Cleaning procedure of 3D printed warm nose heat exchanger and other applications

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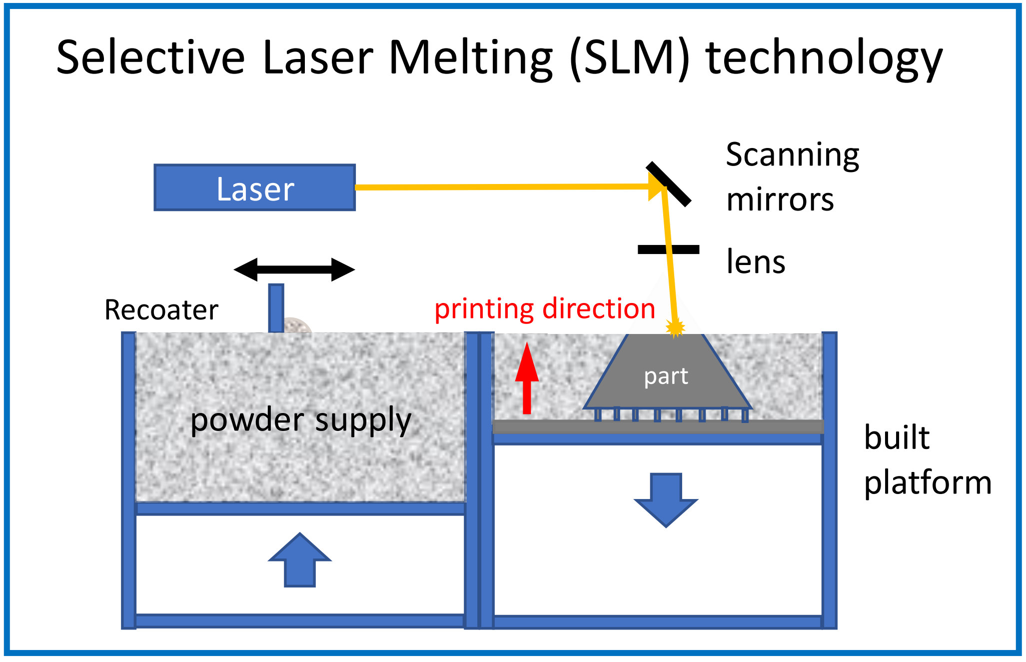
Metal Additive Manufacturing

Metal Additive Manufacturing (AM) is an incredibly interesting solution for high energy physics due to its **design flexibility** and its **light weighting** opportunities and most importantly the **ability to fabricate special components in single occurrences**.

As this technology is spreading in physics experiments, several new details have to be investigated and defined.

Metal Additive Manufacturing

Many special components produced for high energy physics are now made with Selective Laser Melting (SLM) tecnology, also named Laser Powder Bed Fusion (LPBF).



Working principle: the recoater spreads a metallic powder bed on the built platform, the laser selectively melts the powder and part is build layer by layer from bottom to top.

Metal Additive Manufacturing

SLM powder diameter = 15 ÷ 63 um

minor changes might be due to chosen batch (15 ÷ 45 um or 20 ÷ 63 um)

Typical AISI 316L powder data sheet

CERTIFICATE 22041

Physical Test Data

	Minimum	Actual	Maximum
Hall Flow, s/50g		18.0	

Particle Size Data

Sieve Analysis

+45µm	0.6%
-45µm +15µm	97.9%
-15µm	1.5%

Laser Diffraction Analysis

	Minimum	Actual	Maximum
d10 µm	18.0	20.7	23.0
d50µm		32.2	
d90µm		50.1	
	-15.0 µm = 0.6%		

Chemical Analysis (wt %)

El	Minimum	Actual	Maximum
Cr	16.5	**16.7%**	17.5
Ni	12.0	**12.5%**	13.0
Mo	2.0	**2.3%**	2.6
Si	0.3	**0.6%**	0.9
Mn	0.2	**0.6%**	0.8
N		**0.095%**	
O		**0.043%**	
Cu		**0.010%**	
C	0.00	**0.02%**	0.03
P	0.00	**0.01%**	0.04
S	0.000	**0.008%**	0.030
Fe		**Balance**	

Particle Size Distribution measured using Malvern 2000 instrument

Possible new source of pollution → Several cleaning options are available

Cleaning procedure and cleanliness acceptance criteria are one of "new topics".

AM Cleaning process

How clean we should be?

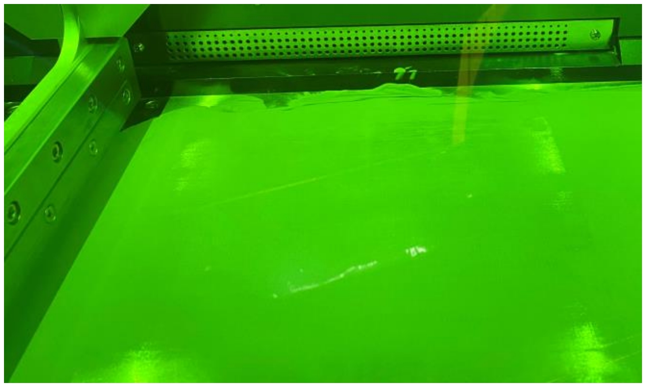
To decide if we can install metal AM component…
cleanliness level and **acceptance criteria** must be **defined**,
depending on location where parts are installed

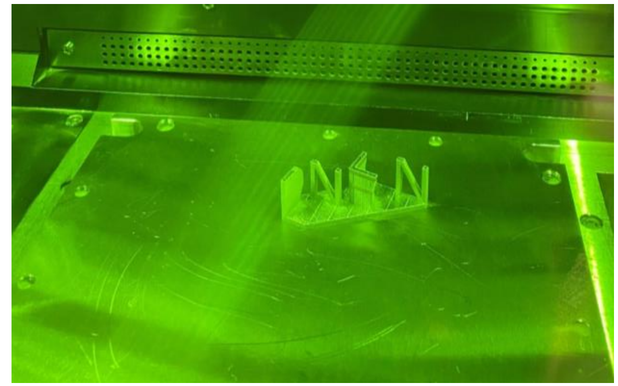
…..to be discussed later in the presentation….

AM Cleaning process

After production **all AM components** are subjected to a **basic cleaning procedure:**

Gross depowdering (printing chamber glove):





This cleaning can be followed by other cleaning steps, before or after, detaching the part with wire-cut EDM.

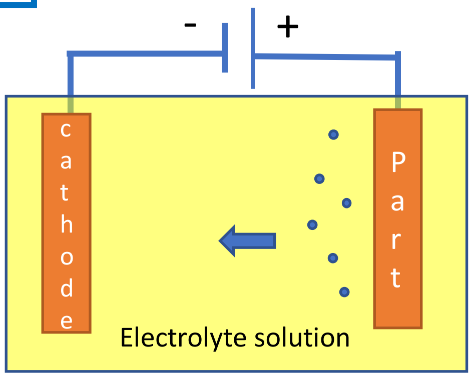
Possible steps:

- flush in a control enviroment
- depowdering unit
- …

Depowdering unit

AM Cleaning processes

Some of the cleaning processes that can be applied to AM



Electrolytic polishing

Workpiece immersed in electrolyte solution and connected to positive terminal of DC power supply acts as an anode. Metal surface is polished by oxidation and then dissolution.

Electrochemical degreasing

Immersion in an electrochemical bath filled with acidic electrolytic solution.

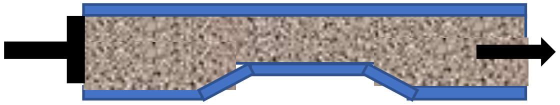
Tumbling

Tumbling media and AM parts are placed in a vibratory tumbler and surfaces are therefore finished through friction

https://www.postprocess.com/overcoming-3d-printing-surface-finishing-challenges/

Abrasive flow machining

Parts are internally cleaned by mean of an dense abrasive paste with is forced in the component



Chemical cleaning

Cleaning is done with an aqueous solution with chemicals or surfactant, then the part needs to be cleaned with ethanol to absorb the residual humidity. The use of chemicals could be a concern for some materials and applications. Cleaning might be done also with organic cleaning (no ethanol step needed).

Flow cleaning

Air and/or high volatility fluid with different velocities are flowed inside the components.

AM Cleaning processes

Parameters influence on AM cleaning:

DESIGN influences

- effectiveness of the basic cleaning procedure
- cleaning procedure appliability

APPLICATION AND FINAL USE influences cleaning procedure appliability

AM design rules related to cleaning:

- avoid blind areas (= powder difficult to remove)
- avoid undercuts (= not perfectly sinterized powder)

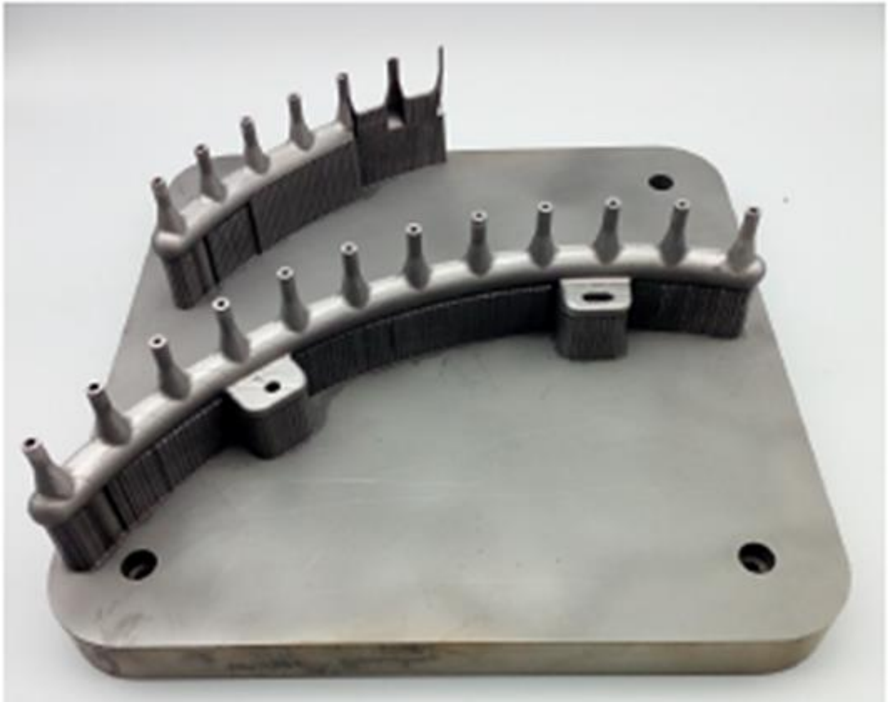
AM Cleaning process

Application and ref. in backup slides

...It is not always possible to follow AM design rules

CASE STUDY:

3D printed manifold for the Outer Pixel Endcap cooling system



More details available here:
https://indico.cern.ch/event/1268355/contributions/5354878/attachments/2629949/4548910/2023-04-17_ITK%20-COOLING-3D-PRINTED-MANIFOLD-INFN-MILANO.pdf

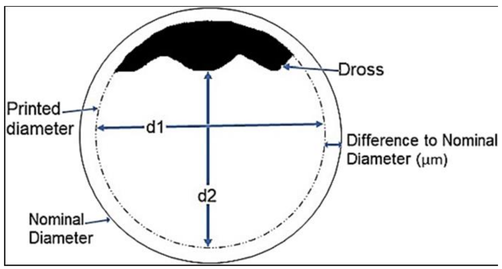
ref. S. Coelli (INFN-Mi) and E. Vaglio (LAMA)

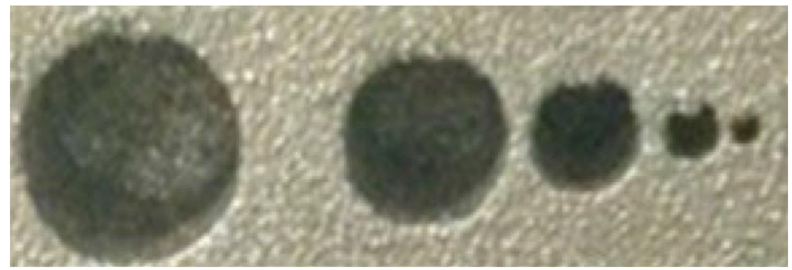
AM Cleaning process

"Low angles might produce not perfectly sintered regions".

<u>Difficulty</u>:

Horizontal pipe → formation of **dross in overhanging region** (D = 10 mm, 5 mm)





Possible **<u>solutions</u>**:

- Design modification (tear drop shape) → not possible 
- Additional cleaning process.

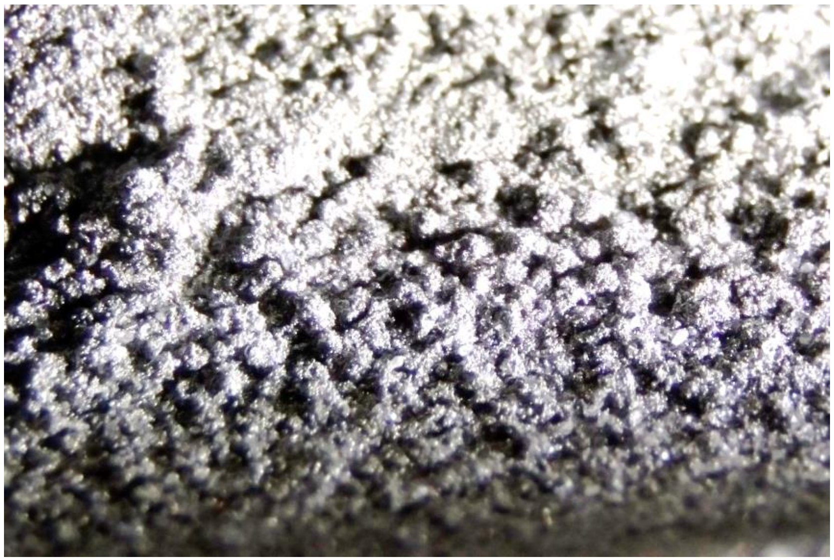
Basic cleaning procedure:

- Flushed with water
- Ultrasonic bath with DI water

Additional cleaning: **Hirtisation®** (chemical - electrochemical cleaning) with company Rena-Fintek (H3000).

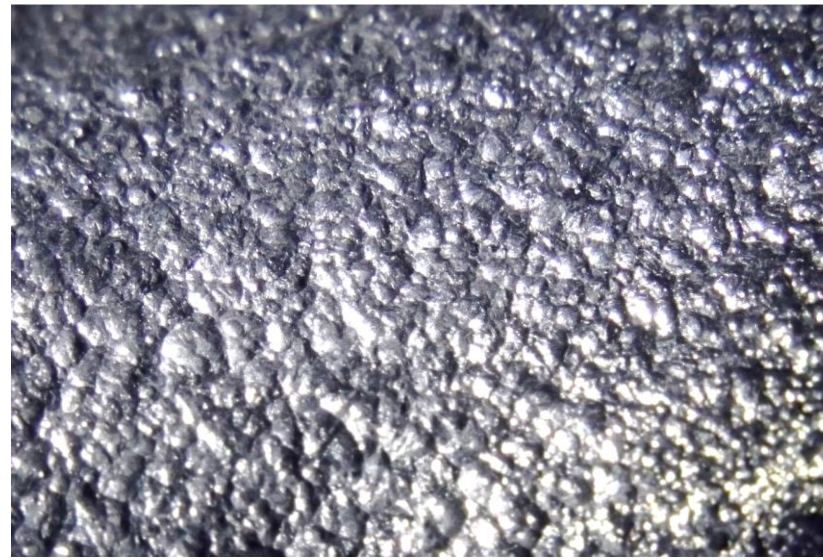
AM Cleaning process

Results



PRE-HIRTISATION

Some criticism were raised on the possibility that some dross fragment could detach due to the coolant flux.



POST-HIRTISATION

Improvement of the surface quality → no visible particle at risk of detachment.

Prepared by Daniele Viganò – INFN MI

AM Cleaning process

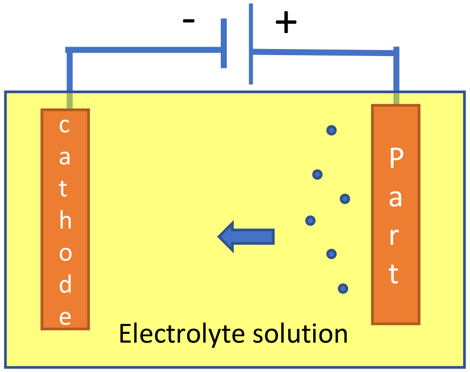
CASE STUDY:
Warm Nose Heat Exchangers

Difficulty: Complex geometry to clean:
- small diameters (1 mm)
- thin walls (1 mm)
- not possible to access the internal part

Basic cleaning procedure adopted:
- Vibration **before** detaching from platform (holes on the platform aligned with the WN HX pipes)
- Flush with air and high pressure DI water (3 – 4 bar)
- Ultrasonic bath (300 W, 26 kHz, 70°C, 30 min)
- Flushed and dried with compressed air

Additional cleaning?

AM Cleaning process



Electrolytic polishing

Not possible to place correctly electrodes and get uniform cleaning.

Electrochemical degreasing

Not possible with WN HX (same reason as above)

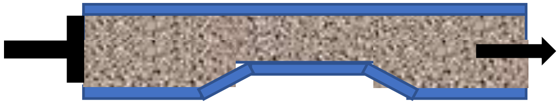
Tumbling

Thin wall and edges might be damaged during the cleaning process (impact against the tank)

https://www.postprocess.com/overcoming-3d-printing-surface-finishing-challenges/

Abrasive flow machining

Dense abrasive paste → Small pipes → very high pressure that can damage the part.



Chemical cleaning

The use of chemicals could be a concern. This solution is kept as back up solution. Preliminary trials were done on material samples.

Flow cleaning

Could be a solution. Air and/or high volatility fluid with different velocities.

AM Cleaning process

Different studies were done to improve the WN HX cleaning:

- Design improvement
- Basic Cleaning procedure improvement

(actual standard procedure @firm is already the timproved).

First tests done on scaled **un-optimized WN HX** to have an idea of what was the starting point.

Sample	Inlet 1	Inlet 2	Outlet
ECD [um]	5.5	4.0	17.6
O [wt. %]	46.6	53.2	7.6
F [wt. %]	13.2	6.8	0.0
Si [wt. %]	20.6	12.3	0.7
S [wt. %]	3.5	12.4	0.0
Cr [wt. %]	1.1	0.5	18.3
Fe [wt. %]	5.5	2.7	62.0
Ni [wt. %]	0.8	0.4	10.4

Particles subjected to Energy-Dispersive X-Ray Spectroscopy (EDS) **Automated Particle Analysis** (APA) in order to evaluate their **size distribution** and determine their **chemistry**

EDMS 2583870

Particle size:

Filter inlet → ~ 5 µm

Filter outlet → 20- 25 µm

AM Cleaning process

Additional Dust Tests on **Scaled WN HX optimized for cleaning**:

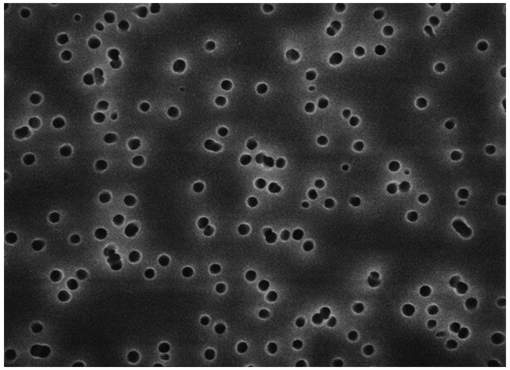
Wash test done after standard cleaning procedure (EDMS [2783986](2783986))

Wash test → performed @Cern chemical lab (Benoit Teissandier).

Procedure:

1. Fill both circuits with Ethanol
2. Close the circuits
3. Place scaled WN HX optimized for cleaning in the ultrasonic bath
   - 30 min
   - Max power
4. Filter the ethanol samples

Filtering done with 3um isopore membrane.



Polycarbonate membrane for microscopy and visual analysis

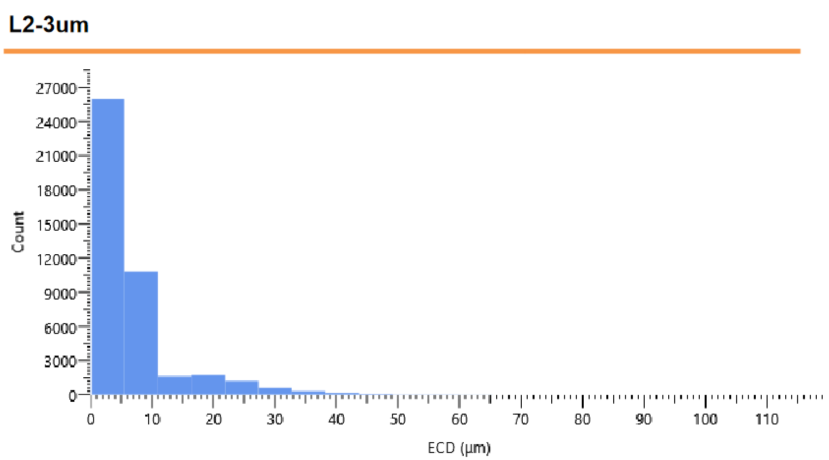
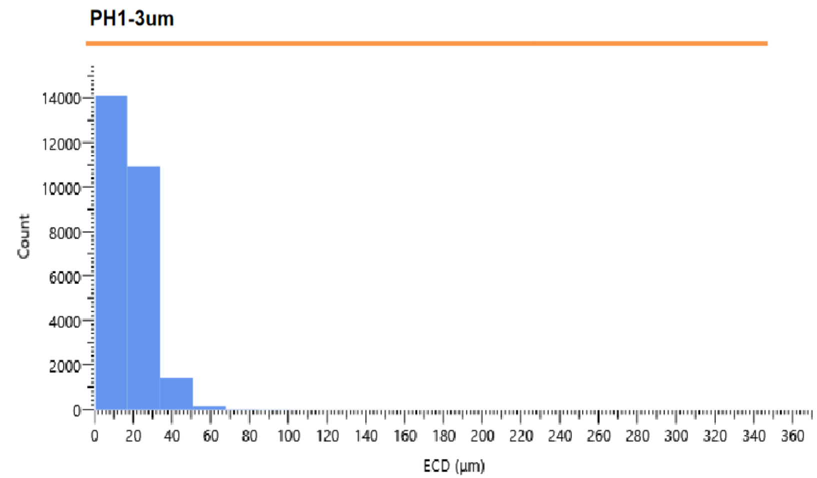
APA analysis (Stephan Pfeiffer) - EDMS 2783986

https://www.merckmillipore.com/CH/en/product/Isopore-Membrane-Filters,MM_NF-C153#documentation

Results:

AM Cleaning process

EDMS [2783986]

Liquid side	2-phase side
	
Area-weighted Mean = 81.44 um^2 → **D=10.18 um** (min 1.56 um^2)	Area-weighted Mean = 340.73 um^2 → **D=20.82 um** (min 1.56 um^2)
Total number of particles: 42790 Particles over 10um ECD: 6552 Particles under 10um ECD: 36238 Fraction of particles over 10um: 15.3% Fraction of particles under 10um: 84.7%	Total number of particles: 26984 Particles over 10um ECD: 19491 Particles under 10um ECD: 7493 Fraction of particles over 10um: 27.8% Fraction of particles under 10um: 72.2%

AM Cleaning process

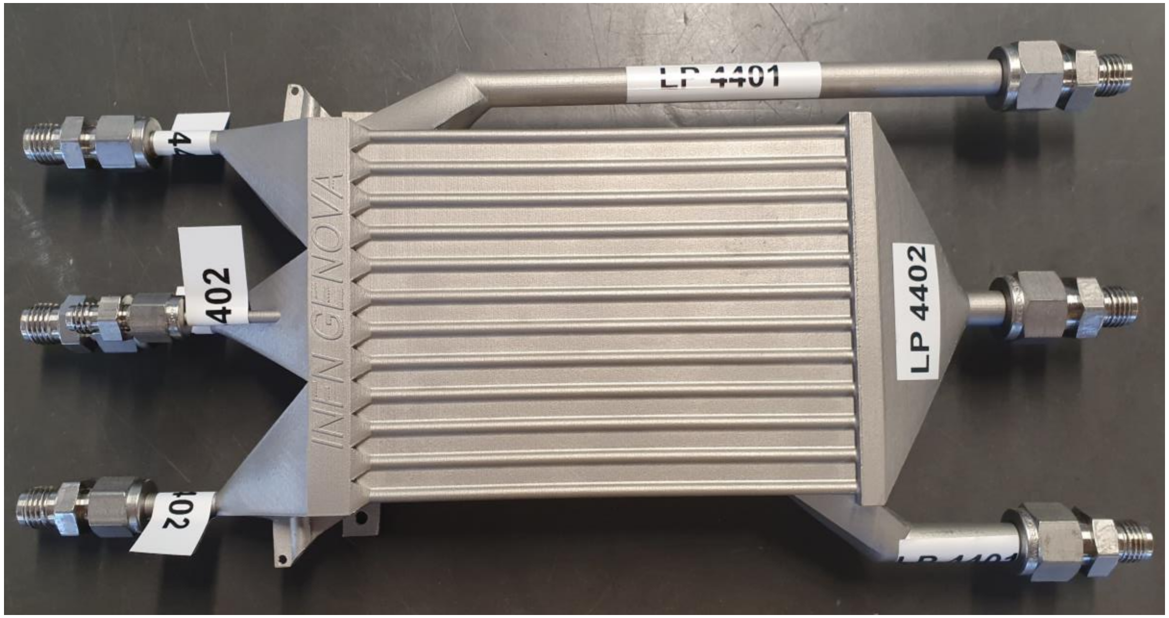
Comments:

- **Preliminary CT scan** also done to verify where powder is localized (after firm cleaning). The tomography **didn't identify any metallic powder stockpile.**
- Some of the particles on 2 phase circuit are bigger. The particles on liquid side are clearly only due to small powder particles residue (spheric particles). Might be further investigated.
- The **main fraction of particles** is below 10 um
- **Requested cleaning level** was **still not defined**

AM Cleaning process

DUST TESTS:

As welding the WN HX for FDR setup was urgent, we decided to proceed with the cleaning of the full scale **WN HX_1022** in order to verify if there is a trend and accept the final cleanliness level for now.



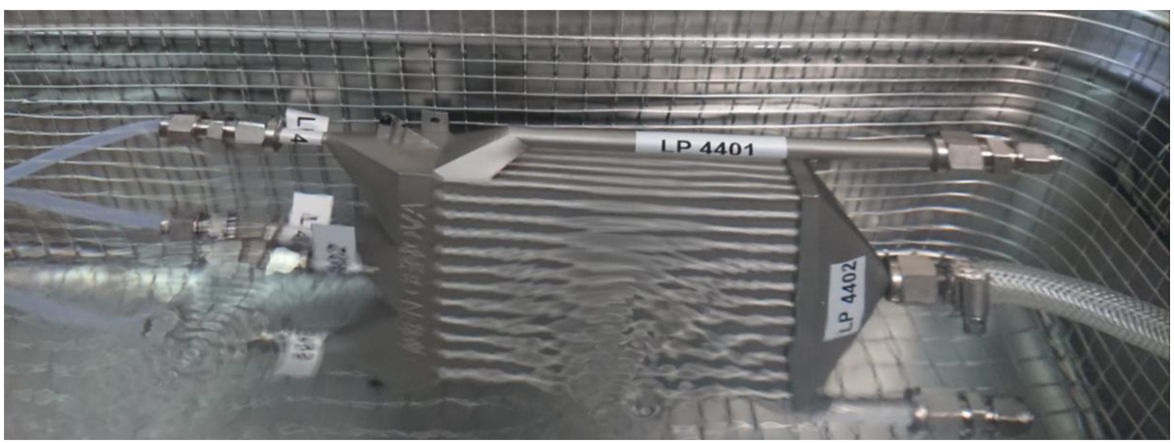
AM Cleaning process

Eth. = WN HX filled with ethanol
W = WN HX flowed with distilled water at 7 bar in 3 different position for 15 min in total
U. B. = ultrasonic bath at max power for 30 min

<u>Cleaning of **WN HX_1022**</u>:

- 3D printing
- Enhanced cleaning procedure @production firm
  - STEP 1 = Filtering after Eth + U.B.
  - STEP 2 = Filtering after W + U.B. & Eth + U.B.
  - STEP 3 = Filtering after W + U.B. & Eth + U.B.
  - STEP 4 = Filtering after W + U.B. & Eth + U.B.
  - STEP 5 = Filtering after only filled with Ethanol and shaked by hand

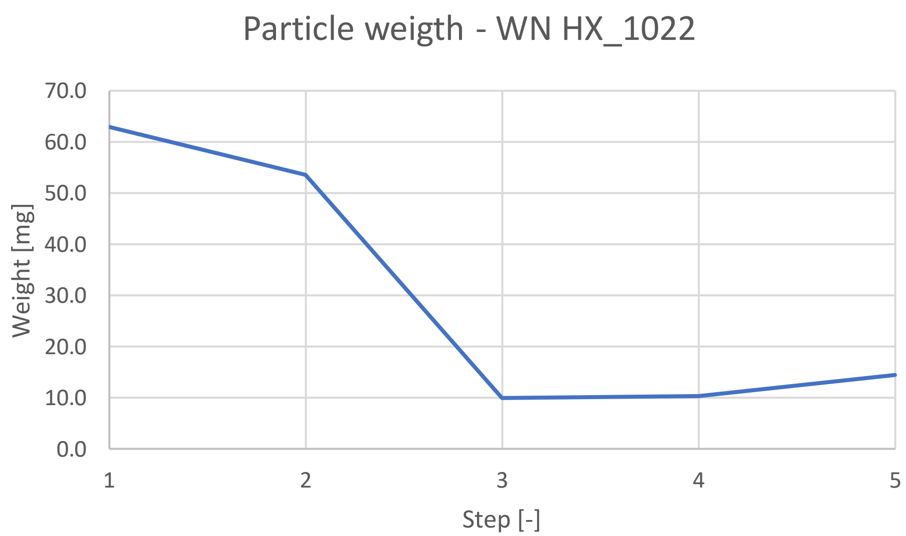
To be noted: ethanol can be used as a solution only for low volumes for security reasons.



AM Cleaning process

Results on WN HX_1022:

- STEP 1 = Filtering after Eth + U.B.
- STEP 2 = Filtering after W + U.B. & Eth + U.B.
- STEP 3 = Filtering after W + U.B. & Eth + U.B.
- STEP 4 = Filtering after W + U.B. & Eth + U.B.
- STEP 5 = Filtering after only filled with Ethanol and shaked by hand



More details:
https://indico.cern.ch/event/1195438/contributions/5024646/attachments/2556760/4406794/20221130_BabyDemoMeeting_Cleaning.pdf

Filter after STEP 5 is now on SEM analysis:

- Particles dimension in different areas of the filter will be checked with optical microscope
- Where the sample with carbom stick is taken will be noted

Estimation of particles collected in <u>real working condition</u> (CO_2, - 40°C, …) will be done by placing a filter before and after the WN HX during cold test and **comparing filter weight**.

AM Cleaning process

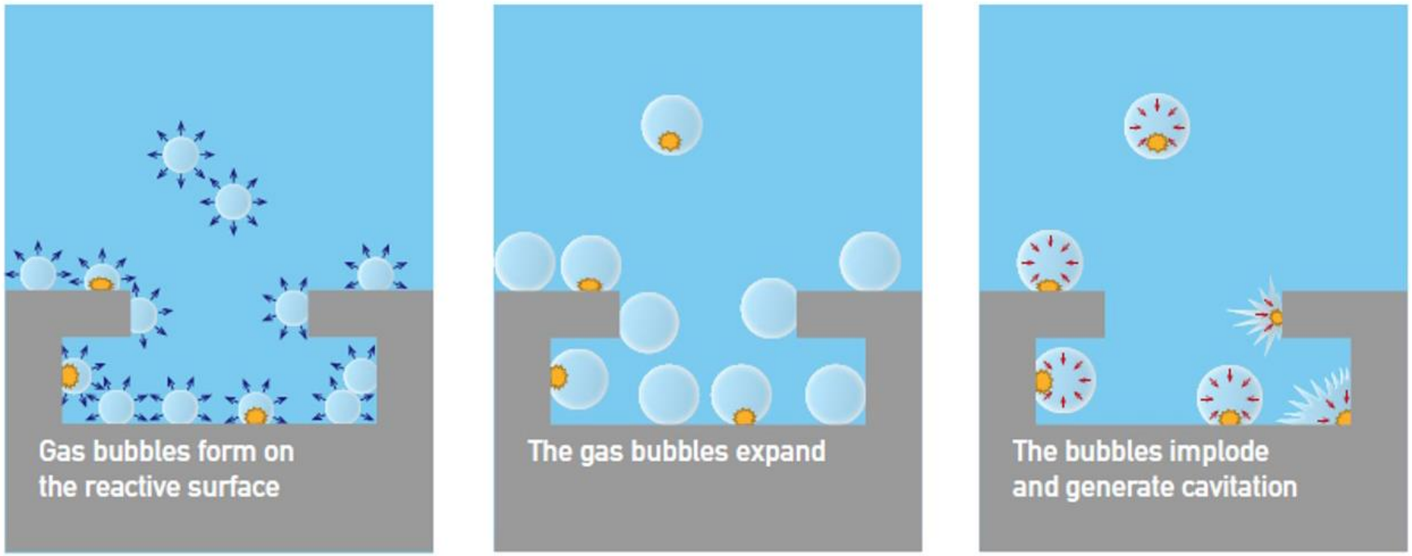
- Study done to understand the cleaning effect on a complex geometry

- To improve our knowledge other cleaning solution are now under evaluation with CERN chemical lab and with the production firm

Working group = B. Teissandier, C. Rossi, R. Gerard, L. Ferreira with Borer company

  - CNP (Cyclic Nucleation Process)

- Interesting cleaning solution both for AM and chemicals removal



Due to the isostatic distribution of pressure change in the cleaning fluid, Cyclic Nucleation is highly effective in cleaning difficult geometries like undercuts and narrow channels.

https://www.lpw-reinigungssysteme.de/besondere-reinigungsverfahren-zyklische-nukleation-cnp_en/

AM Cleaning process

- AIM: Compare effectiveness of different cleaning techniques for AM and other applications:
    - Chemistry procedure: Ultrasonic bath of sample closed and filled with Ethanol.
    - Cyclic Nucleation
    - «standard» cleaning procedure: Ultrasonic and detergent?

- Sample elements:
    - Small blind capillaries of different dimensions
    - Small open and waved capillaries of different dimensions
    - Lattice «sintered» structure

- All geometries will be 3D printed.

- Samples will not be treated thermally to avoid particles crumbling

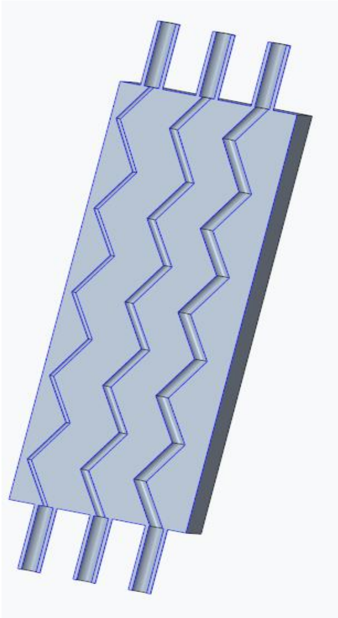
- Sample thickness = 10 mm. Can be cut with EDM wire-cut if needed for inspection

- All parts are provided of 20 mm pipes to fill the sample and close it if needed

(e.g. if we want to fill it with Ethanol)

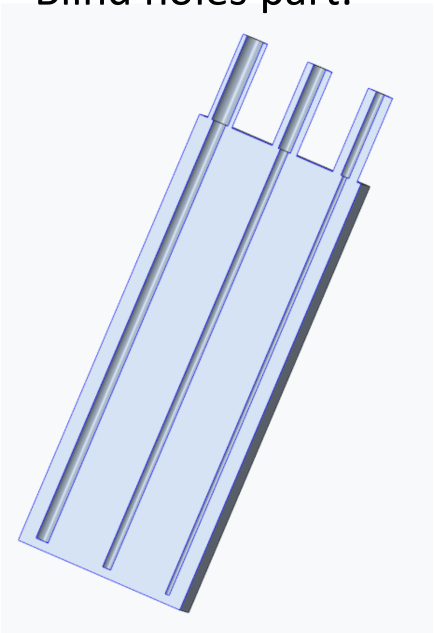
AM Cleaning process

Capillaries part:



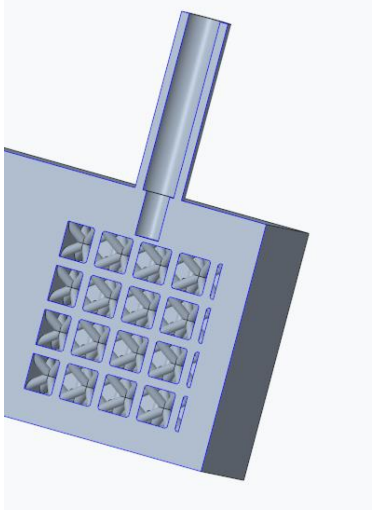
Winding capillaries of 1 mm. 2 mm and 3 mm, L ≈ 100 mm

Blind holes part:



D = 1 mm. 2 mm and 3 mm, L ≈ 75 mm, blind holes

Sintered part:



To simulate a sintered structure

Parts will be 3D printed at the same company (Lara) to have the same machine, parameters, material, etc and might be also printed at CERN

AM Cleaning process

We saw different techniques and good results…

The main question still remain…..How clean we should be?

To decide if we can install a AM component…

cleanliness level and **acceptance criteria** must be **defined**, **depending on location where parts are installed**

Strategy and rules must be defined to proceed

Questions?

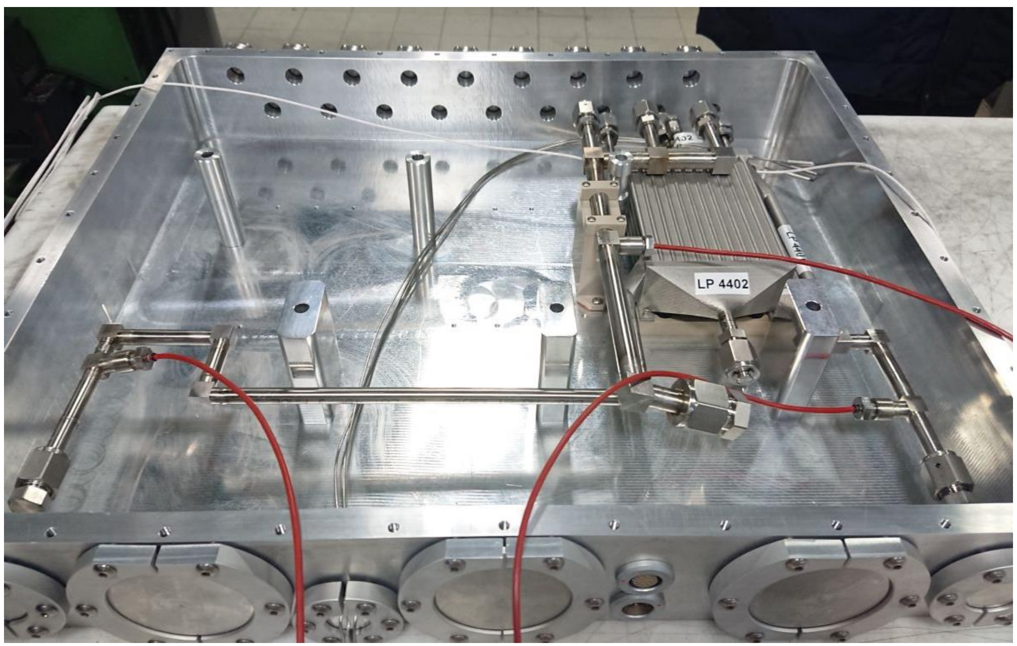
WN HX Cleaning

BACKUP SLIDES

Some of the ATLAS ITk work ongoing:

Strips and Pixel WN HX

(materials AISI 316L and Ti6Al4V Gr. 5)



EC Pixel manifold

(materials Ti6Al4V Grade 2 (pure Ti))

INFN GENOVA

LA.RA SOLUZIONI MECCANICHE AVANZATE

INFN MILANO

LAMA FVG

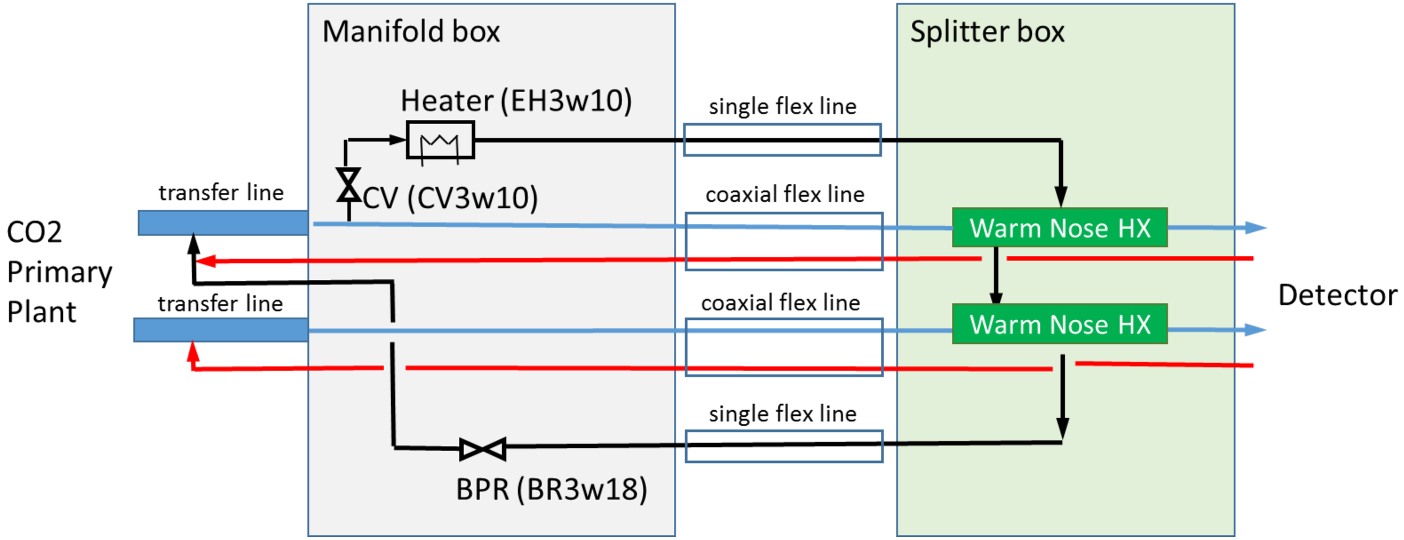
Additive manufacturing

EDMS 2307865
EDMS 2307866
EDMS 2722680

Warm Nose heat exchanger (WN HX) are foreseen in ATLAS ITk upgrade for Strips and Pixel systems.

Warm nose working principle:

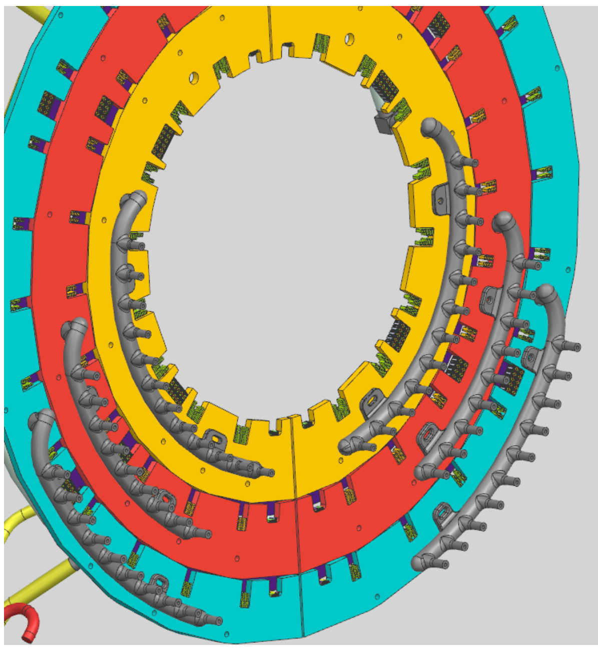
- avoid that instability, related to the Liquid Super Heating, occurs at the detector
- regulate detector temperature without technical intervention in its unreachable areas during runs.

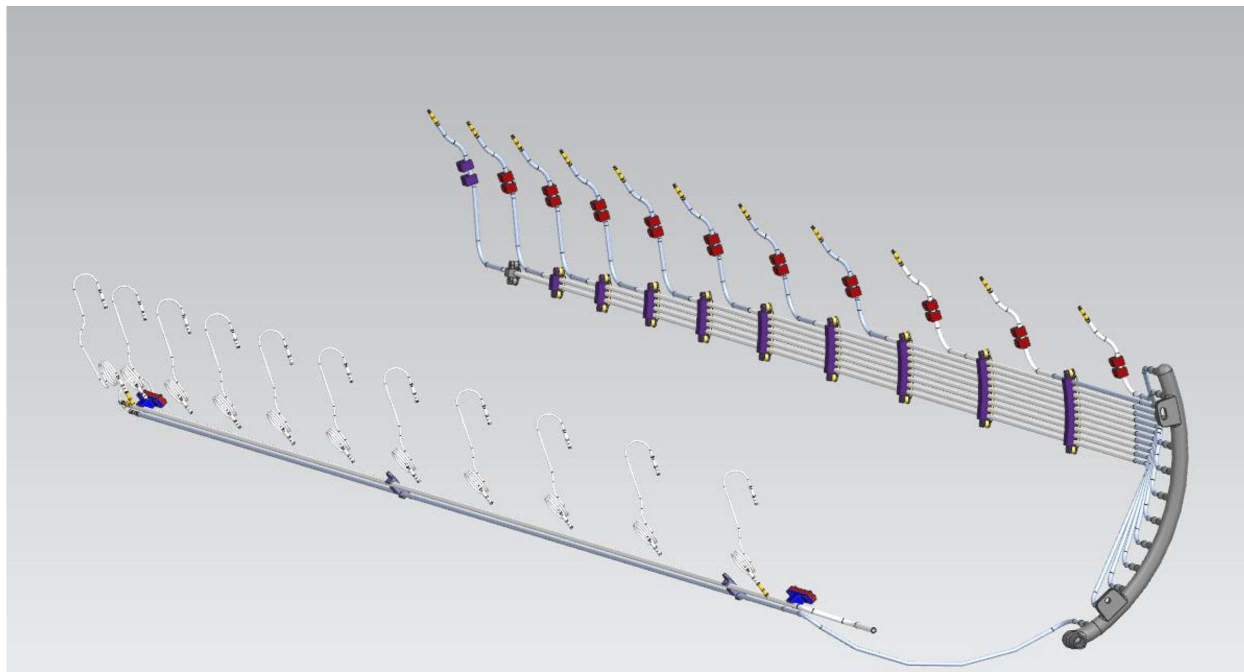


- Part of liquid CO_2 is drawn from the main stream and processed to reach the 2-phase status, i.e. WN loop
- Temperature in WN loop is controlled by a Back-Pressure Regulator (BR3w18).
- WN HX→ heat exchange between WN loop and subcooled liquid directed to the detector.
- Heater (EH3w10) to get WN loop in 2-phase and control the vapour quality of the loop
- Mass flow in the circuit is controlled by a carrel valve (CV3w10)

Additive manufacturing

ATLAS ITk Outer Pixel Endcap (EC) cooling system → design of the 12 Half-Shells manifolds that collects CO_2 exhaust flow from all the evaporative lines of the Half-Ring Pixel detectors.





Many details available here
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- The use of common water might produce corrosion problems due to chlorine presence. To be noted that the WN HX will come in contact anyway with common water (EDM cut)

- De-ionized water could neutralize chlorine. Both external and internal surface must come in contact with de-ionized water after common water

WN HX Cleaning

Discussion outcome and Next steps

- Sven printed for Bell project at Layerwise (now Materialize)
  - First trial was «dirty»
  - The firm then improved cleaning (trade secret on how)

  → *which procedure was used for the cleanliness checked? Cleaning check done by layerwise. Checked later with alcool and paper filer probably. Running over years now with no problems.*

- No details on the Layerwise cleaning procedure details, but it is a chemical cleaning (info from Romain)

  → *we have a backup solution*

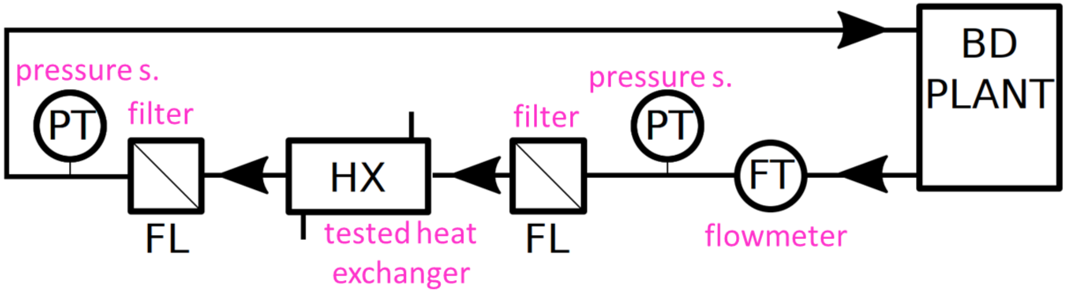
  → *I would keep it as a backup solution, but not the final one (unknown on details, chemicals,..)*

AM Cleaning process

DUST TEST

Analysis started in August/September 2020 on low mass WN HX

WASH TEST on Liquid and 2ph circuit:

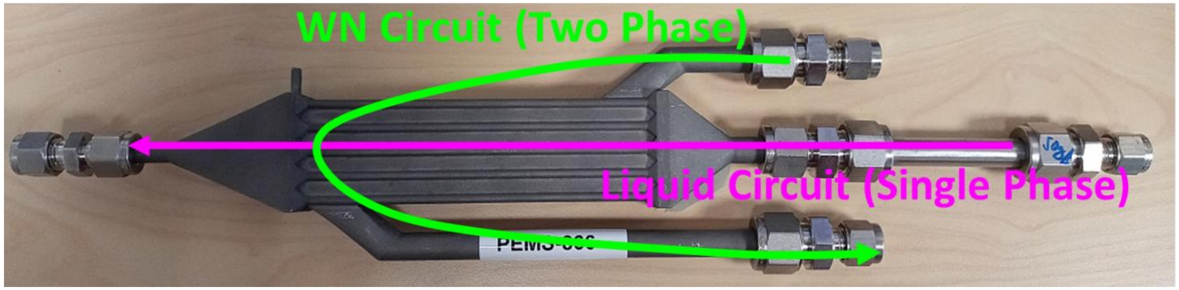


Circulation of liquid CO_2@-40 °C for 1 month on L and 2ph circuit. Sintered filters (0.5 um) were placed before and after the WN HX @Dust Test setup.

Thanks to Martin Doubek

Cleaning process

DUST TEST



After CO2 circulation the filter were flushed with Ethanol at room temperature (free circulating liquid through the piece).
The circulation lasted was around 1 minute and fluid recovered is approximately 250 ml from each pipe.

Fluid was then filtered and a chemical analysis were subjected to Energy-Dispersive X-Ray Spectroscopy (EDS) Automated Particle Analysis (APA) in order to evaluate their size distribution and determine their chemistry.

The results of the particles analysis of the filter outlet were compared with the one of the inlet on order the determine differences in particle size and composition and gain information about the filter quality.