Evaluation of the suitability of Pellet Additive Manufacturing technology for the design & manufacturing of complex ceramic parts

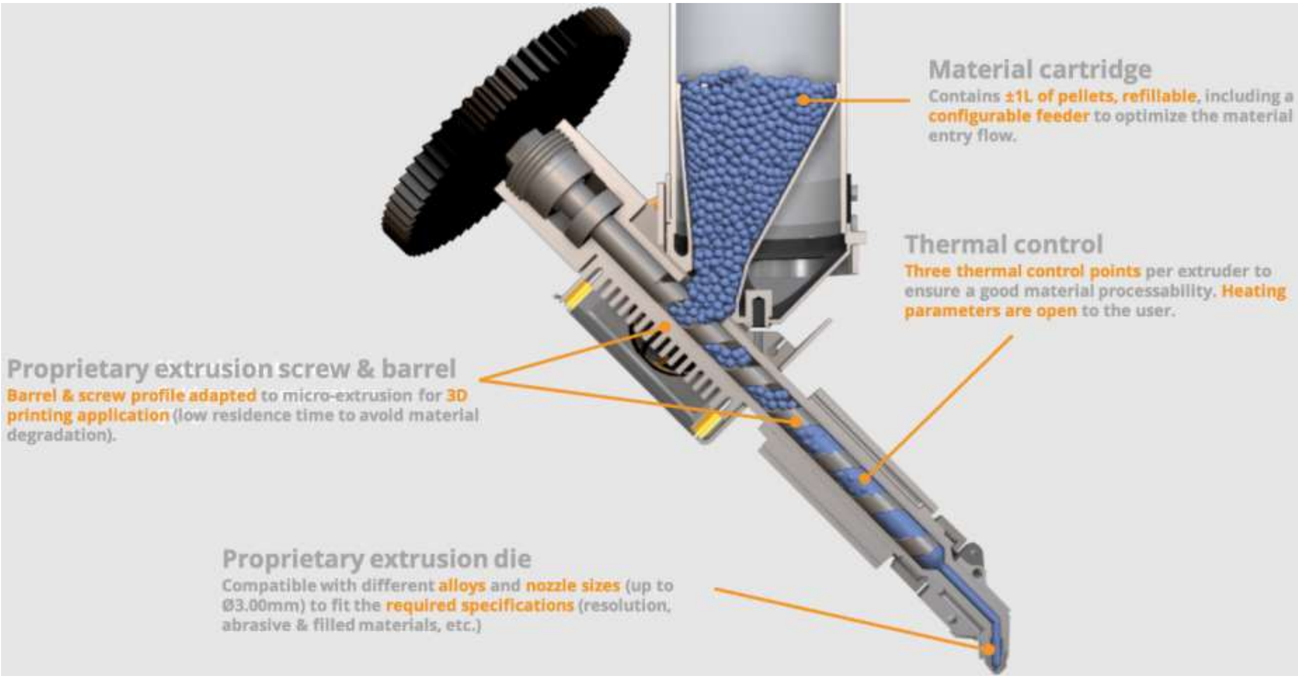
Delmotte C.[1], Martic G.[1], Juste E.[1], Spitaels L.[2], Ducobu F.[2], Riviere-Lorphevre E.[2], Bossu J.[3], Delaunois F.[3], Petit F.[1*]

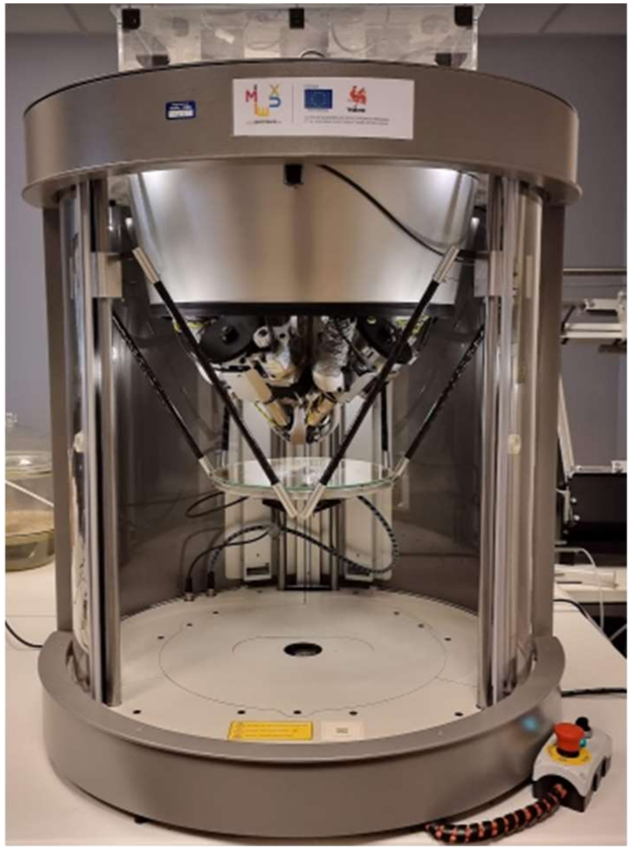
1. Belgian Ceramic Research Centre (BCRC), Mons, Belgium
2. University of Mons – Machine Design & Production Engineering, Mons, Belgium
3. University of Mons – Metallurgy Unit, Mons, Belgium

*Contacting author : f.petit@bcrc.be

What is the « Pellet Additive Manufacturing » technology ?

The process is inspired by the FDM approach: the filled thermoplastic material is melted in the extrusion head and conveyed to the printing nozzle by a heated endless screw.





Pollen AM- PAM series C
used in this work

Main challenges of AM extrusion

❑ How to achieve high density parts ?

- Obtaining defect-free parts (delamination, joints between beads, cracks, porosity) is a tricky business !

❑ How to improve the surface finish ?

Internal surfaces undercut or inaccessible for finishing

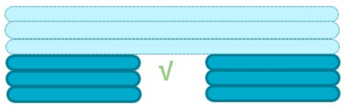
High roughness
(« staircase » effect)

- Disadvantage: surface finish << injection-molded parts
- Improved surface quality may require the use of finer nozzles = at the expense of printing time and increasing the risk of defects
- Difficult, if not impossible, to machine surfaces after the object has been completely manufactured (undercuts, internal channels, etc.) + prohibitive cost

Hybridization is a way to overcome these limitations

- Hydridization combines the advantages of additive manufacturing and machining, with the particular aim of improving the surface finish and overall quality of the parts produced.

Post milling – some areas are no longer accessible



Sequential milling/printing – almost all areas are accessible

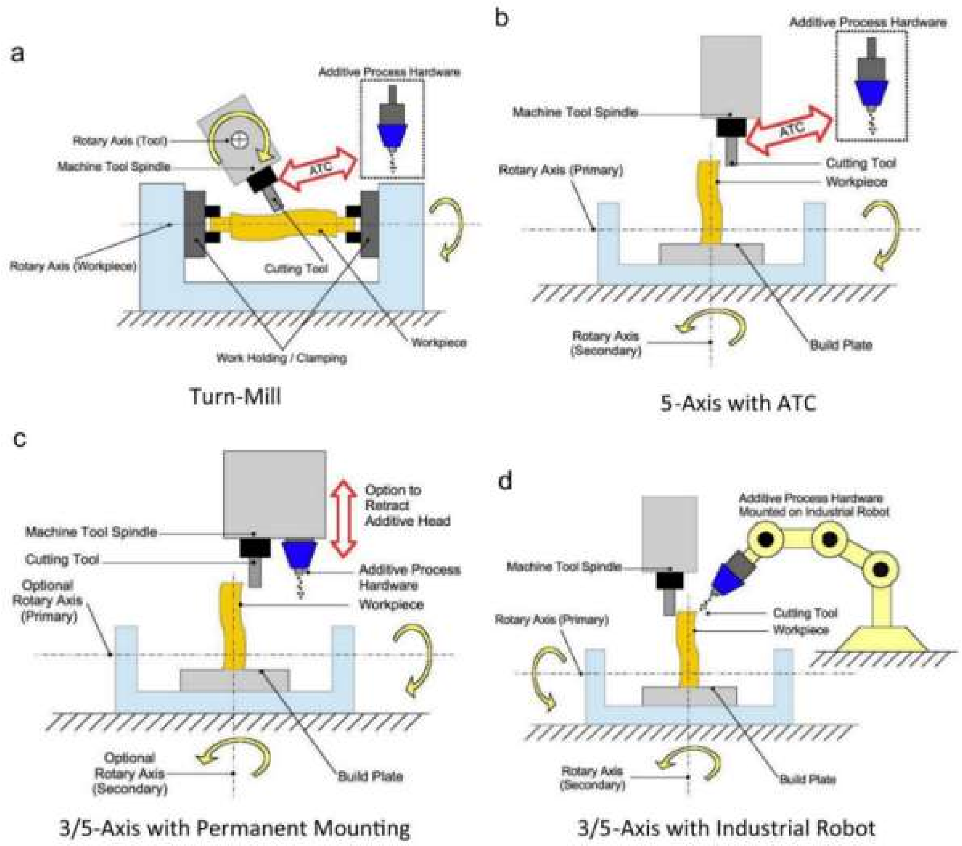
- Why hydridization with PAM/FDM technologies is relevant ?

✓ The printed material is made of easy-to-machine thermoplastic

✓ Extruded material solidifies rapidly

✓ Cheap compatible CNC equipment

✓ Machining residues can be easily recycled



Different concepts of hybridization according to [1]

[1] J. M. Flynn, A. Shokrani, S. T. Newman et V. Dhokia. « Hybrid additive and subtractive machine tools - Research and industrial developments ». In : International Journal of Machine Tools and Manufacture (2016), p. 79-101.

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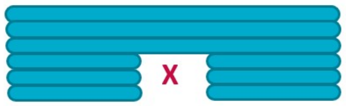

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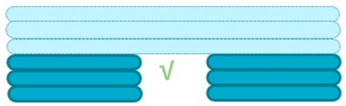

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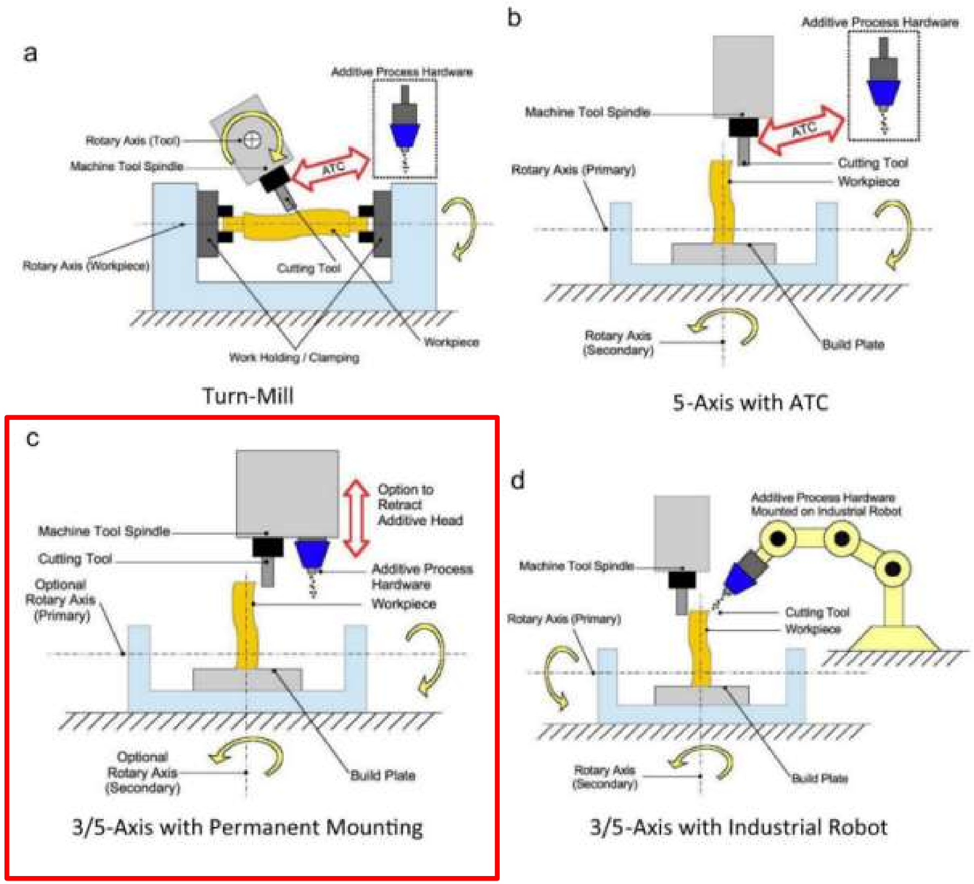

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Objectives of this work

1. **Evaluate the possibility of shaping ceramic parts with PAM technology**
   - Commercial ceramic grade selection (zirconia)
2. **Optimize processing conditions (printing, debinding, sintering)**
   - Defect-free parts (visual inspection, delaminations, warping)
3. **Assess the capability of PAM technology to produce parts with characteristics similar to those of CIM parts.**
   - Density, strength, hardness
4. **Demonstrate the benefits of additive/subtractive hybridization for thermoplastic extrusion technologies**
   - Integration of a cutting tool into the equipment, CNC/3D printing interfacing, post-processing, evaluation of test parts

Selected feedstock for the study

- Black Zirconia(INMAFLOW K2015 – INMATEC Technologies Gmbh)

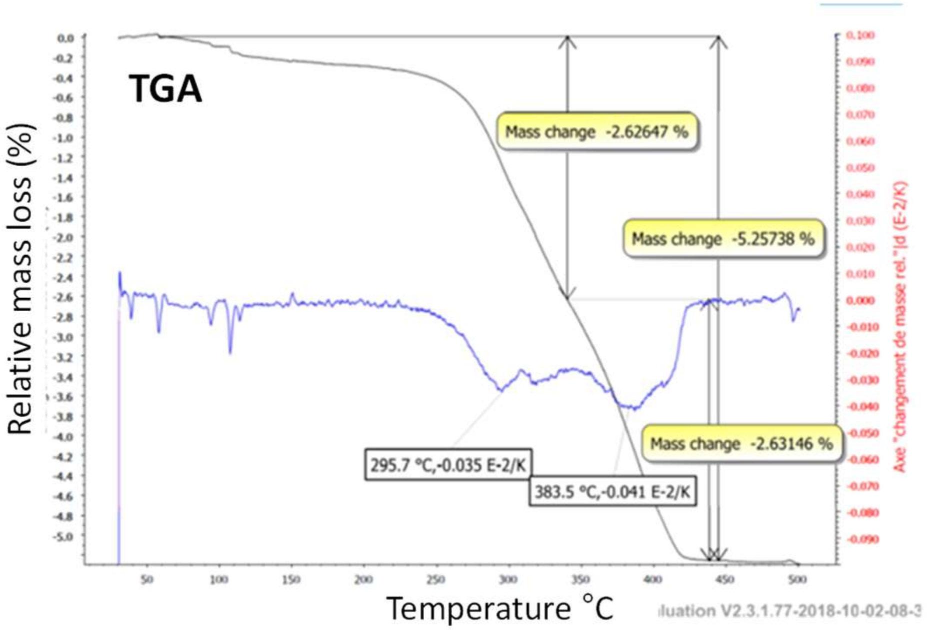
Expected good flow properties – suitable for AM-extrusion

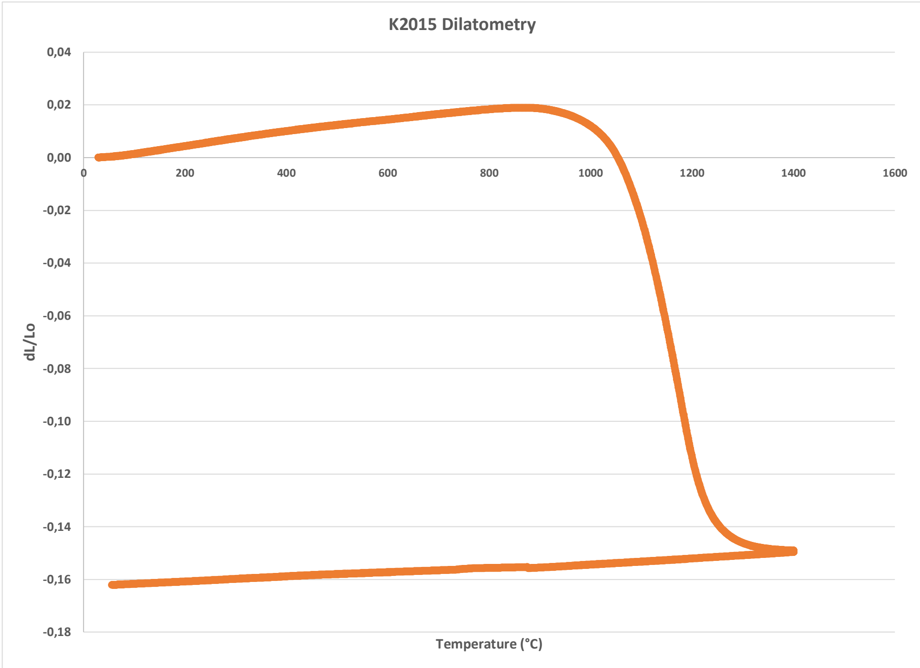
Feedstock	ZrO_2, 94.5%, Y_2O_3-partially stabilized, TZ-black (Tosoh Corp)
Sintered density	6,00 g/cm³
Shrinkage	17%
Binder	Polyamide based
Debinding	2 steps : - Aceton bath - Thermal debinding up to 325°C
Sintering	1400°C under air

Shaping processing (CIM)	
Mould temperature	15 – 30 °C
Heating zone temperature	110 °C
Nozzle temperature	150 °C

Supplier datasheet

Feedstock characterization - TGA, He pycnometry, dilatometry





Test conditions :

- On acetone debinded granules (38h)
- 5°C/min up to 600°C
- 1h dwell

❑ Mass loss

- After acetone debinding : **10%**
- After thermal debinding : **15%**

❑ He picnometry of debinded granules : **5,94 g/cm³**

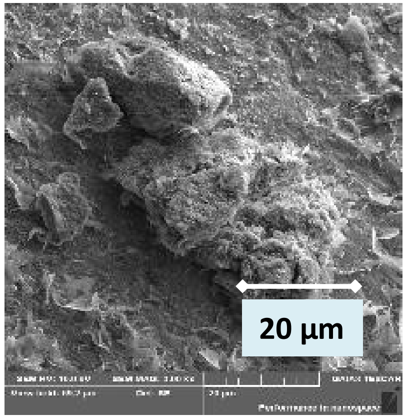
❑ Sintered density approx. 6

Test conditions :

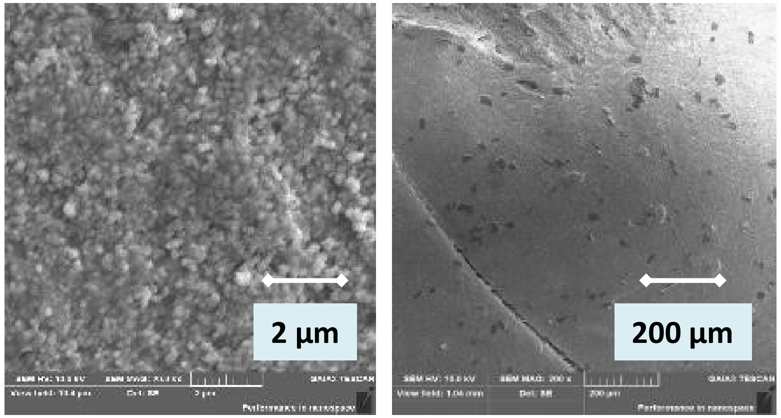
- 30°C -> 1400°C (2°C/min)
- 1400°C / 1h
- 1400°C -> 30°C (3°C/min)

Feedstock characterization – pellet assessment

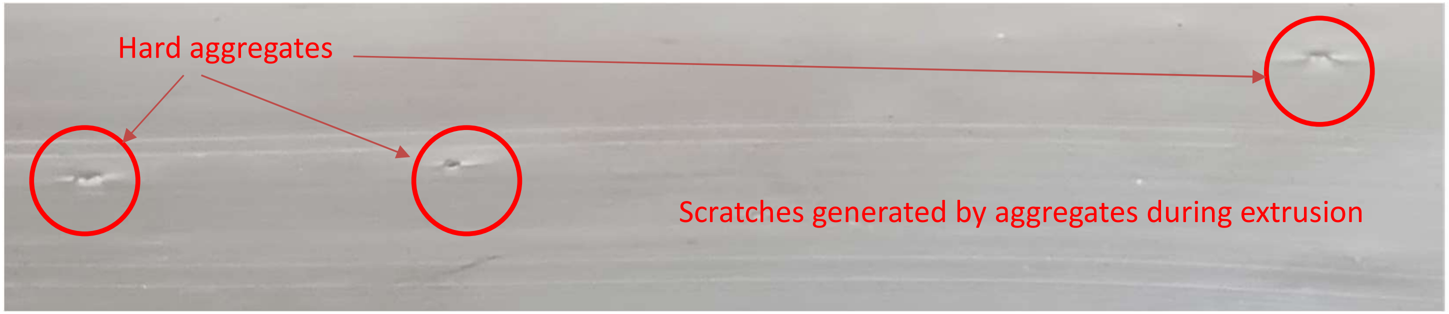
Raw granule



Debinded granules



Some hard aggregates are occasionally detected in the raw granules (extrusion of a 0.3 mm thin sheet)
(Harmless for CIM, may be problematic for AM extrusion : fine nozzle clogging ?)



Printing optimization

Huge parameter space:

- Extrusion settings
  - Temperature control: screw, nozzle, building plate, fan cooling.
  - Adjustment of printing speed/material flow
  - Nozzle diameter (0.4, 0.6, 0.8, 1.0, 1.2, 1.8 mm)
- Layer filling
  - Lines, gyroïds, concentric…
  - Interlayer spacing, interfilament spacing

Trade off required !

Preliminary settings for printing:

Temperature settings (according to feedstocks characteristics)

- 1 mm nozzle diameter
- Nozzle temperature: 150°C
- Extruder temperature: 110°C
- Cold point temperature: 43°C
- Building plate temperature : 35°C

Flow settings (according to machine supplier)

- « Extruder flow » : 35 %
- Printing speed: 20 mm/s

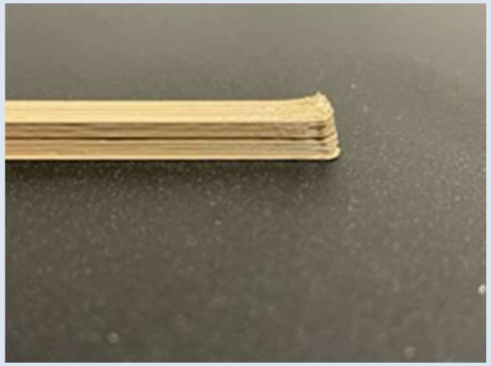
Printing strategy (following pure thermoplastic settings)

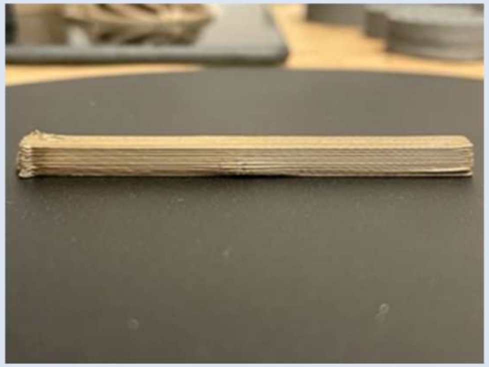
- Layer thickness : 1/3 nozzle diameter
- Overlapping = 1/4 * nozzle diameter
- Line filling
- Infill density : 100%

Printed parts (for testing)

- Test bars : 60 mm x 6,5 mm x 4,7mm
- Test discs : 40 mm diameter x 2 mm

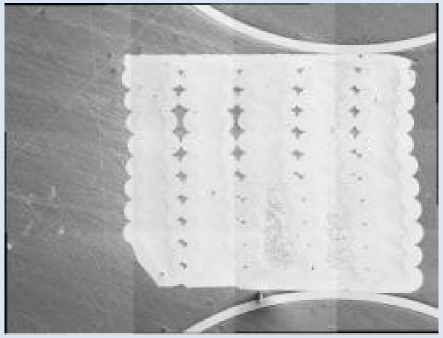
First printing … and some defects


Delamination


Improper part/building plate adhesion


Warping


Holes


Inter-filament voids

Fine tuning is required !

How to remove defects ?

1. Fine nozzles generate more defects (= statistically, more filaments = more issues, more prone to clogging)

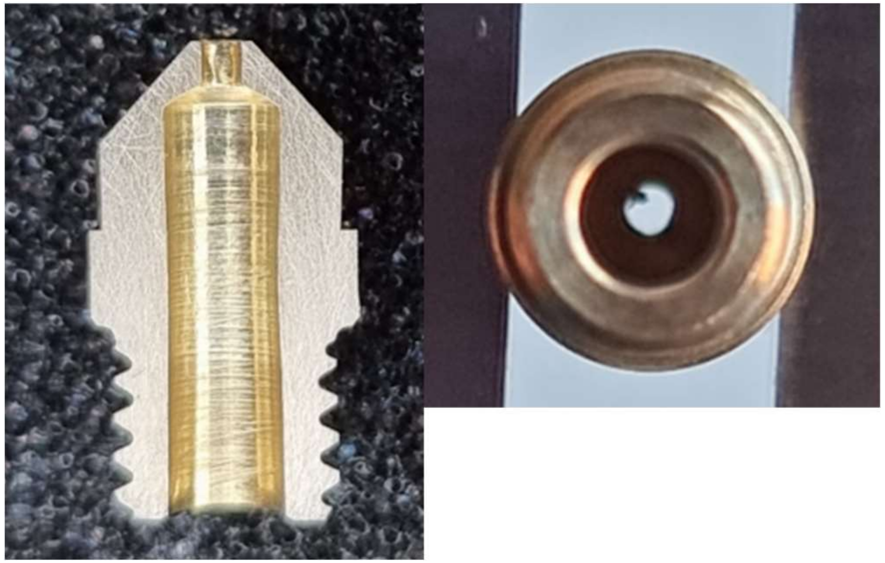
-> Prefer large nozzles (> 1 mm) for a more reliable process

2. Most internal defects can be avoided through over-extrusion (= increase flow and/or decrease speed)

-> More pressure on the deposited material -> better connection between filaments

3. Unexpected yet **major influence of the internal shape of the nozzle**

Original nozzle



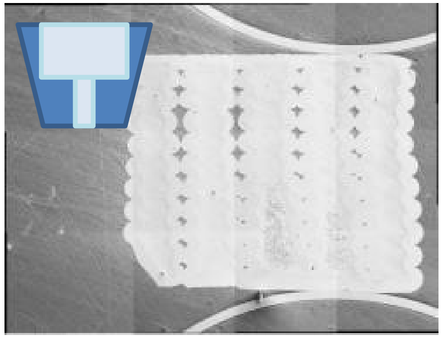
Alternate nozzle

Influence of nozzle shape / nozzle material

Identical processing conditions

Nozzle A

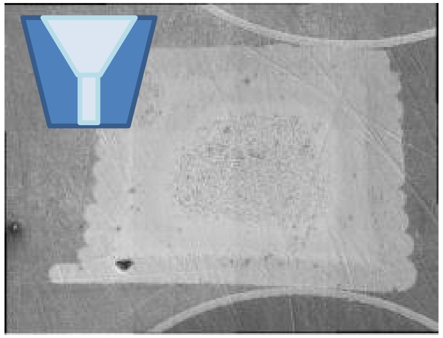
Sharp/Steel



Macroscopic defects

Nozzle B

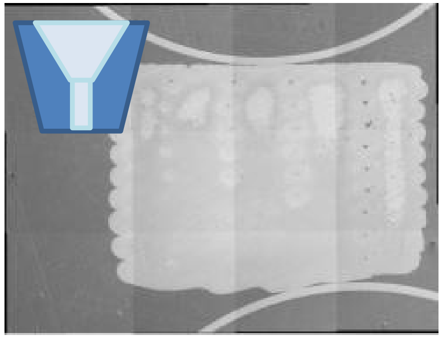
Smooth/Steel



Shape distorsion but almost no defects

Nozzle C

Smooth/Brass



No defects, no distorsion

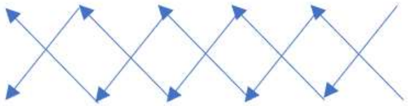
Sintered properties	Nozzle A		Nozzle B		Nozzle C
Apparent density	**5,81-5,97**		**5,78-5,96**		**5,94-5,98**
Open porosity	**0,5 - 3,4%**		**0,8 - 3,2%**		**0,3 - 0,8%**
Relative density	**96,8 - 99,5%**		**96,3 - 99,3%**		**99,0 - 99,7%**

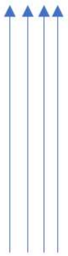
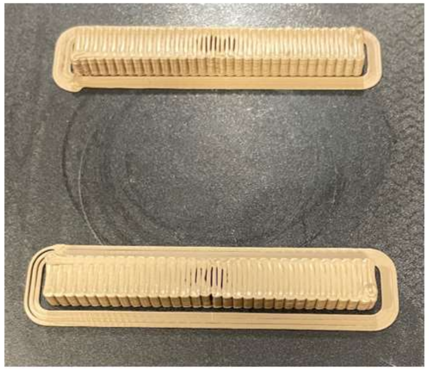
Less scattered
More reliable

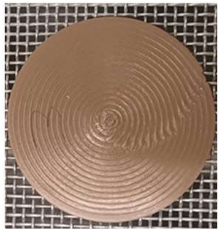
Influence of filling

- Is there any obvious influence of the filling in strategy on the sintered density ?





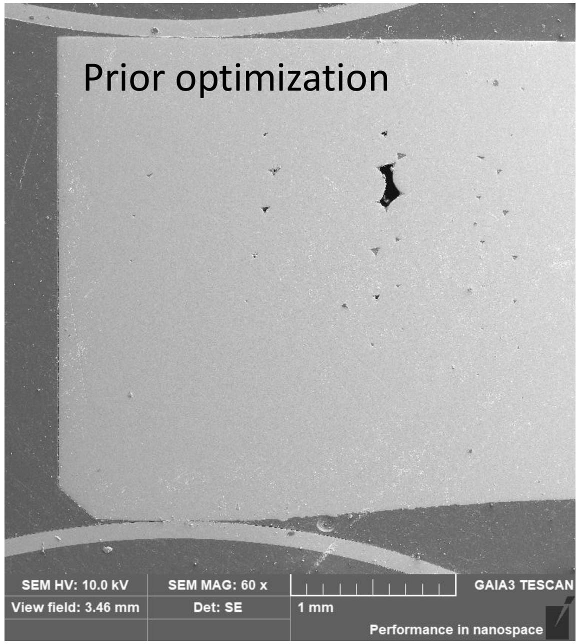



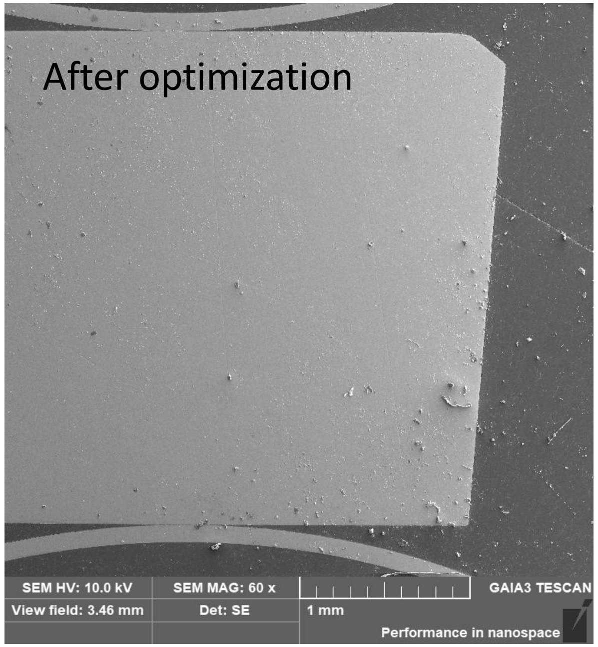



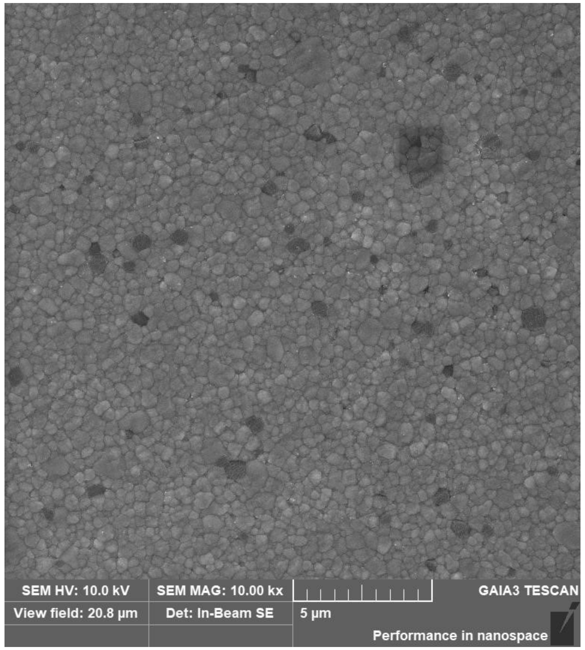

Sintered density	D app	Poro ouv	D rel
Alternate	5.94	0.9%	99.1
	5.95	0.9%	99.1
Concentric	5.97	0.5	99.6
	5.98	0.3	99.7
	5.96	0.5	99.4
Triangle	5.94	1%	99.0

- In the absence of contours, no appreciable difference was noted (100% infill)
- The influence of contours has not been assessed but could create gaps with the fill.

Improved process & mechanical properties







Nozzle material	Density (max.)	3-Points Bending strength (MPa)	Vickers Hardness
Brass	99,7	963 +/- 13	1394 +/- 29
Steel	99,3	877 +/- 45	1397 +/- 28

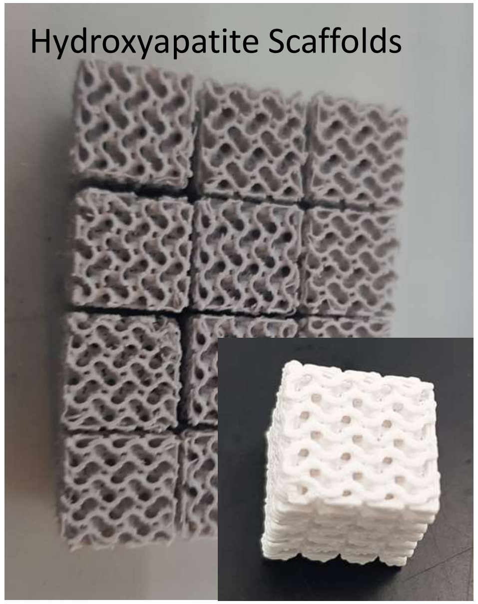
Just a few examples… zirconia & other materials


TPMS zirconia filters


SiC


Alumina radome

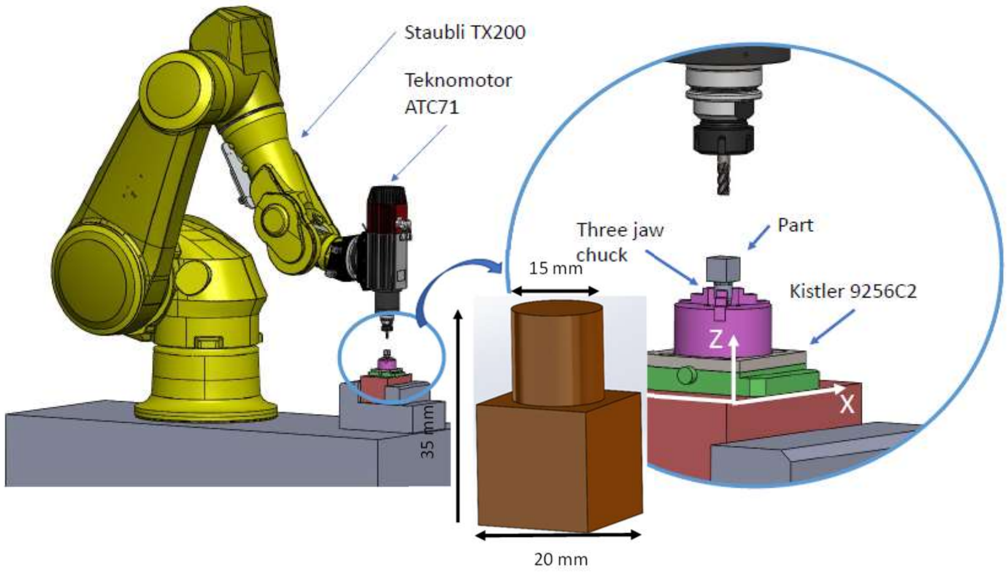

Fine zirconia mesh


Hydroxyapatite Scaffolds

On the road to hybridization … machining optimization

Two main objectives :

- **Determine the most suitable tools**
- **Determine the optimized cutting setting**



- Standardized samples held in a chuck with concentric jaws.
- Chuck connected to a load cell (Kistler 9256C2) which measures dynamic forces up to 250N.
- Machining is carried out by a Staubli Tx200 six-axis robot equipped with an 8kW Teknomotor ATC71 spindle.

- External faces of the cube are pre-machined.
- 3 repetitions of 3 passes are made with constant cutting parameters (forces are continuously recorded).
- Surface roughness and microscope images are then taken before the cutting parameters are changed.

A Systematic Approach to Determine the Cutting Parameters of AM Green Zirconia in Finish Milling, L. Spitaels & al., *J. Compos. Sci.* **2023**, *7*(3), 112

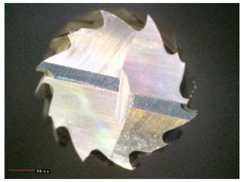
Machining optimization

Tools selection

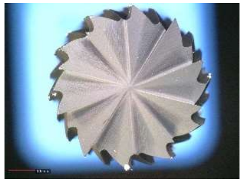
- No existing tool for green ceramics in the conventional suppliers' catalogues
- Choice of 3 standards tools
- Relatively low cost (~ 25€ /tool to ~ 95€/tool)

3 mm

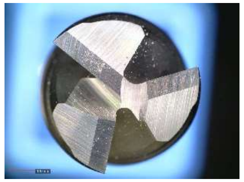
Tool 1
Thermoplastics



Tool 2
Composites



Tool 3
Universal



Cutting settings

Four cutting parameters were used: the cutting speed (v_c), the feed per tooth (f_z), the depth of cut (a_p) and the width of cut (a_e)

Tool	v_c [m/min]	f_z [mm/tooth]	a_p[mm]	a_e[mm]
Thermoplastics	383	0.030	3	0.5
	443	0.030	3	0.5
	306	0.030	3	0.5
	383	0.036	3	0.5
	383	0.024	3	0.5
	383	0.030	3.6	0.5
	383	0.030	2.4	0.5
	383	0.030	3	0.6
	383	0.030	3	0.4
Composites	324	0.030	3	0.5
	389	0.030	3	0.5
	259	0.030	3	0.5
	324	0.036	3	0.5
	324	0.024	3	0.5
	324	0.030	3.6	0.5
	324	0.030	2.4	0.5
	324	0.030	3	0.6
	324	0.030	3	0.4
	324	0.030	3	0.5
Universal	270	0.030	3	0.5
	309	0.030	3	0.5
	228	0.030	3	0.5
	270	0.036	3	0.5
	270	0.024	3	0.5
	270	0.030	3.6	0.5
	270	0.030	2.4	0.5
	270	0.030	3	0.6
	270	0.030	3	0.4

The experiments aim at determining the working domain for v_c,min, f_z, a_p and a_e.

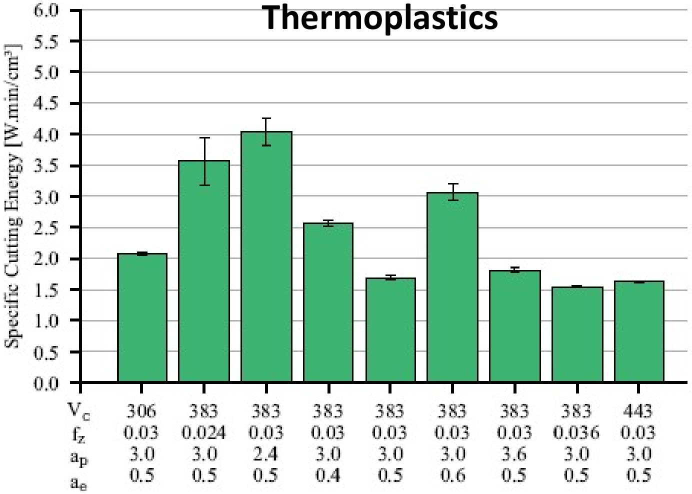
Tool	v_c [m/min]	f_z [mm/tooth]	a_p[mm]	a_e[mm]
Thermoplastics	383			
Composites	324	0.030	3	0.5
Universal	270			

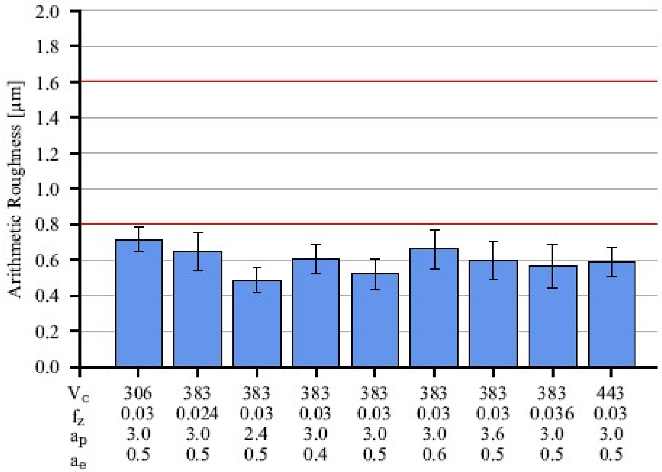
Initial settings

- $$F_{tot,RMS} = \sqrt{\frac{1}{3} \cdot (F_x^2 + F_y^2 + F_z^2)}$$ "Average force"
- $$h_m = 2 \cdot f_z \cdot \sqrt{(\frac{a_e}{D}) \cdot (1 - \frac{a_e}{D})}$$ Mean chip thickness (D is the tool diameter)
- $$W_c = \beta \cdot \frac{\sqrt{\frac{1}{3} \cdot (F_x^2 + F_y^2 + F_z^2)}}{120 \cdot a_p \cdot f_z \cdot \sqrt{(\frac{a_e}{D}) \cdot (1 - \frac{a_e}{D})}}$$ Specific cutting energy

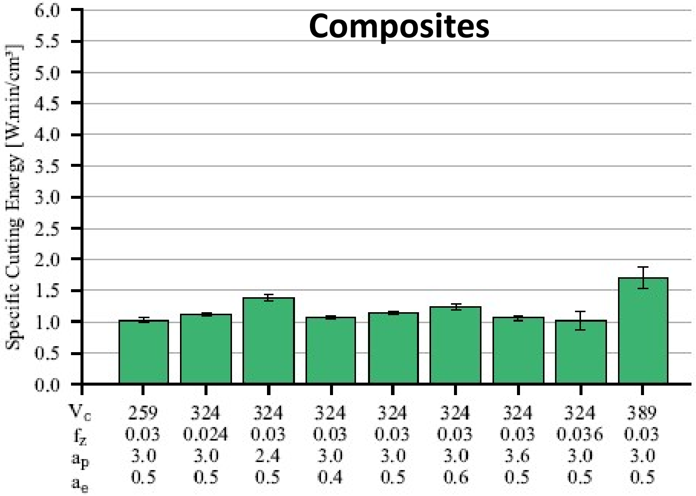
Specific cutting energy & roughness

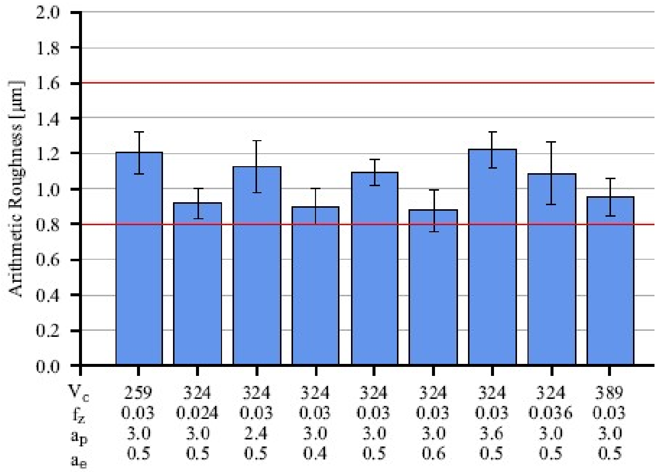
Tool 1 Thermoplastics



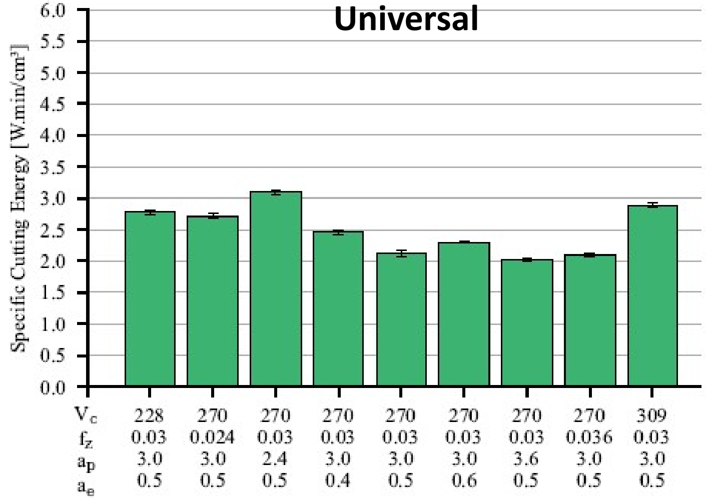


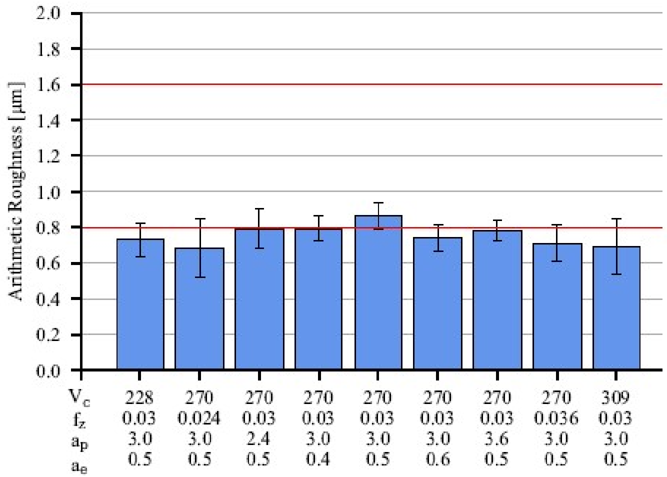
Tool 2 Composites





Tool 3 Universal

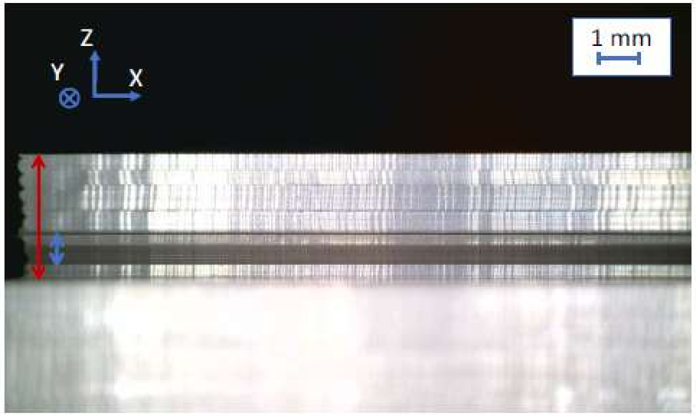


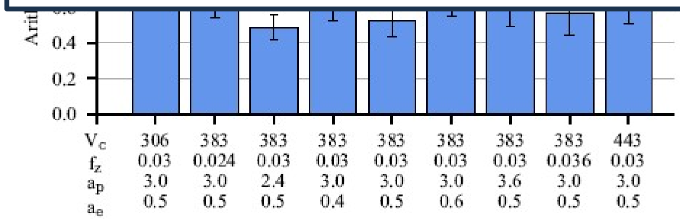


Specific cutting energy & roughness

Tool 1
Thermoplastics

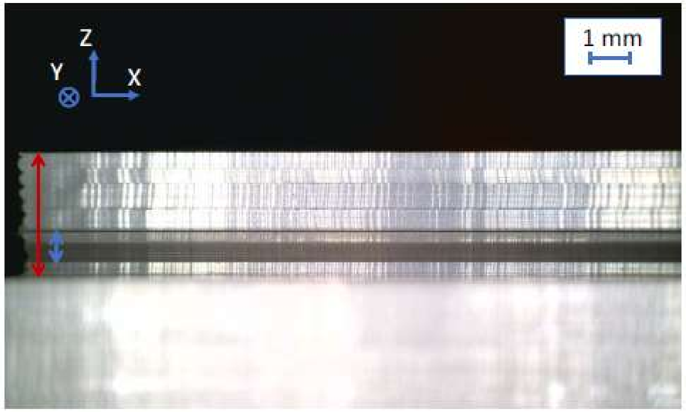
- Repeatable cutting energy
- Very smooth < 0,8 µm
- No defects

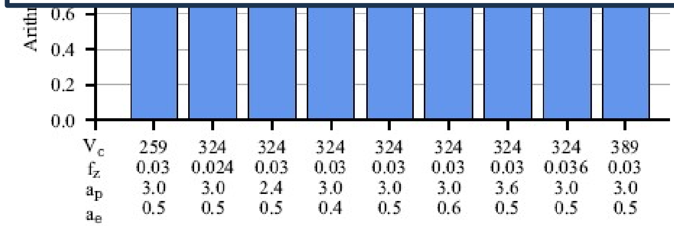




Tool 2
Composites

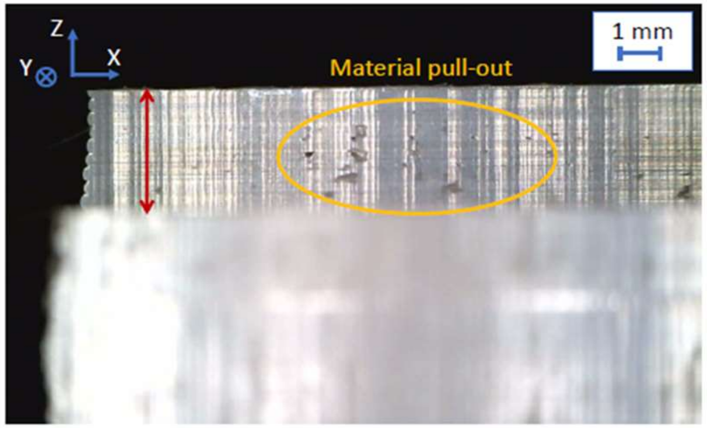
- Repeatable cutting energy
- Smooth < 1,6 µm
- No defects

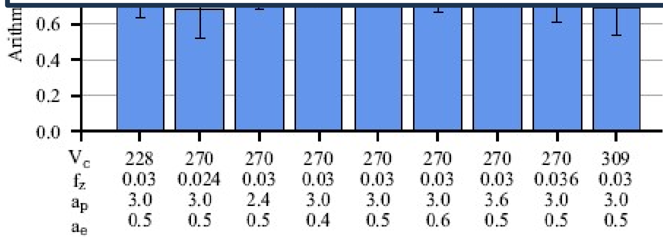




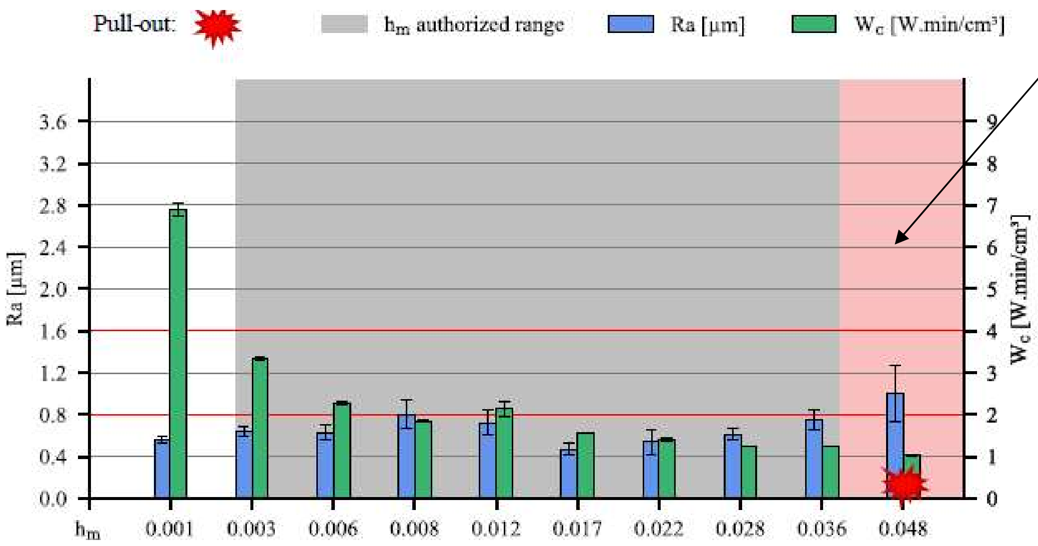
Tool 3
Universal

- Repeatable cutting energy
- Creation of defects





Optimized conditions



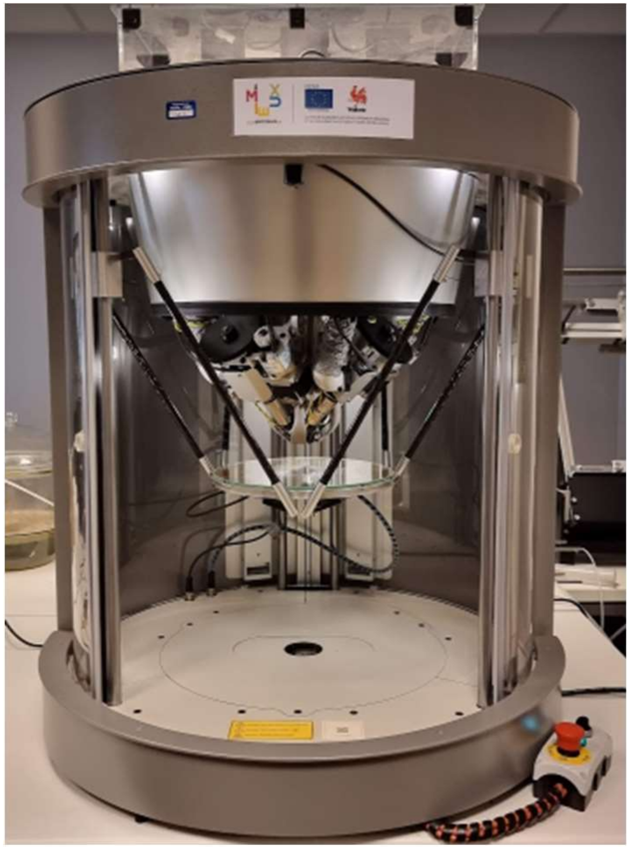
Permissible range for chip thickness

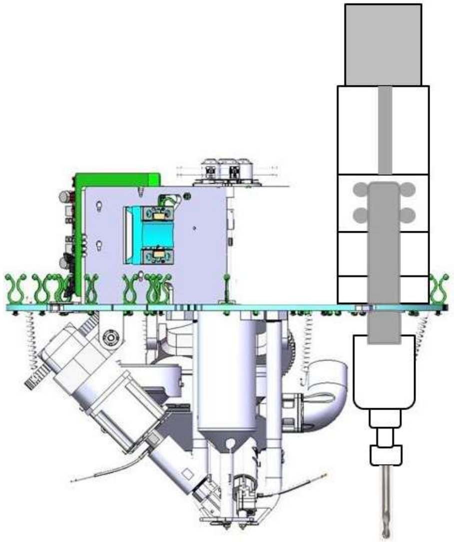
Material removal rate : from 5 to 10 cm3/min
Thermoplastic tool approx. 4 times cheaper than composite ones
Very low Ra for the thermoplastic tool

The thermoplastic tool is by far the most suitable option !

Hybrid PAM - hardware

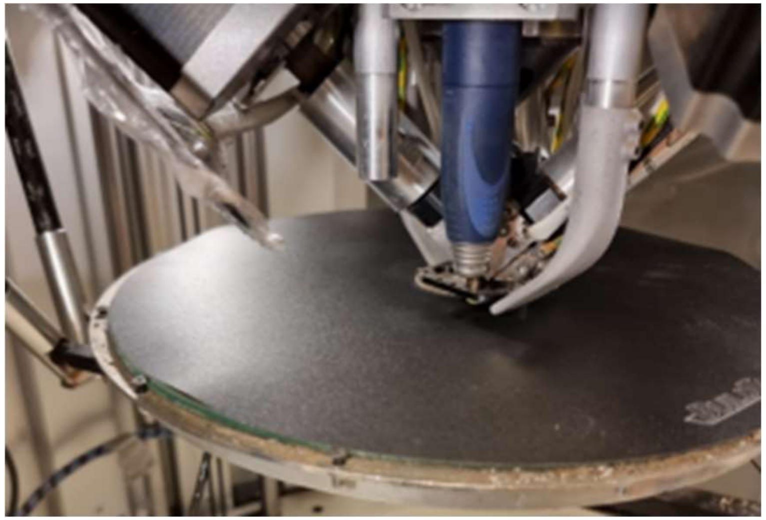
Objective : add milling capabilities to the PAM system





Spindle :

- Max speed : 50 000 rpm
- Max torque : 6 N.cm



Hybrid PAM - software

Objective : how to ensure a proper communication between the various machine components?

1) CAM software selection

2) Create a suitable post-processor

-> make gcode toolpaths compatible with PAM-gcode

-> XYZ zero offsets, disable printing…

3) Define the printed and milled parts

-> Printed part is enlarged so as to compensate shrinkage during sintering + offset outline for milling

4) Select a generic 3D milling strategy

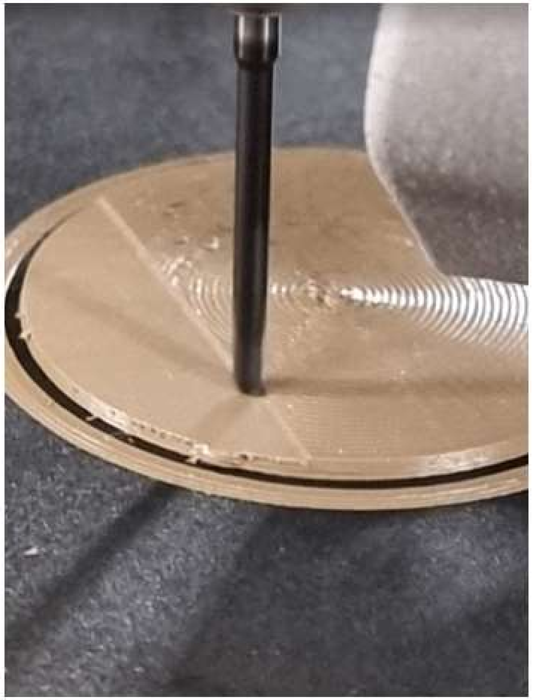
-> Compatible with most use cases

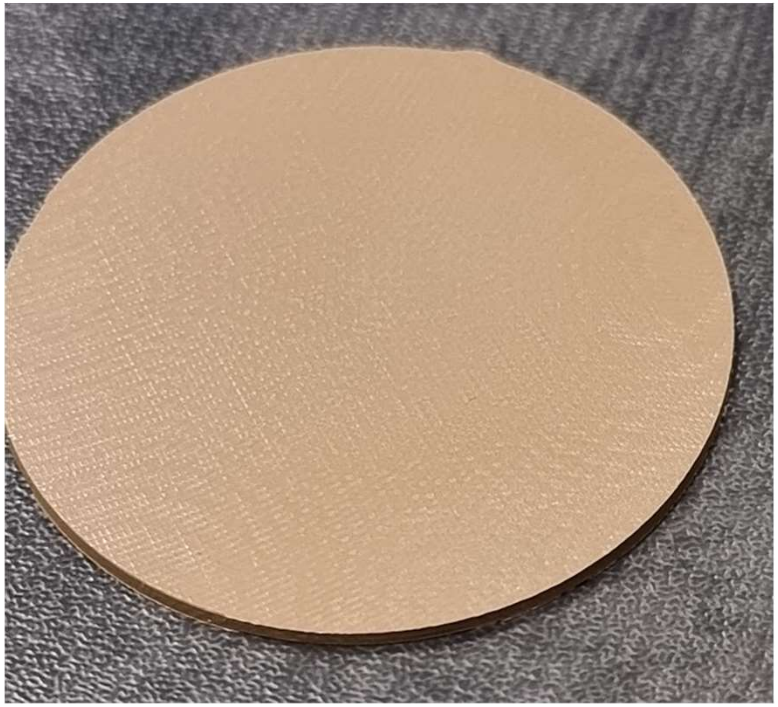
5) Testing various type of parts

-> Gradual complexity :

- Planar surfacing (single printing/milling sequence)
- Post-finishing of a complex shape (single printing/milling sequence)
- Full hybrid of a complex shape (multiples sequences of printing & milling)

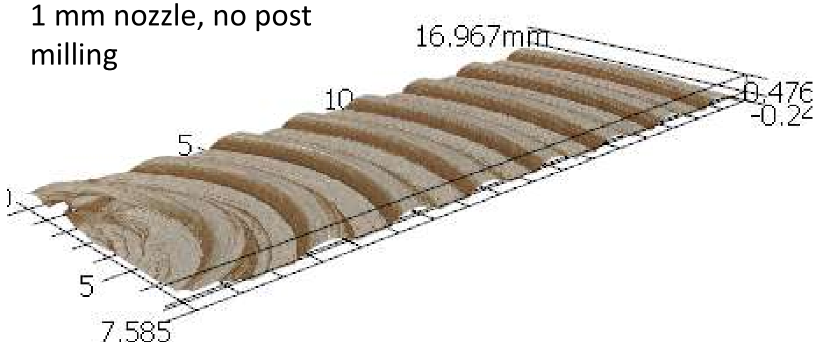
Hybrid PAM - 2D

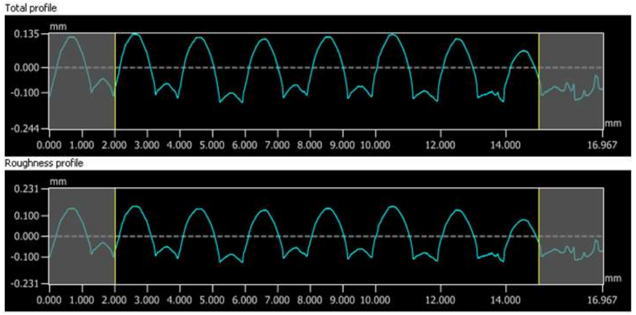




Hybrid PAM - 2D

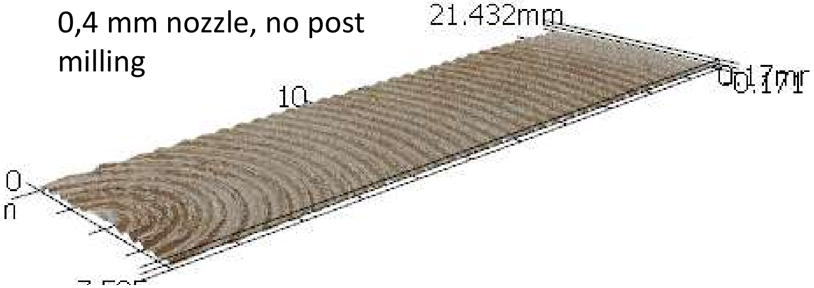
1 mm nozzle, no post milling

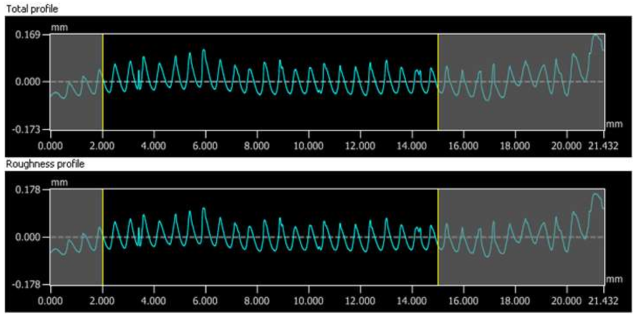




No.	Measurement name	Measured value	Unit
1	Ra	0.080	mm
2	Rz	0.274	mm

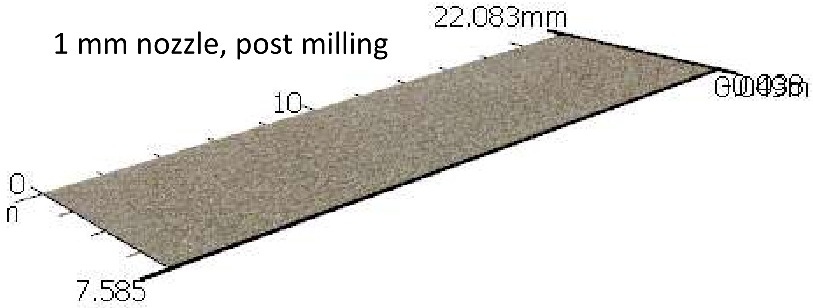
0,4 mm nozzle, no post milling

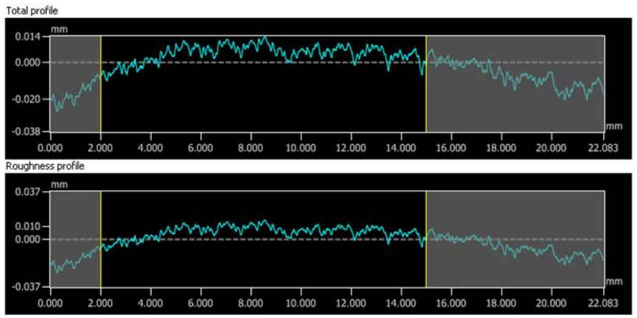




No.	Measurement name	Measured value	Unit
1	Ra	0.032	mm
2	Rz	0.164	mm

1 mm nozzle, post milling

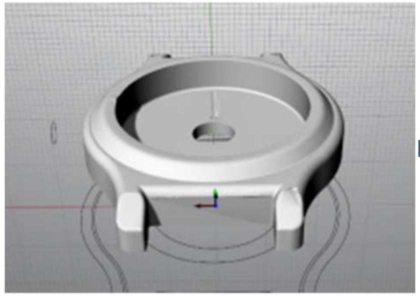




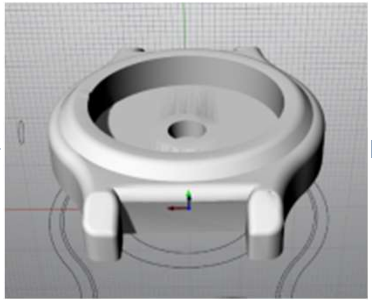
No.	Measurement name	Measured value	Unit
1	Ra	0.007	mm
2	Rz	0.024	mm

Hybrid PAM – from 2D to 3D - post-finishing

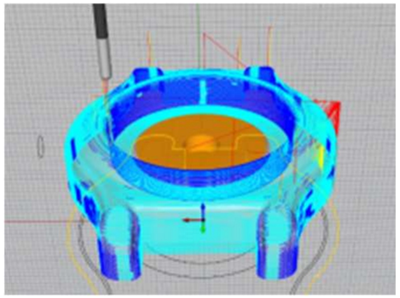
Step 1 : CAD software



Original file

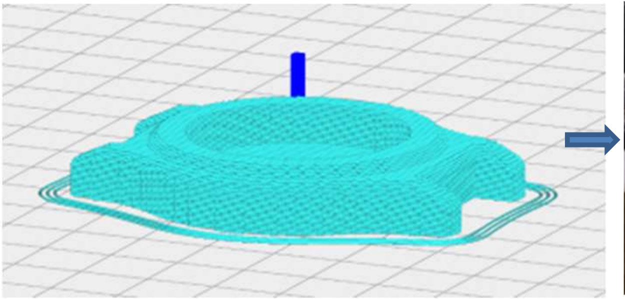


Enlarged model



Toolpath calculation

Step 2 : PAM: CURA + home made post-processor



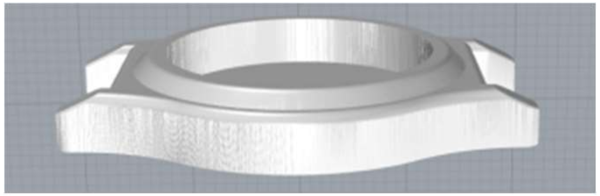
Enlarged sliced model

Enlarged printed model

Milled green body

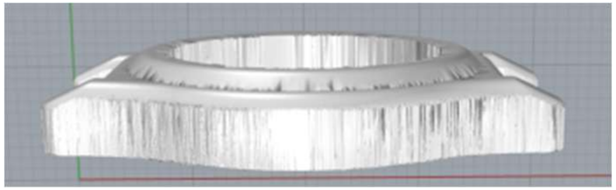
Hybrid PAM – from 2D to 3D - post-finishing

CAD file



0,6 mm offset

Over-sized CAD file



Printed zirconia watch case with 400 µm nozzle - as-built.

Printing time ~ 3 hours

Watch case printed with a 1 mm nozzle - Approx. 30 minutes

Watch case printed with a 1 mm nozzle - Approx. 30 minutes

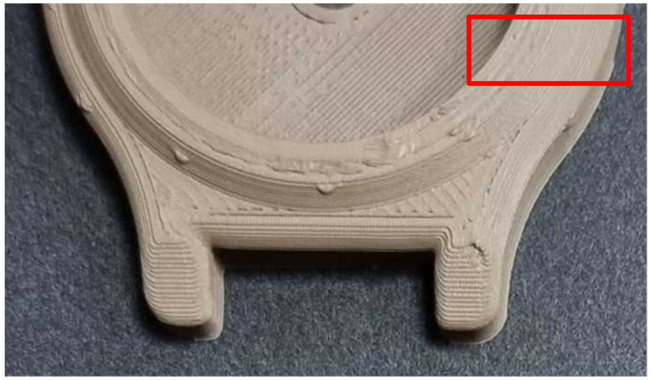
Manufacturing time ~ 1 hour

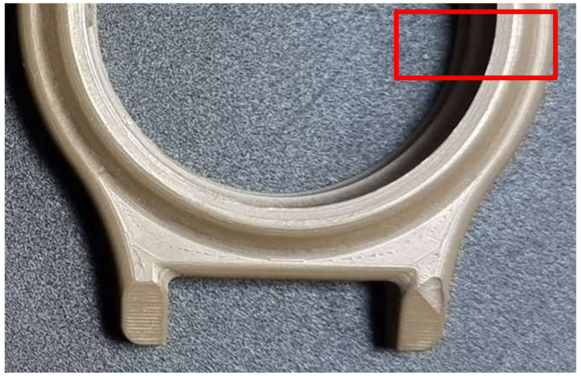
Hybrid PAM – from 2D to 3D - post-finishing

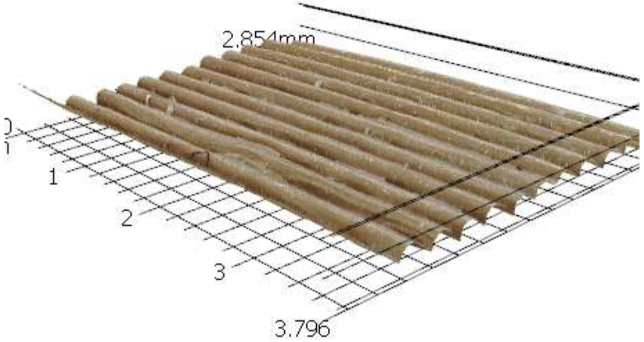
Surface finish of a case printed on the PAM with a 0.4 mm nozzle

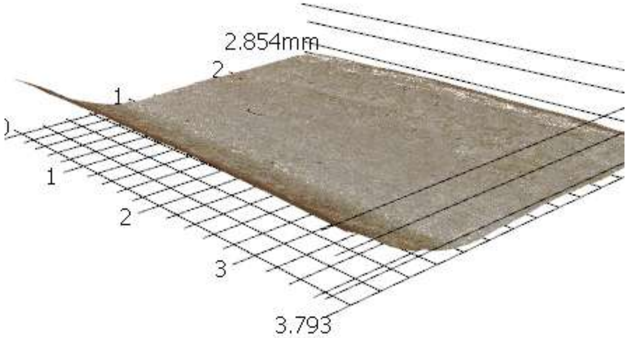
Surface finish of the watch case after a finishing pass

Hybrid PAM – from 2D to 3D - post-finishing







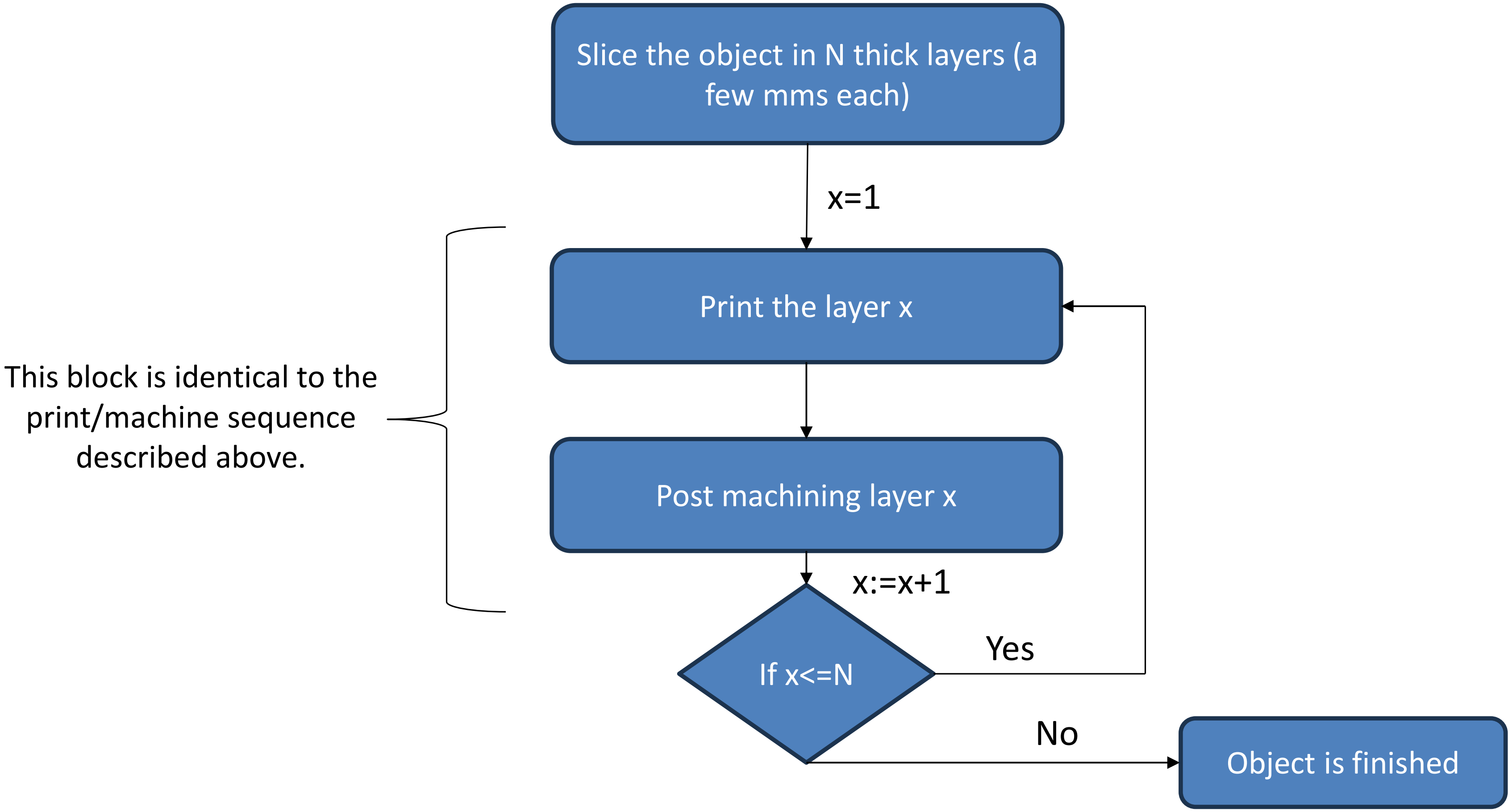


No.	Measurement name	Measured value	Unit
1	Ra	0.015	mm
2	Rz	0.056	mm

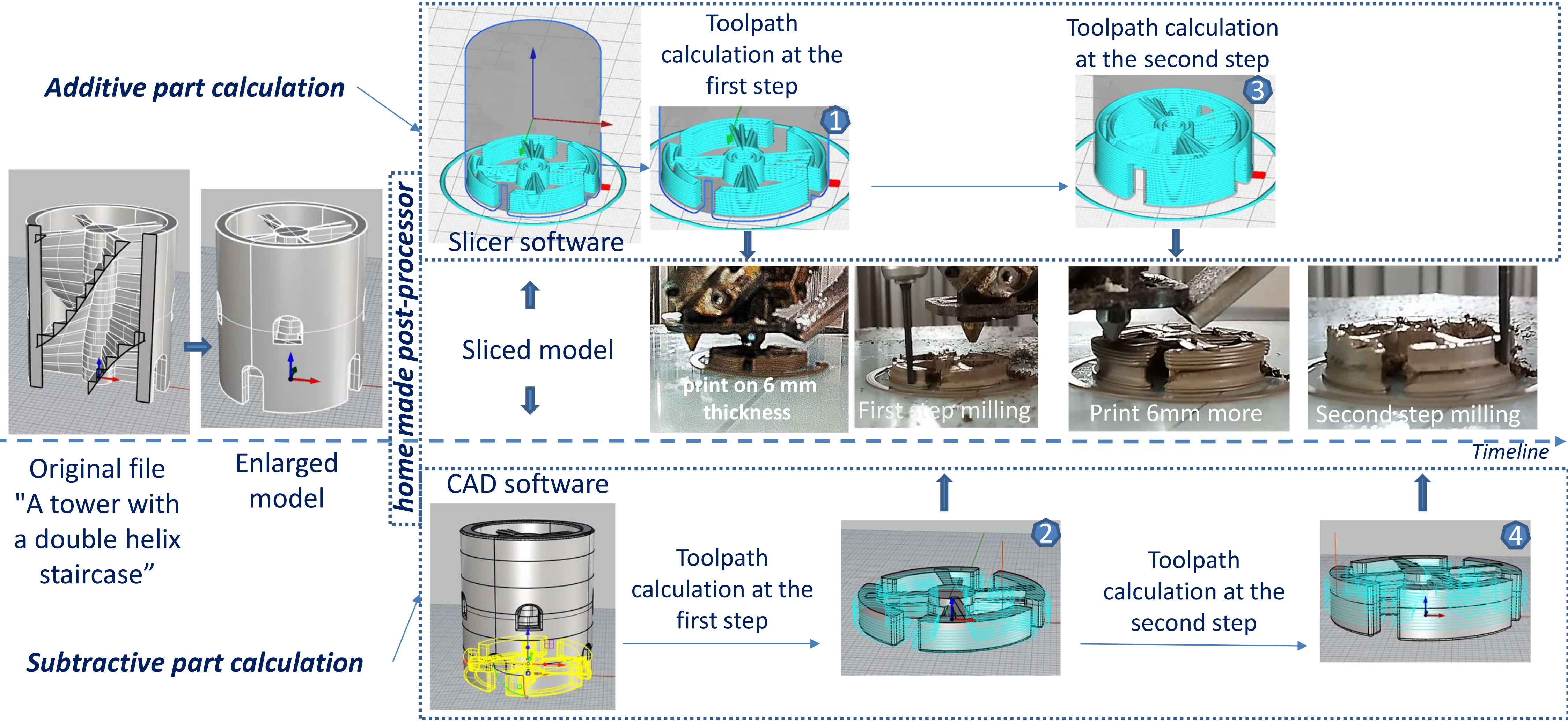
No.	Measurement name	Measured value	Unit
1	Ra	0.001	mm
2	Rz	0.003	mm

Hybrid PAM – Complete chain additive/subtractive

How to go from post-finishing to full hybridization ?



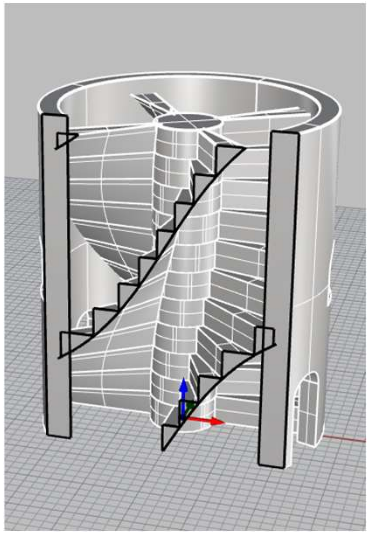
Hybrid PAM – Complete chain additive/subtractive



*Ref: TFE de Joris ALBOS , FPMS Umons

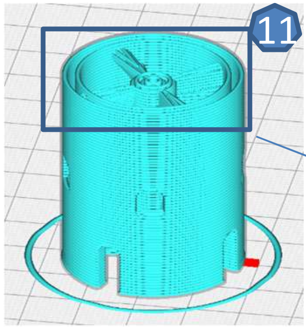
Hybrid PAM – Complete chain additive/subtractive

Additive part calculation



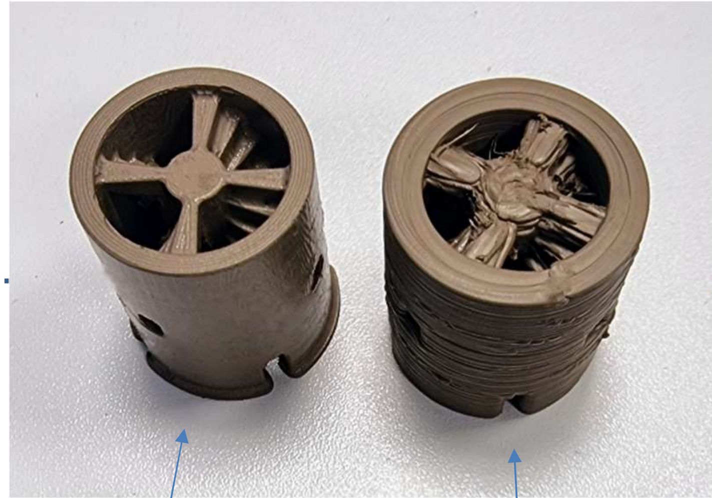
Original file
"A tower with a double helix staircase"

home made post-processor



Slicer software

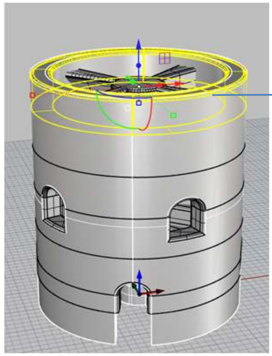
print the last slice



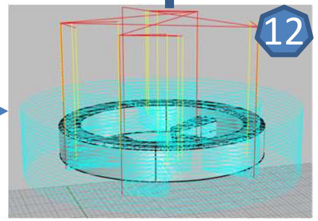
Toolpath calculation at the last step

Timeline

CAD software



Toolpath calculation at the last step



40 minutes for milling

40 minutes for printing (nozzle 1.8mm)

10 minutes for tool motion

Subtractive part calculation

Much shorter manufacturing time (:7)
90 minutes against 650 minutes (nozzle 0.4mm)

BioCAM - Additive Manufacturing Applied to Bioceramics

BioCeramic additive manufacturing is a revolution in the personalized treatment of various pathologies, particularly in the regeneration of various anatomical parts of the human body such as dental, maxilofacial, skeletal or cranioplastic.

Given the growing interest in these topics, we are pleased to invite you to t**he first international Workshop dedicated to ceramic additive manufacturing for biomedical applications.** Jointly organized by the Belgian Ceramic Research Centre (BCRC) and the European Ceramic Society (ECerS), with the support of the EMC (Additive Manufacturing) and Bioceramics ECerS Networks and of the JECS Trust, the Workshop will be held **in Mons on 6th and 7th December 2023**. The event will combine invited presentations by renowned international researchers with significant contribution in the field from different countries and an open call for papers.

The Workshop will be followed on **8th December by a one-day MasterClass** during which international experts will share their experience of different additive technologies (Robocasting, Pellet Additive Manufacturing, Powder Bed Processes, Stereolithography) through theoretical lectures and practical applications on the BCRC equipment.

Located in the heart of Wallonia and close to the French border, in Mons, BCRC is a major research center in Belgium, particularly in the field of materials science. The conference will be an opportunity to strengthen the links and collaborations between researchers, clinicians and industrialists but also to inspire a new generation of scientists on this topic of major societal importance.

We look forward to welcoming you in December 2023!

- **Ceramic Additive Manufacturing**
- **Workshop + Masterclass**
- **MONS, december 2023 !**

https://ecers.org/

Come along and join us !

Thank you for your attention !