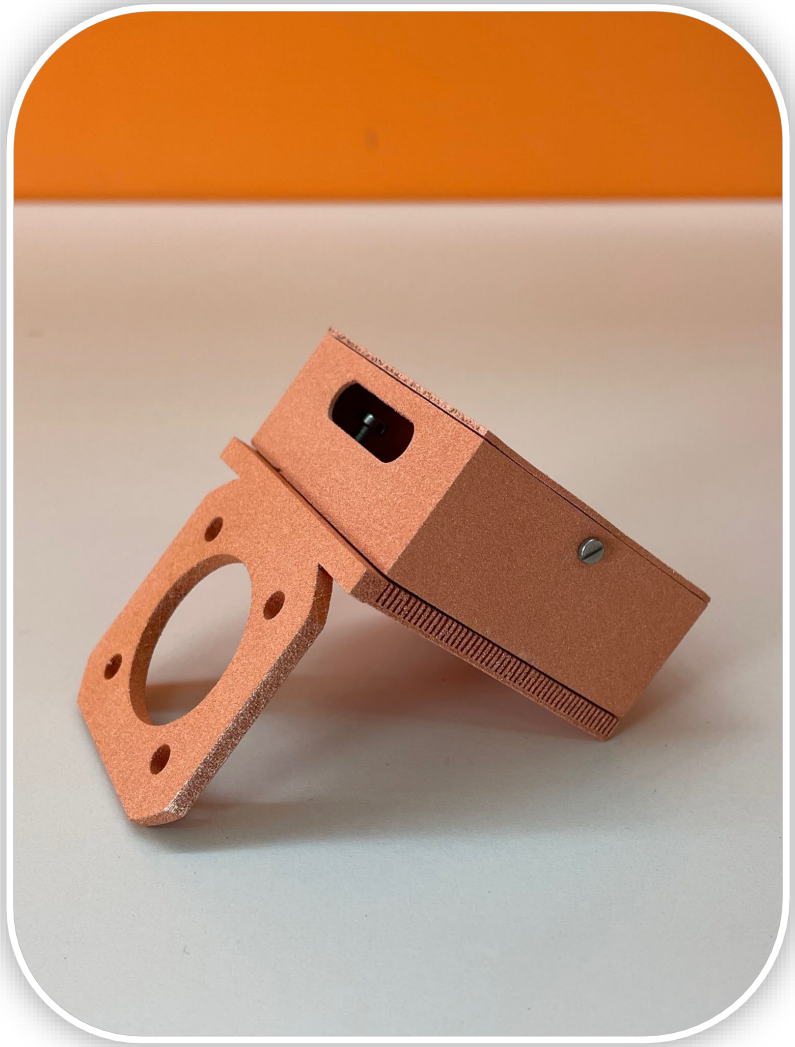
SLM Cu-printed objects @ HAMMER (LNGS)

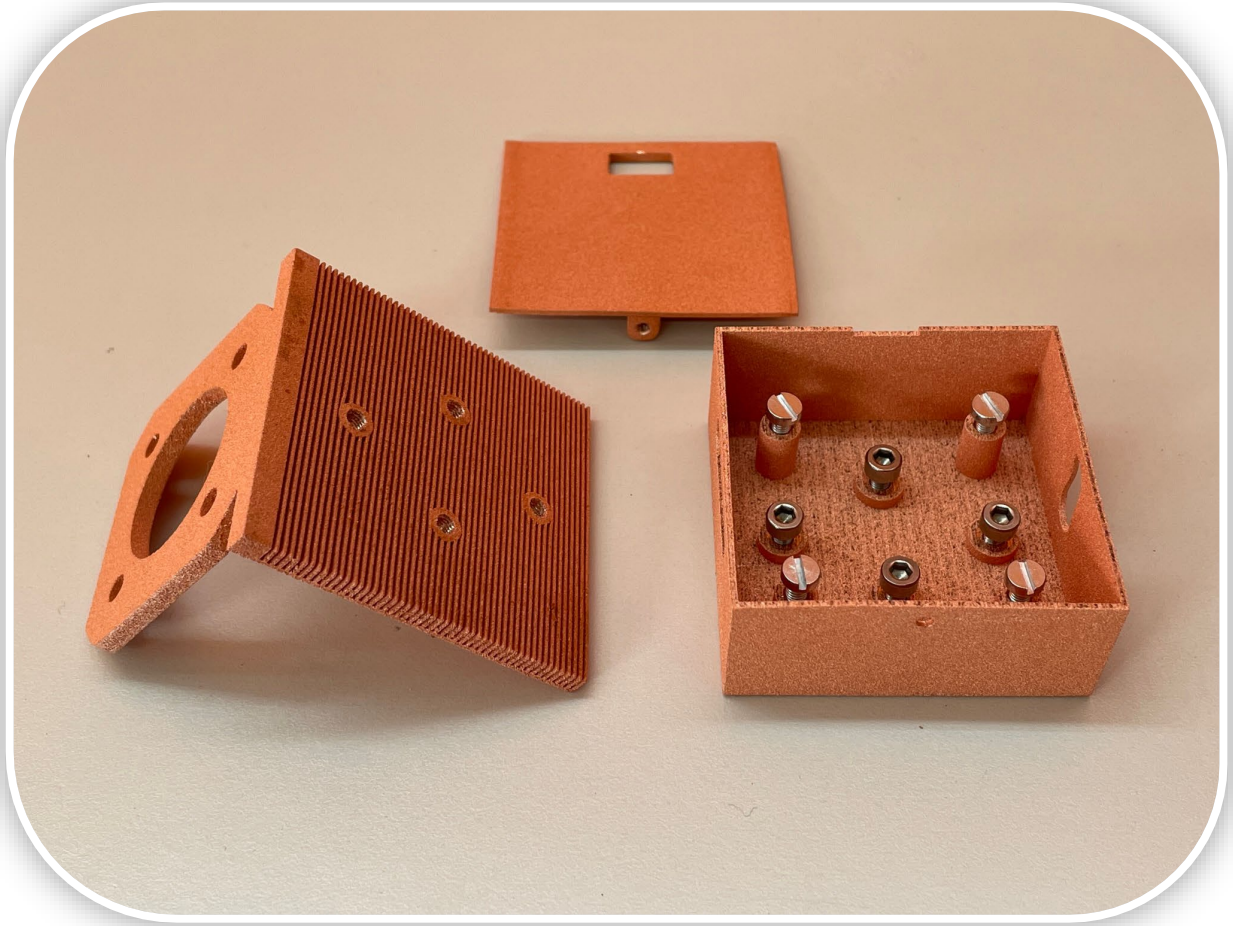


Target Holder for LUNA Experiment

Low weight frame for Bolometric light detector (CUPID-0 R&D)

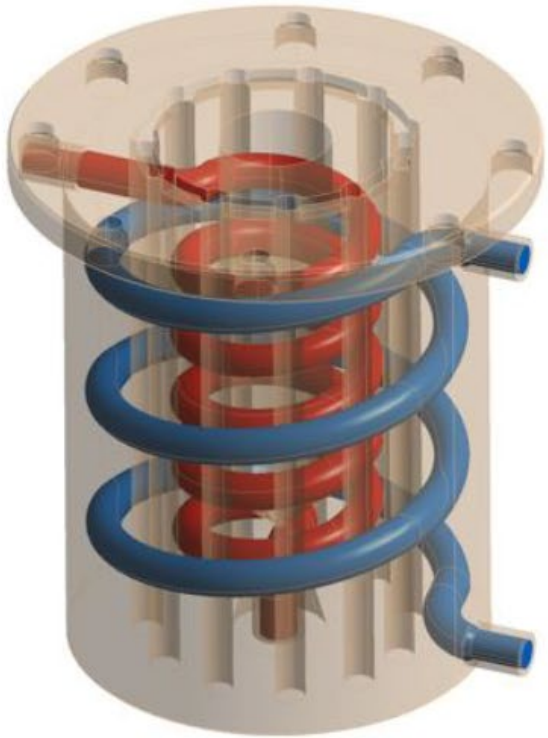
SLM Cu-printed objects @ HAMMER (LNGS)

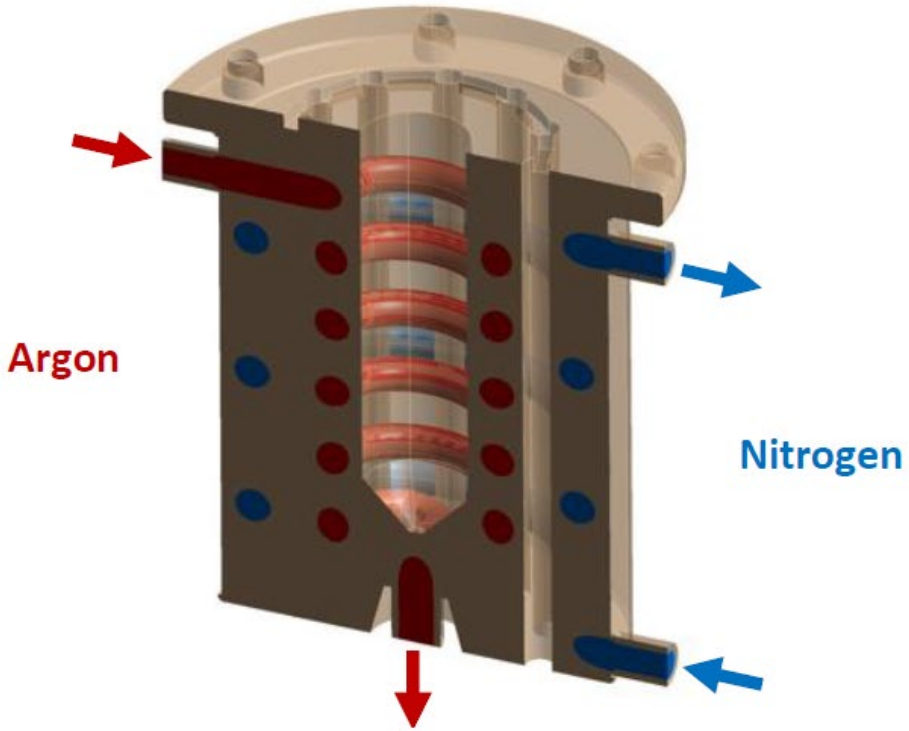


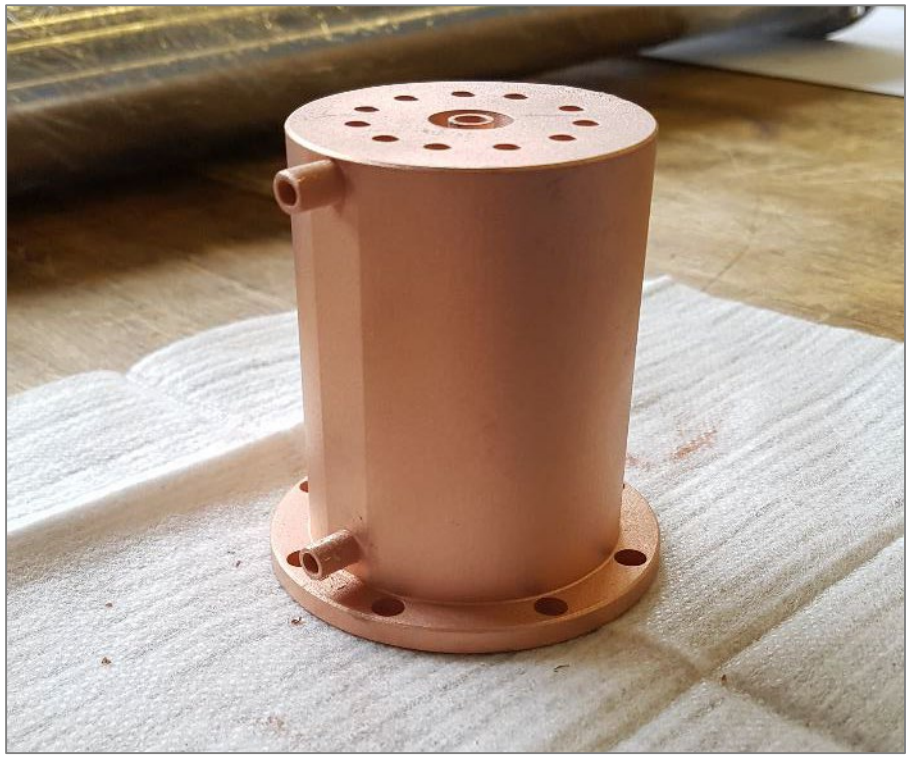


box High heat-exchange Electronic box for Ptolemy

SLM Cu-printed objects @ HAMMER (LNGS)







R&D Heat-exchanger for Dark Side

D. Cortis, A. Lalli, D. Orlandi, in *Design Tools and Methods in Industrial Engineering II. ADM 2021. (Springer)*
https://doi.org/10.1007/978-3-030-91234-5_51

Cu Powder

The «commercial» powder (≈5 μm particle size) is normally certified between 99.5% and 99.9% of purity. This is clearly not sufficient for our scope.
Moreover we performed a HPGe measurement on “standard” powder:

ICPMS	^{238}U	20±10 ppt
HPGe	^{226}Ra	3±1 mBq/kg

Produce the powder from Ultrapure Copper

CU Atomizer, will be installed within fall 2022 @ Hammer

Etching/Passivation activity @ LSM

Ali DASTGHEIBI FARD - LSM

- **Etching :**

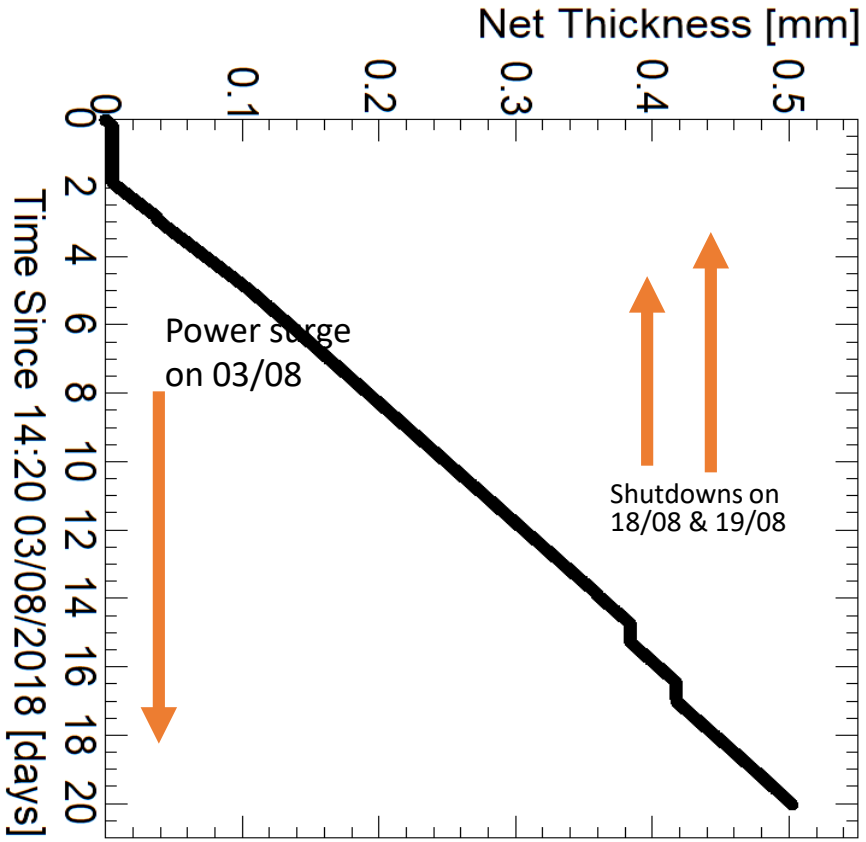
Using 1:3:96 mixture of H2SO4, H2O2, and >18 MΩ pure water

- **Passivation :**

Using 1% citric acid diluted with >18 MΩ pure water

Etching/passivation operation after 500µm copper plating (Electroforming copper method)

Operation for plating of 500 µm of cupper

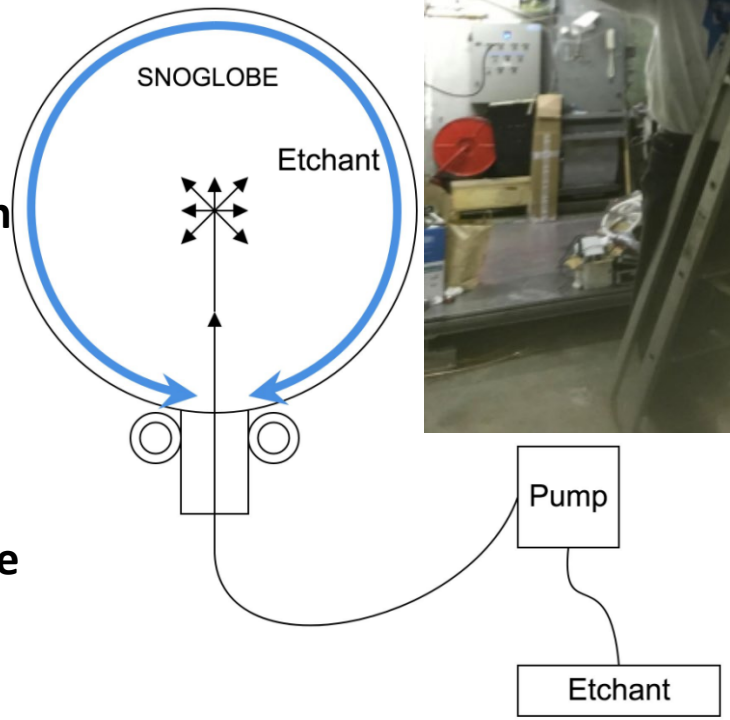


Surface Quality

Electroplating of copper (collaboration with PNNL)

Etching activity of inter surface of a sphere (Spherical detector of NEWS-G DM experiment)

- Cleaned by chemical etching using 3% H202, 1 % H2SO4 in water
- Samples of etchant collected during operation
- Spectrophotometer used to measure copper concentration of etchant
- ≈2 µm of copper was removed
- Etching took ~5.5 minutes
- After rinsing, surface was passivated using 1% citric acid solution
- Volume was flushed with nitrogen
- Sphere was sealed and vacuum pumped after operation to minimise further contamination
- Sphere has remained sealed since
- Either in vacuum, nitrogen overpressure or filled with operating gas



Summary of requests for WP5.6

Participant Number/Short Name		
	Cost (€)	**Justification**
Personnel	84 k€	1FTE/year x 1 year in LNGS
Personnel	168 k€	2 FTE/year x 2 year to be shared between LSC and LSM
Equipment	15k€ 60 k€	Instrumentation and equipment to increase EF-CU production (LSC) Alpha Counter + Readout (LSM)
Other goods, works and services	10k	Manufacturing of components (LSM,LSC)
Remaining purchase costs	20k	Consumables (LSC, LSM,LNGS)
Total	357k	

Summary of deliverables for WP5.6

1. **Design** of 3D copper structures to hold rare event detectors
2. **Studies** and selection of surface cleaning protocols in copper electroformed pieces suitable for future common applications across network laboratories
3. **Design** and **construction** of enlarged electrolytic bath to increase the production capacity and to obtain different geometries in the copper electroforming facility
4. **Production** of few kg of ultrapure 3D powder
5. **Measurement** of surface radioactivity of large copper made with EF and 3D printers
6. **Testing** of detectors assembled with ultrapure copper.