Synthesis of freestanding porous alumina and copper nanowires for high power laser applications

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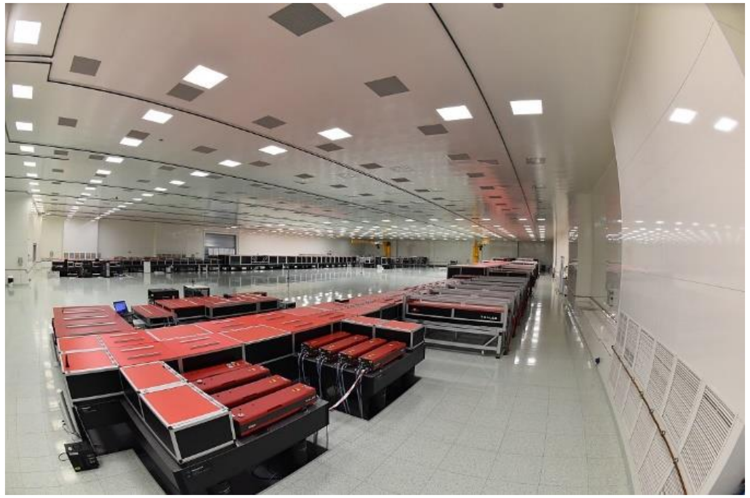
Short introduction

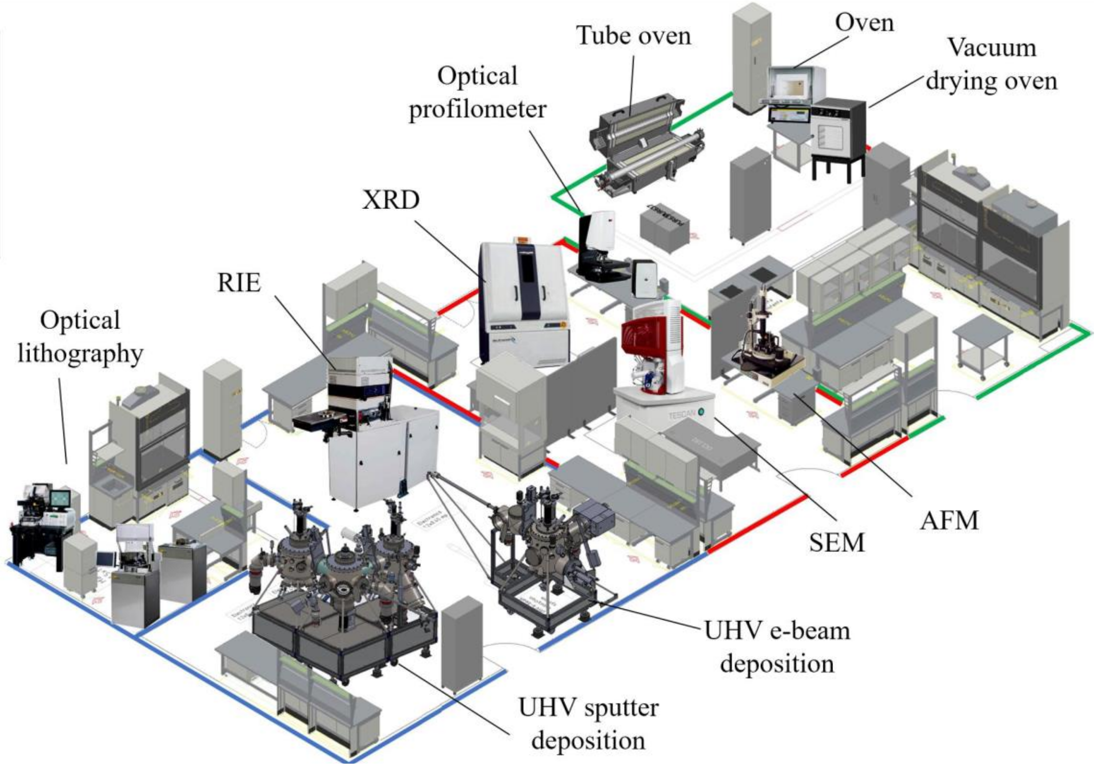
Porous alumina and metallic nanowires: an overview

Experimental

Results

ELI-NP Targets Laboratory



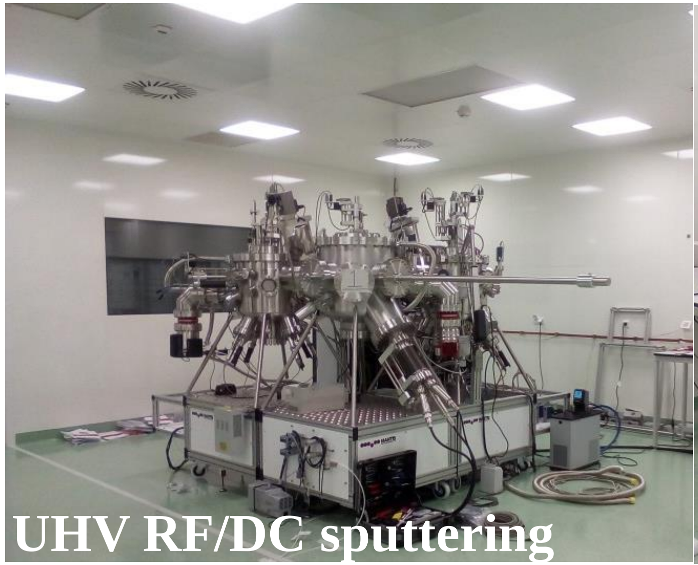


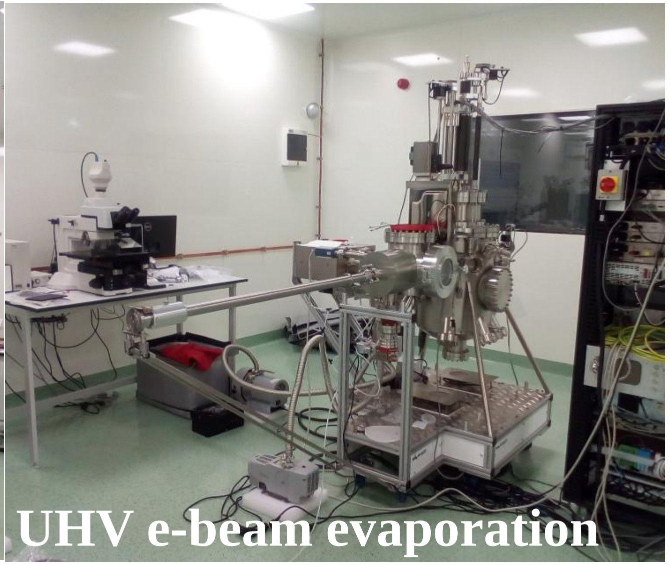
- ✓ Provide targets – in-house manufacturing and characterization
- ✓ Perform R&D activities – of new target designs with improved performances

- Total surface area 268 m^2
- ISO 7 and ISO 6 cleanliness
- 4 designated rooms:
  nanofabrication, characterization,
  chemistry room, microassembly

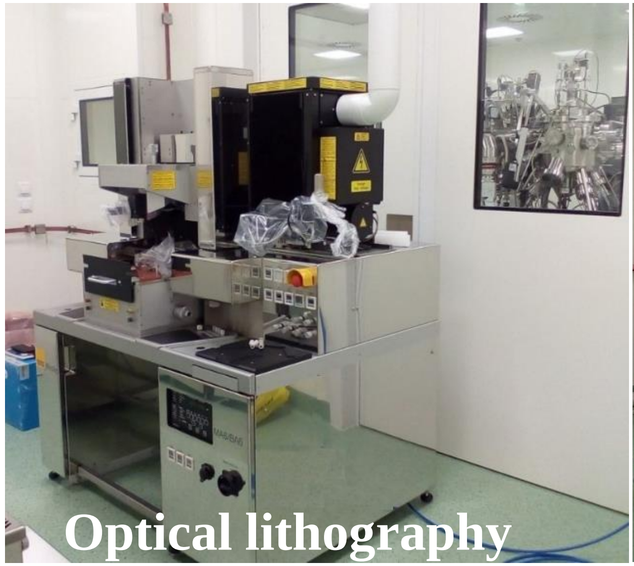
ELI-NP Targets Laboratory - capabilities

Deposition techniques


UHV RF/DC sputtering

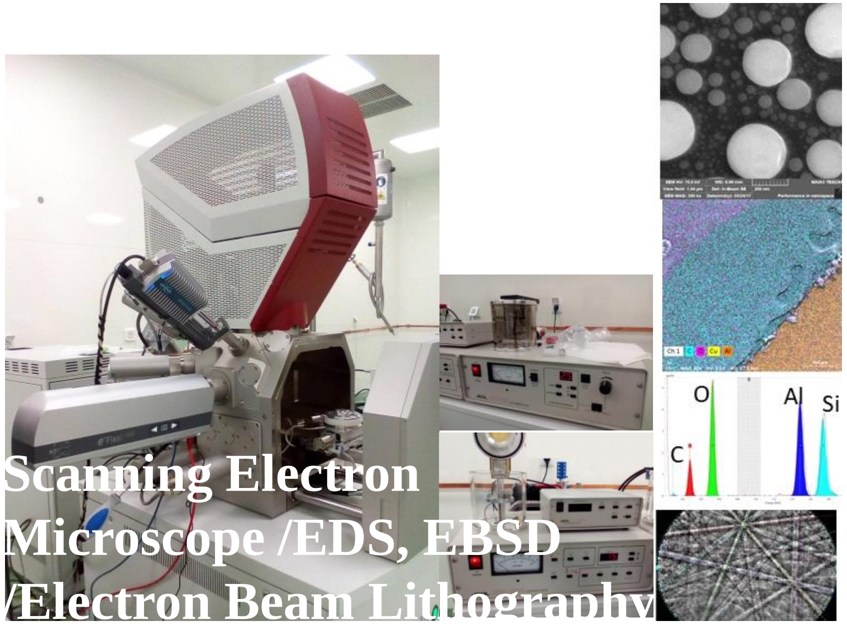

UHV e-beam evaporation

Structuring techniques


Optical lithography

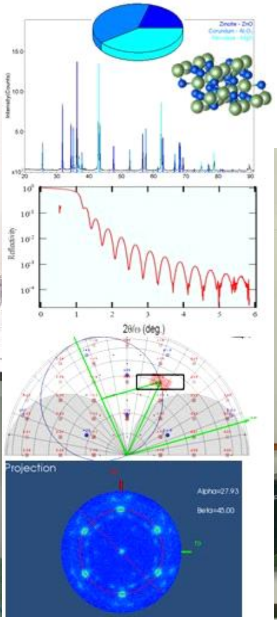

Reactive ion etching

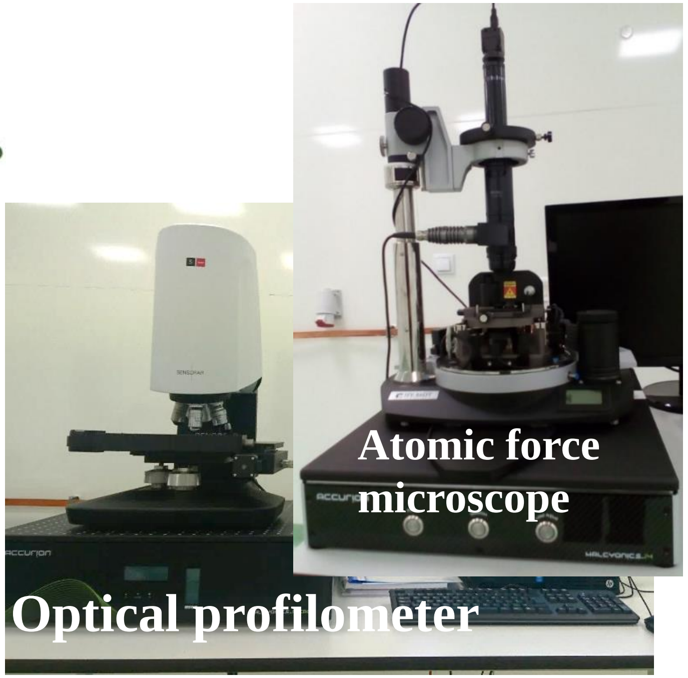
Characterization



Scanning Electron Microscope /EDS, EBSD /Electron Beam Lithography

X-ray Diffractometer




Optical profilometer

Atomic force microscope

Cleaning/conditioning methods

- Plasma (O_2, Ar, SF_6)
- Ion beam (Ar)
- Thermal treatments

Tubular oven

Calcination oven

Benchtop plasma treatment system

Micro-machining / mechanical processing

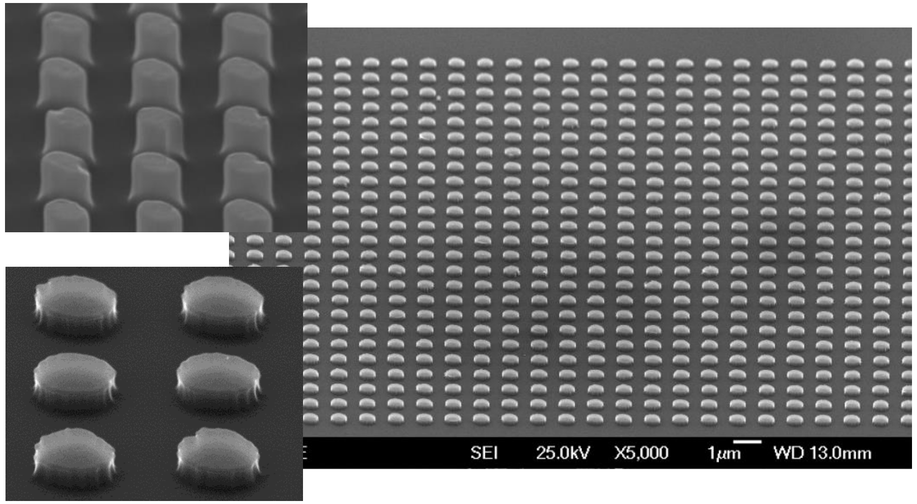
Cutting machine

Lapping/polishing machine

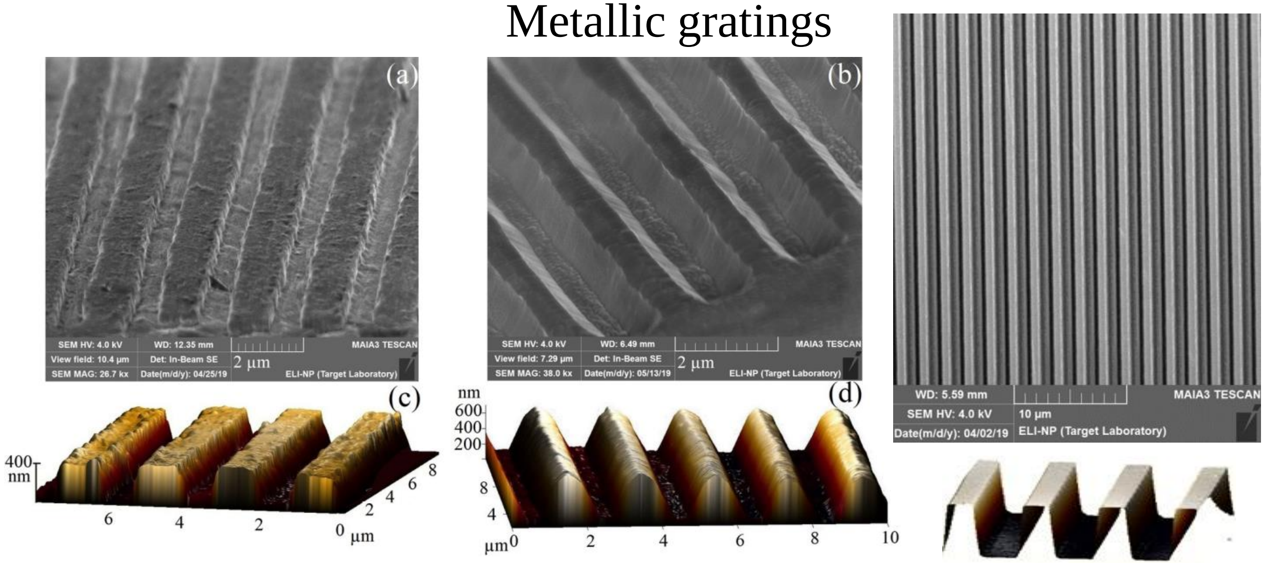
Electrochemical set-up

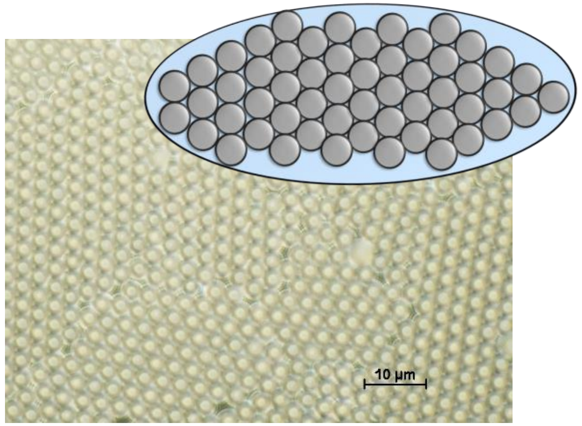
On-going research: micro/nanostructured targets, free-standing targets

Metallic micro/nano dots

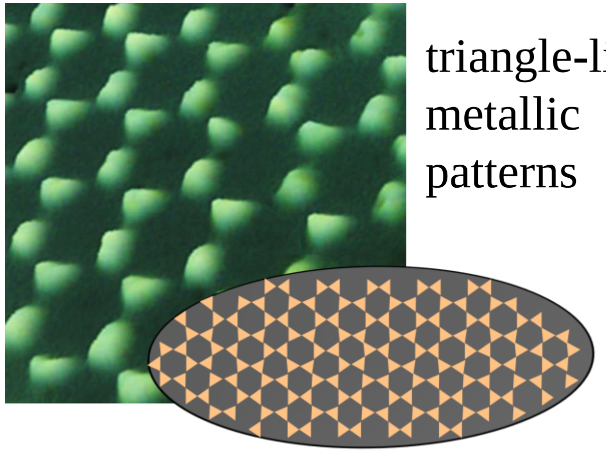


Metallic gratings



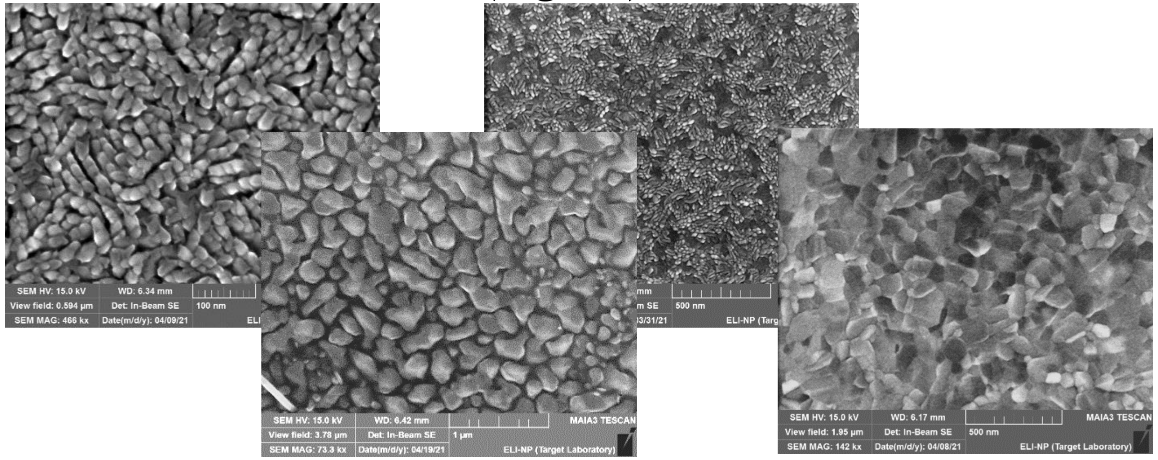


microspheres



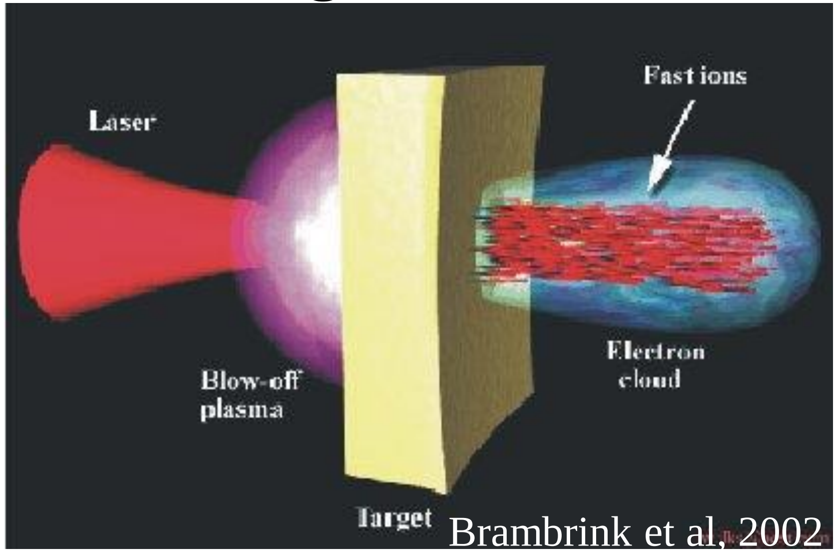
triangle-like
metallic
patterns

Metallic (high-Z) films



1.1. Short introduction

Laser Targets



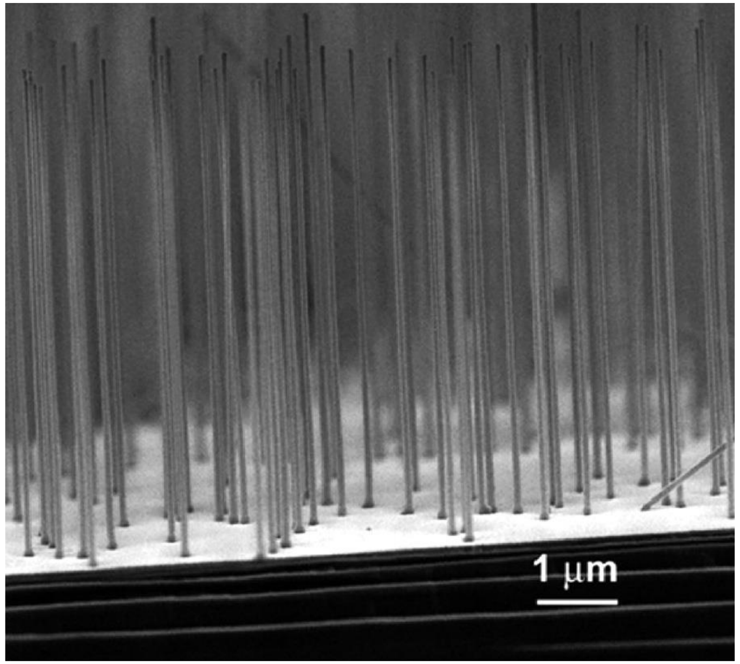
Types of targets:

- solid (thin/thick/ultra-thin films, multi-layer, foams, nanospheres, snow clusters, NWs, gratings, nanoparticle, micro-cone...)
- cryogenic
- gas jet
- liquid crystals, and so on.

Nanowires (NWs)

NWs are particularly interesting as targets for laser experiments → allowing the investigation of physical light-matter interaction phenomena *(particle acceleration, dynamic compression, radiation source...)*

NWs are structures with high aspect ratio with unique **magnetic, optic and electric properties.**



Particle-in-cell simulations and laser-driven ion acceleration experiments on NW arrays (brush-like geometry) showed **improved laser absorption** (on-going research).

Using these structures as targets, the improvement of the energy of the accelerated ion at intensities (laser irradiance) of **10^{21} -10^{23}** W/cm^2 can be investigated.

1.2.Methods for NW synthesis

Bottom-Up

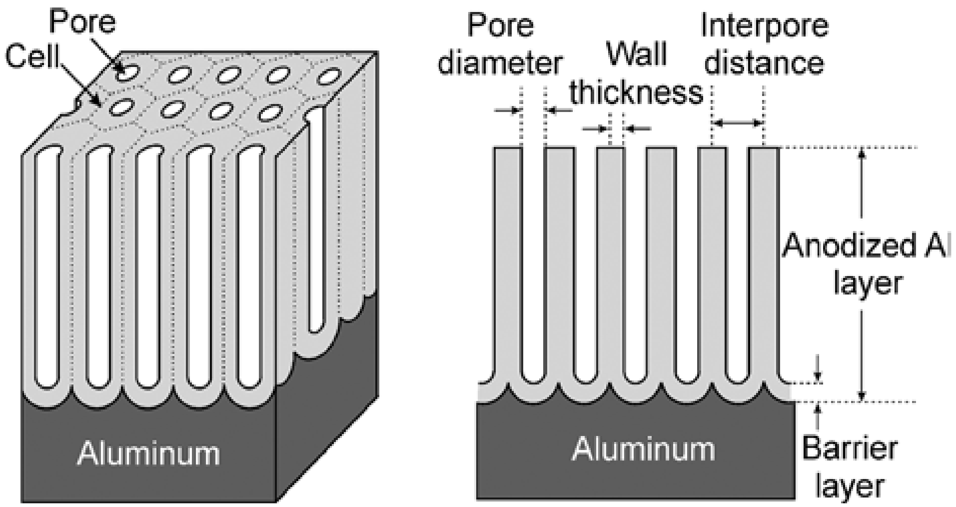
- Vapor-Liquid-Solid
- Chemical Vapor Deposition
- Hydrothermal
- Molecular Beam Epitaxy
- Electrodeposition → Advantages:
  - wide range of tunable parameters (material, diameter, height)
  - low-cost equipment and materials

Top-Down

- Reactive Ion Etching
- Metal Assisted Chemical Etching
- Lithography

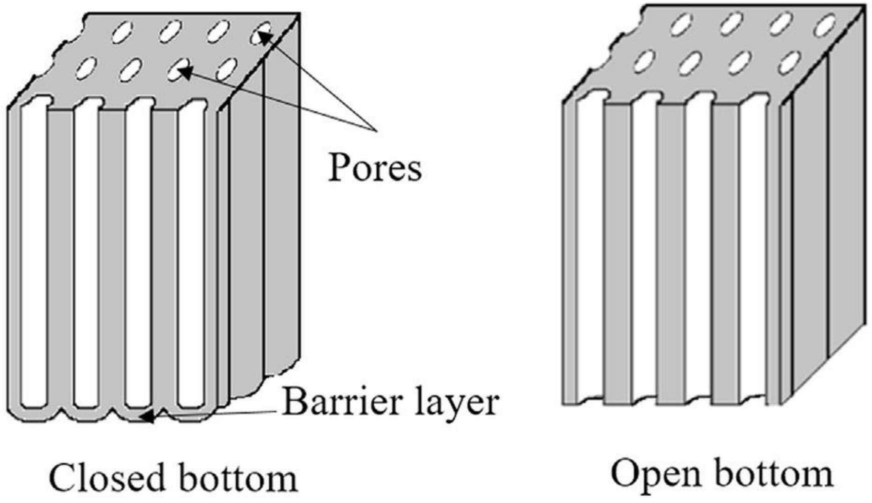
1.3. Anodization process

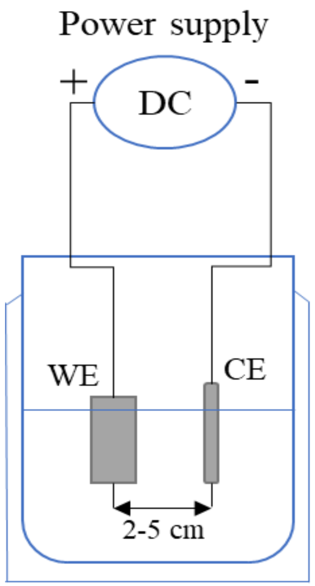
- Al anodization is an electrochemical process (made in an electrochemical cell) that transform Al in porous alumina.
- This cell is equipped with a double glass wall and two electrodes connected to a DC power supply in constant voltage (potentiostatic) or constant current (galvanostatic) mode: anode → Al plate, cathode → inert.
- Between the anodized Al and non-anodized Al is the ***barrier layer***, a continuous layer of alumina, at the bottom of the pores.
- Other characteristics of the porous alumina are *anodized layer, pore diameter, interpore distance, wall thickness*.
- Freestanding alumina can be obtained by detaching the anodized layer from the aluminum substrate by electrochemical means.



(Sulka 2008)

Free-standing alumina
(no Aluminium substrate)



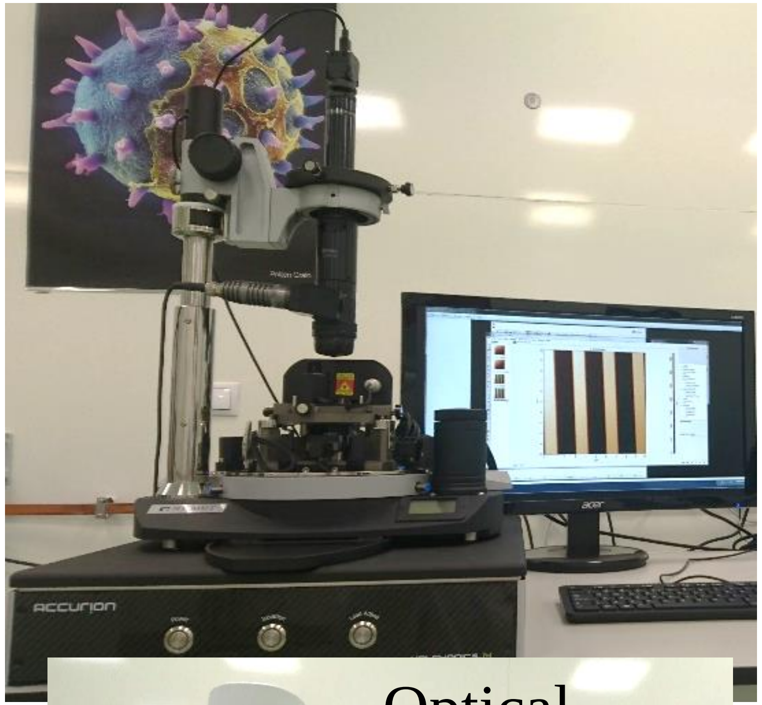


2.1. Electrochemical set-up

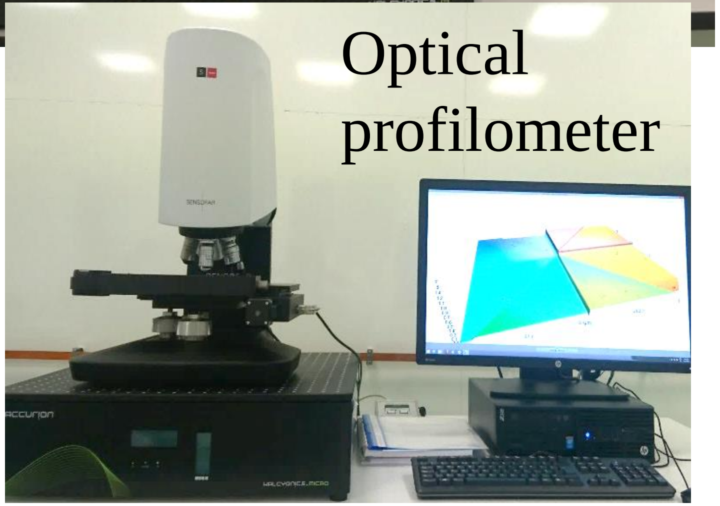
Electrochemical set-up, used for aluminum anodization included a double wall glass electrochemical cell connected to a chiller, a DC power supply, 2 bench-top multimeters for current and voltage monitoring, connected to a PC interface, and a stirring and heating plate. Also, for pulsed electrodeposition, an oscilloscope, an amplifier, and a programmable power supply were also used.

2.2. Characterization techniques

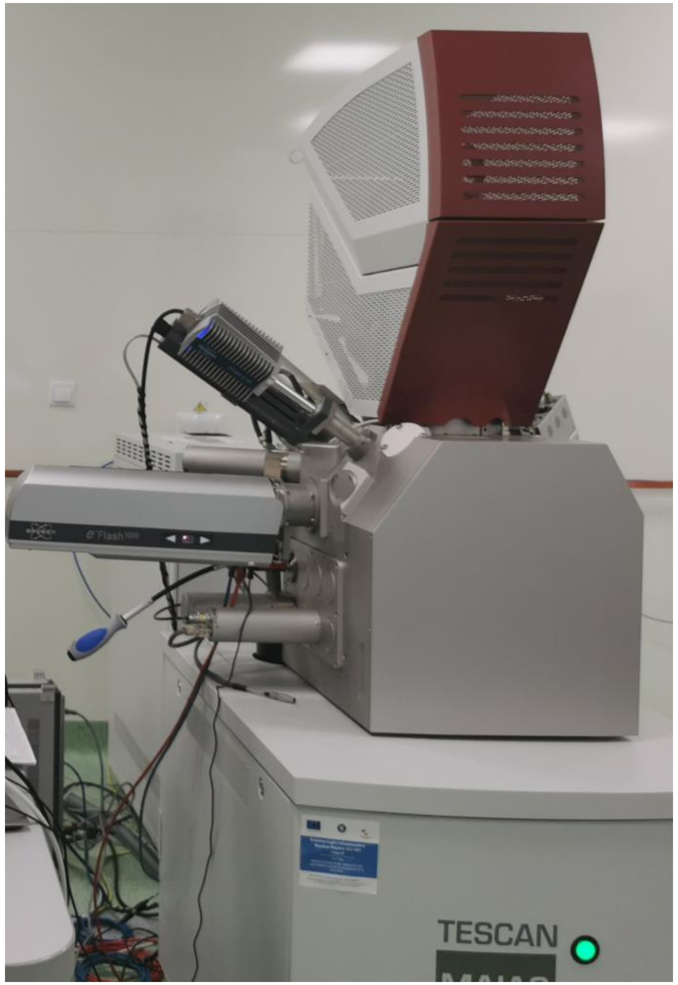
Atomic force microscopy (AFM)



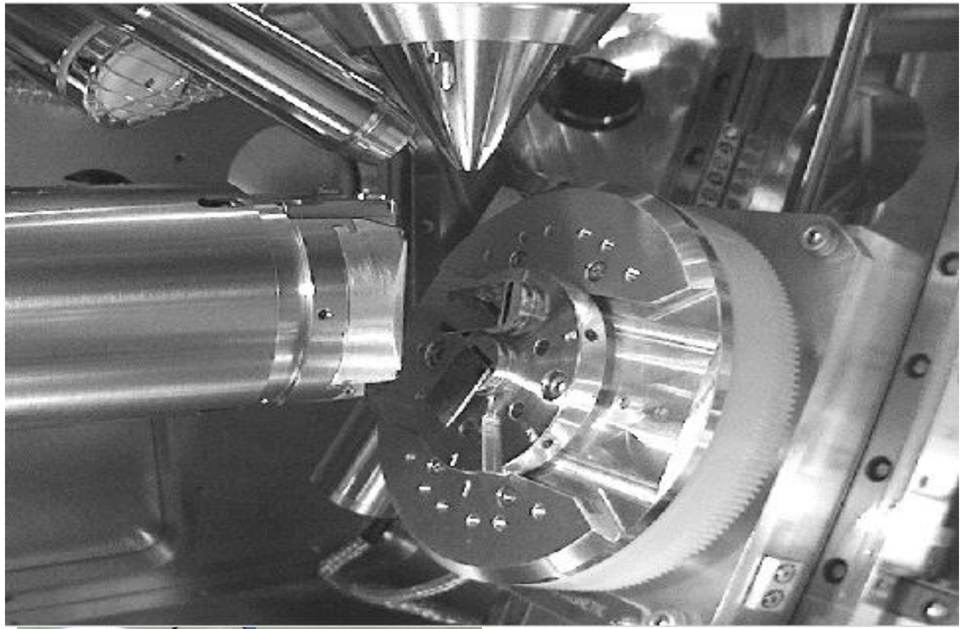
Optical profilometer

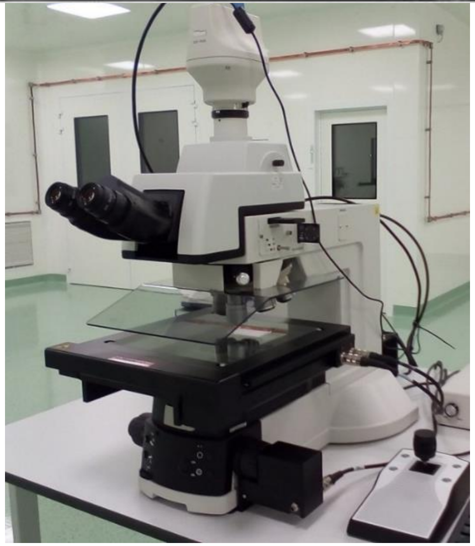


Scanning electron microscopy (SEM)



Energy-dispersive X-ray spectroscopy (EDS) & Electron Backscatter Diffraction (EBSD)





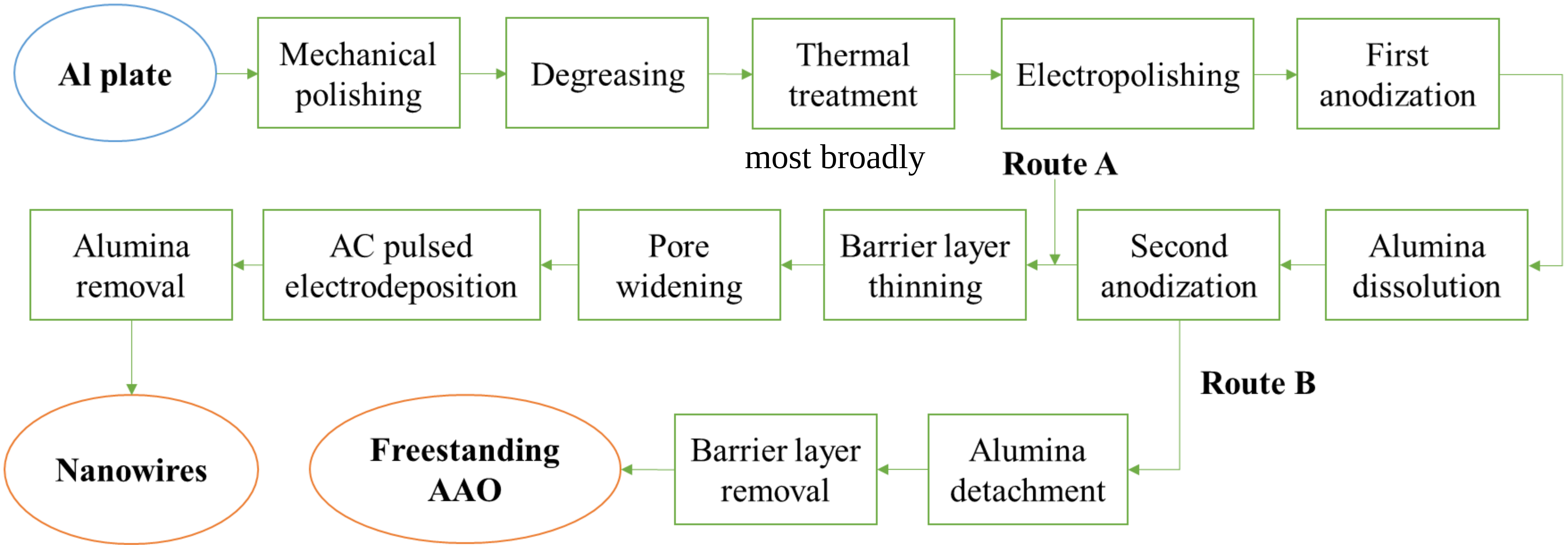
Optical microscope

3. Results

- The aluminum plate is prepared by mechanical polishing, degreasing, thermal treatment before the two-step anodization method.
- After the second anodization two routes were tested:

  A) NWs on aluminum substrate synthesized by AC pulsed electrodeposition

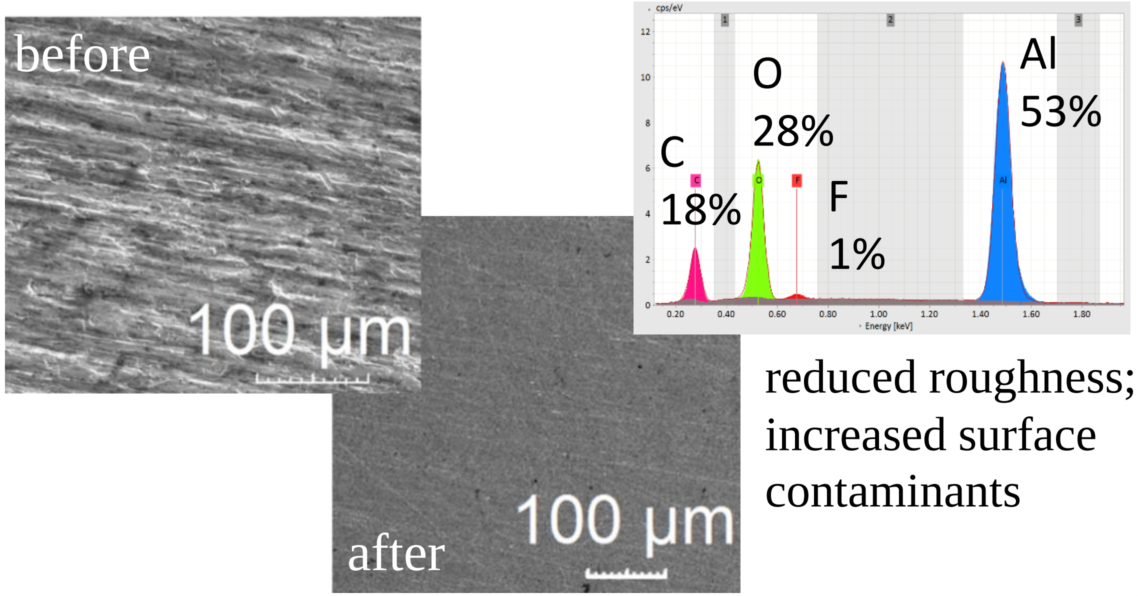
  B) freestanding porous alumina template by electrochemical detachment.



General block diagram of the process

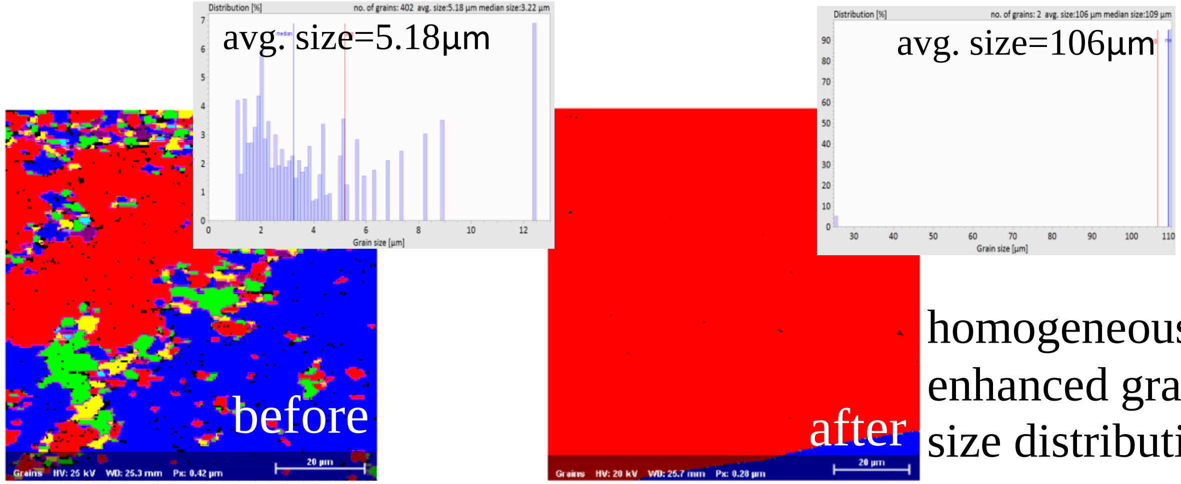
3.1. Aluminum foils pre-processing

Mechanical polishing



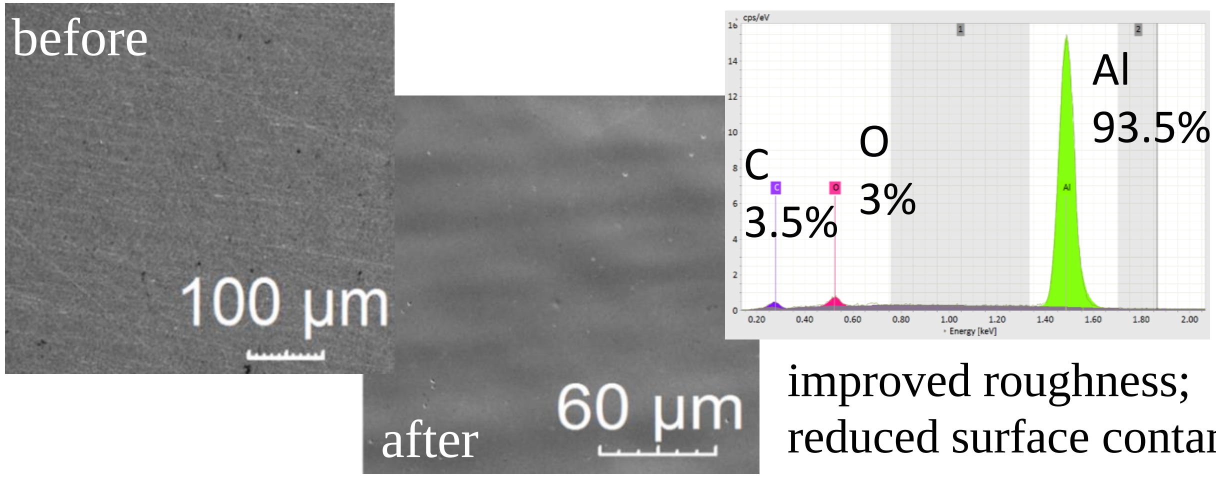
reduced roughness;
increased surface
contaminants

Thermal treatment

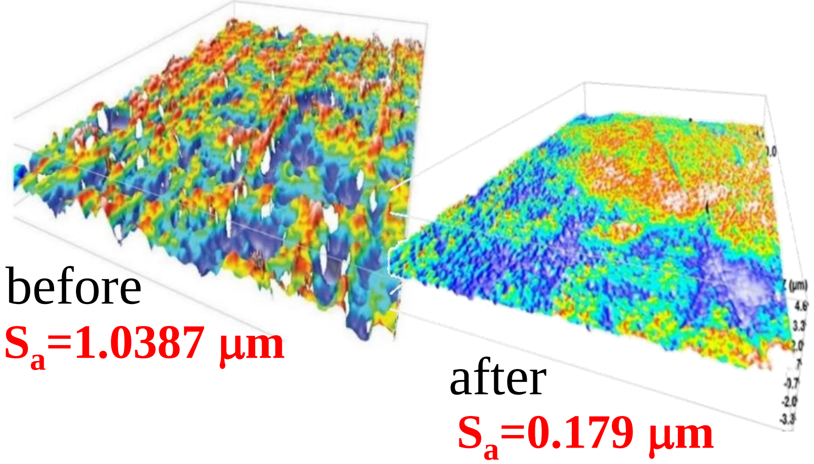


homogeneous,
enhanced grain
size distribution

Electropolishing



improved roughness;
reduced surface contaminants



3.2. Synthesis of porous alumina and copper NWs on Al substrate

One-step anodization and pulsed AC electrodeposition

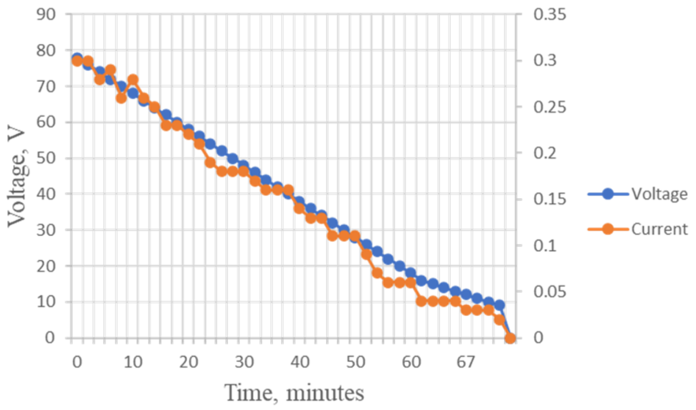
Conditions:

One-step anodization process

Anodization voltage set at 40V for 10 minutes, after which was raised by 0.5V /5 seconds until it reached 120V and held for 70 minutes.

Barrier layer thinning

By electrochemical method



Alumina dissolution

Partly liberation of NWs: 2min, 1M NaOH at 40C

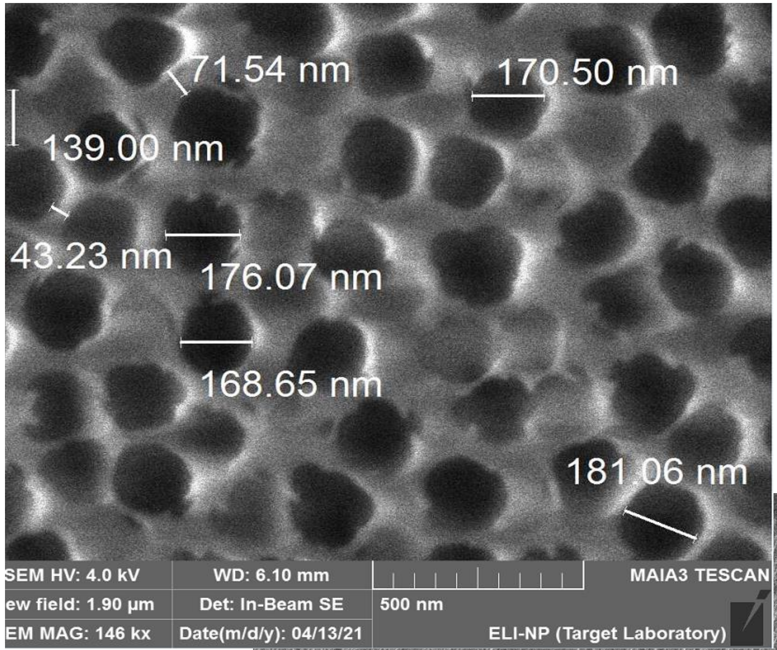
Fully dissolved after 3.5min same solution.

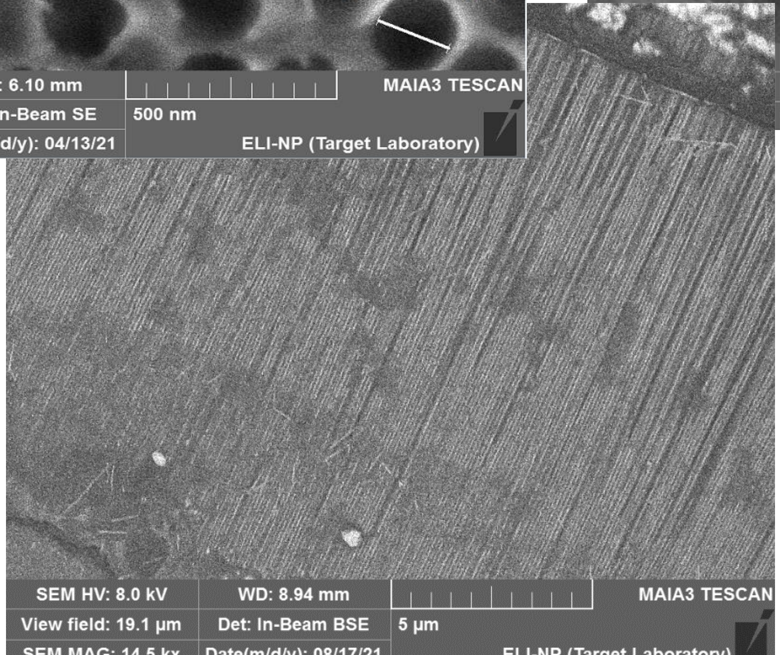
- **Pulsed AC electrodeposition** frequency= 200Hz
- voltage ±15V
- Electrodeposition solution: 0.5M $CuSO_4$ and 0.57M boric acid (H_3BO_3) (pH 3-3.5).
- Deposition duration= 30 min
- Edges and back of Al plate was thoroughly covered with nail polish.

3.2. Synthesis of porous alumina and copper NWs on Al substrate

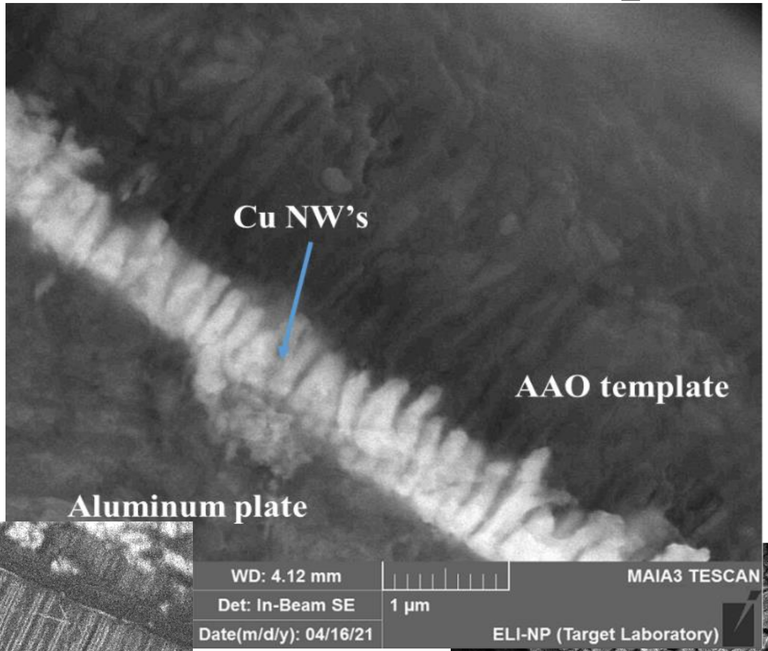
One-step anodization and pulsed AC electrodeposition Results

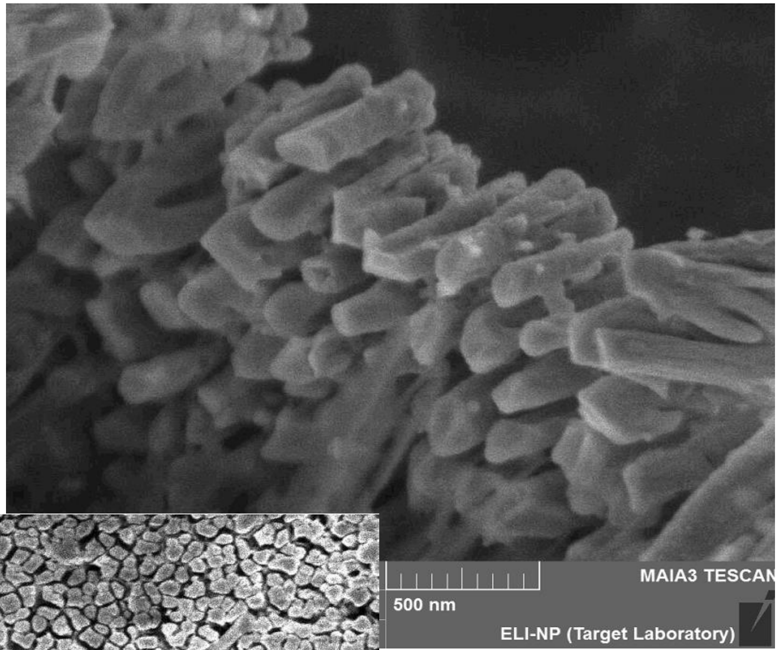
Anodization

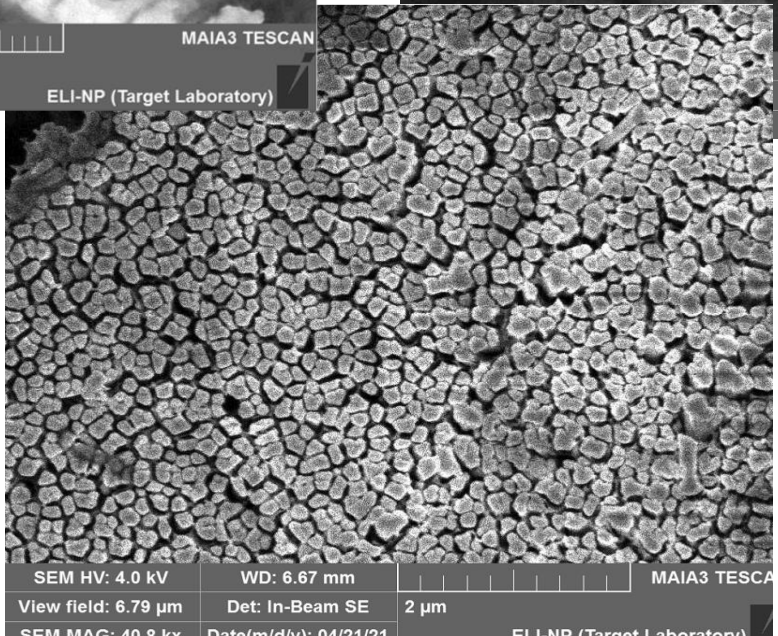




Pulsed AC electrodeposition and dissolution

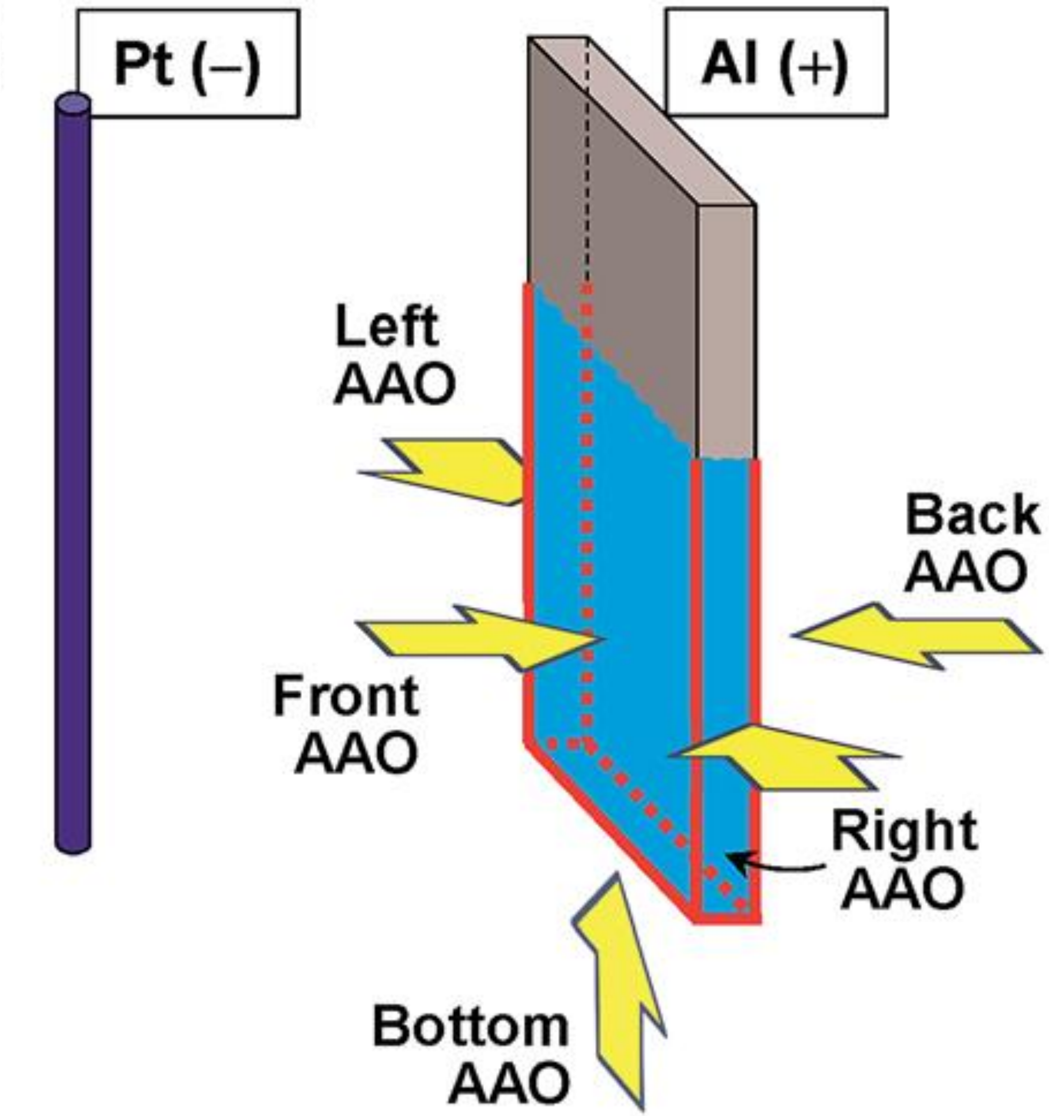






3.3. Synthesis of freestanding porous alumina template

- Freestanding alumina (**AAO**) was obtained using the detachment of porous alumina from the Al plate.
  Stair-like reverse biases applied after 2nd anodization as an environment-friendly method (Hong et al 2015).
- 0.3M oxalic acid - chosen as electrolyte for anodization process, as it allows synthesis of **smoother and more uniformly arranged pores and walls.**



(Hong *et al.* 2015)

3.3. Synthesis of freestanding porous alumina template

Optimized thick AAO template

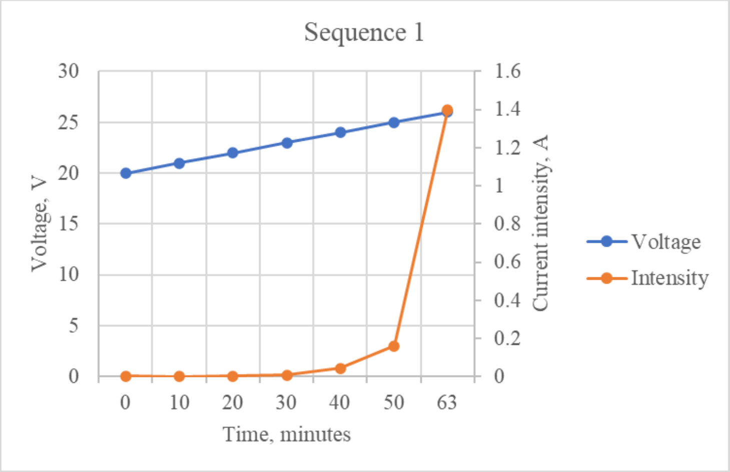
Conditions:

Anodization process

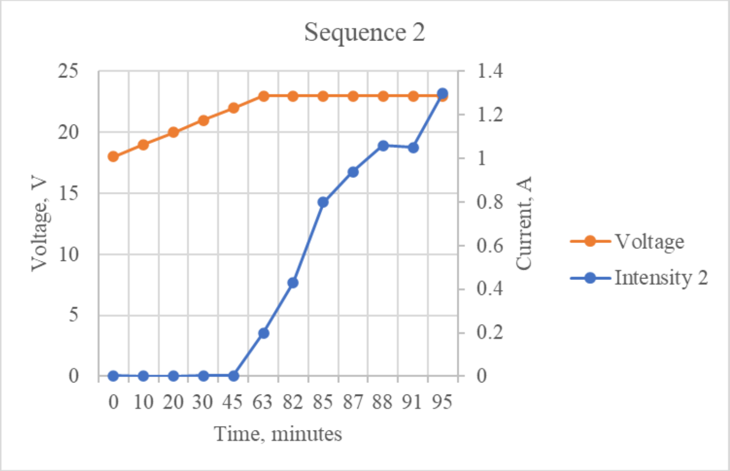
0.3 M oxalic acid $H_2C_2O_4$ electrolyte, 40V. Cathode: cylindrical electrode Ti covered with Pt.

Alumina detachment

The electrodes are changed:
- aluminum plate becomes the cathode (-)
- Ti/Pt counter electrode becomes the anode(+)
- Stair-like reverse biases are applied starting at 18V to 26V.

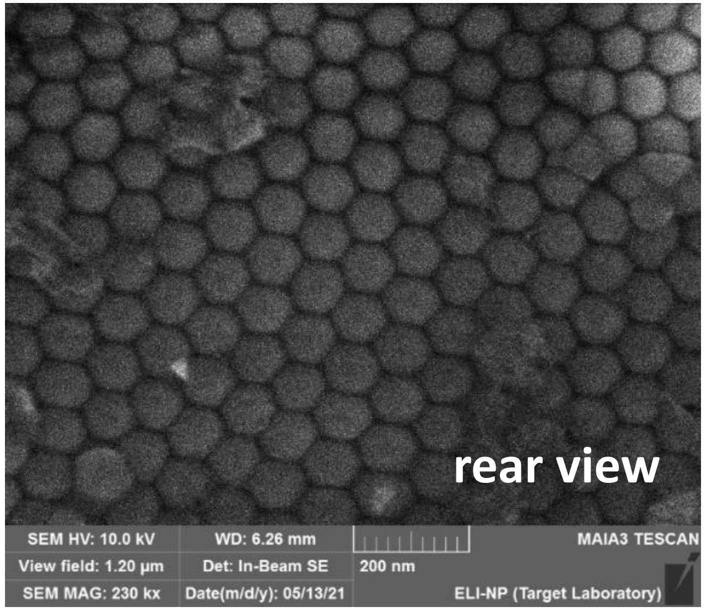


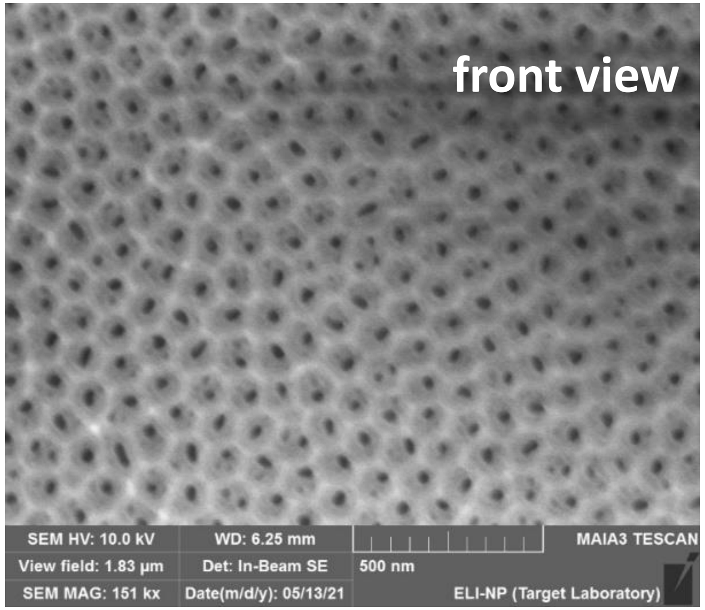
	First anodization		Second anodization		
Sequence	Time, h	Temperature, C	Time, h	Temperature, C	**AAO thickness, µm**
1	6	10	11.5	10	60-63
2	6	15	2	15	7-8
3	6	15	3	15	20-23

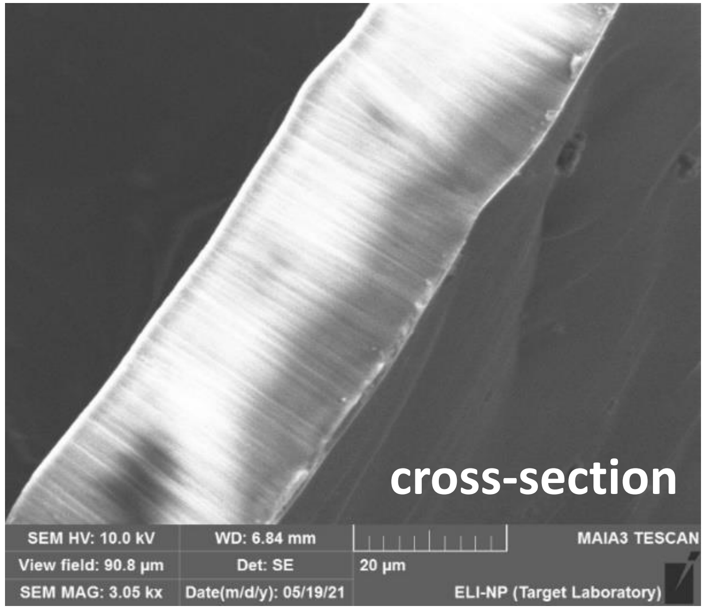


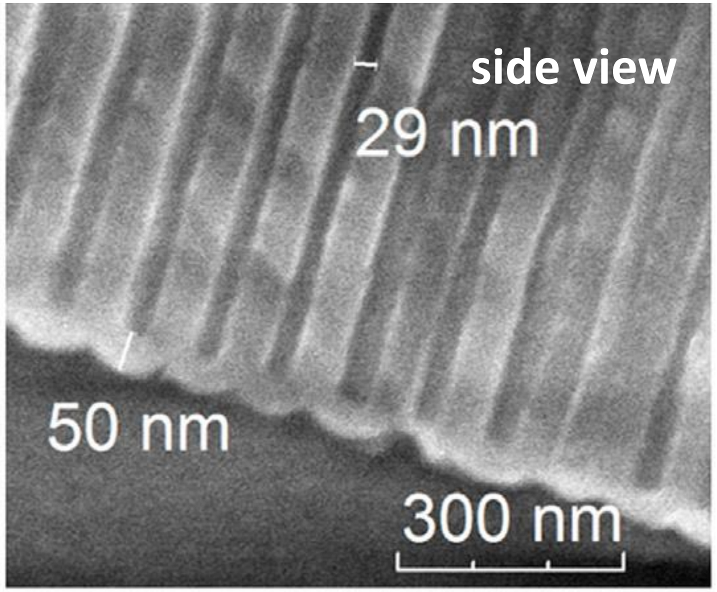
3.3. Synthesis of freestanding porous alumina template

Optimized thick AAO template

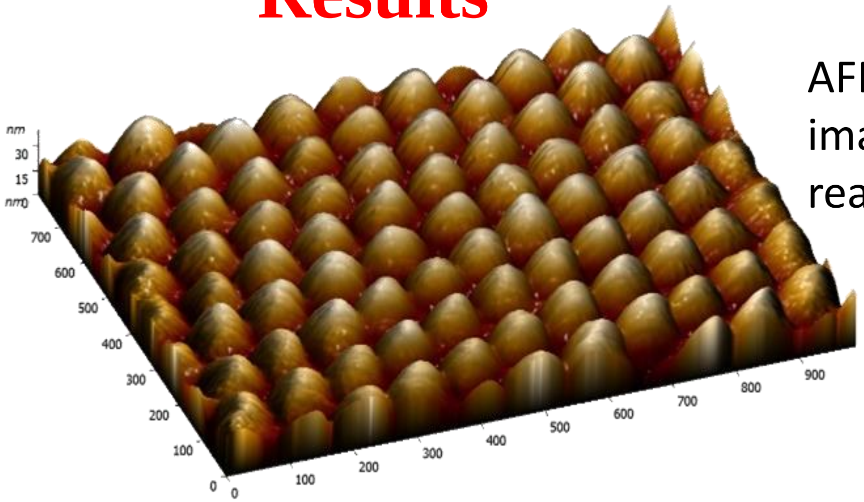




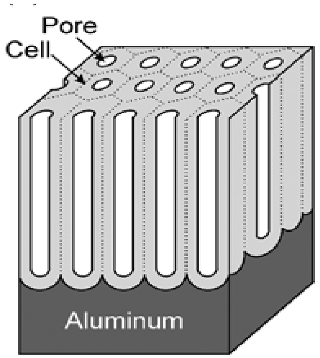


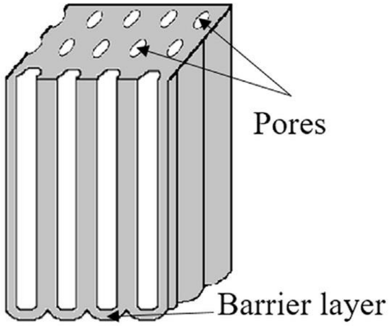


Results

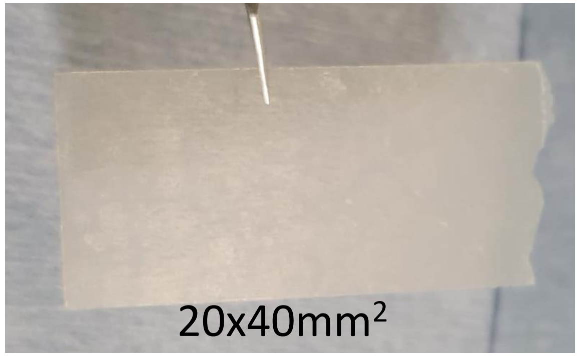


AFM 3D image - rear view

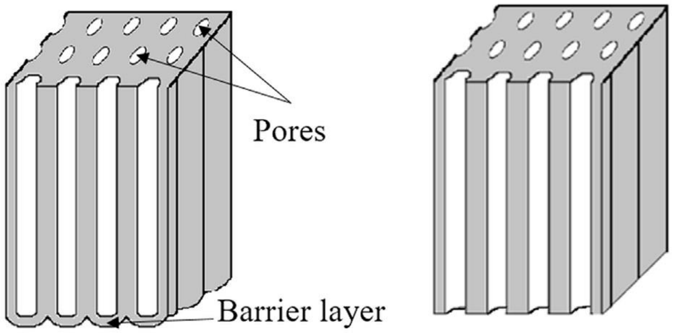




Closed bottom



Open pores on both sides

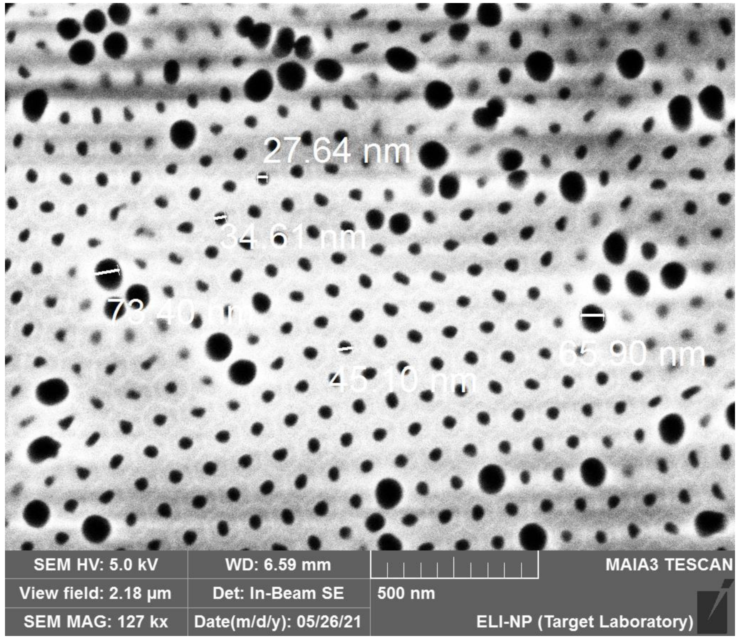


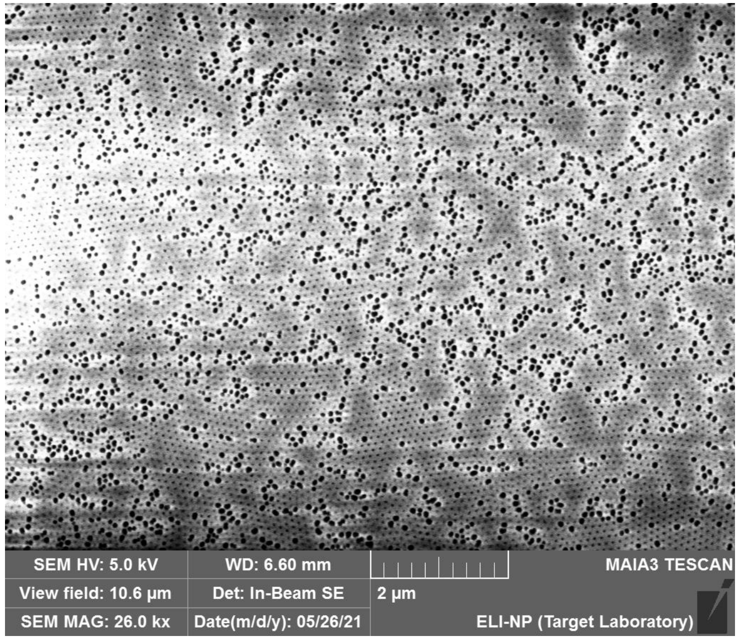
Closed bottom

Open bottom

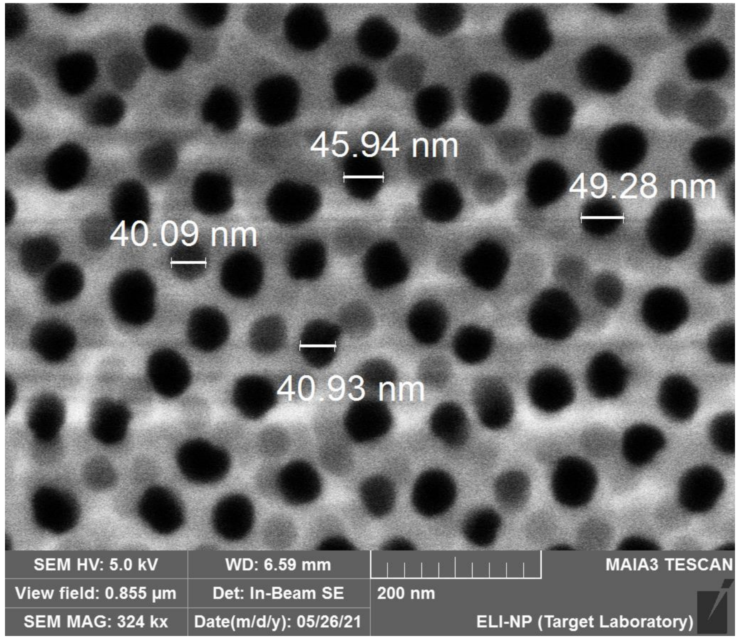
Chemical etching in:
-NaOH 2M
-H_3PO_4 0.5M

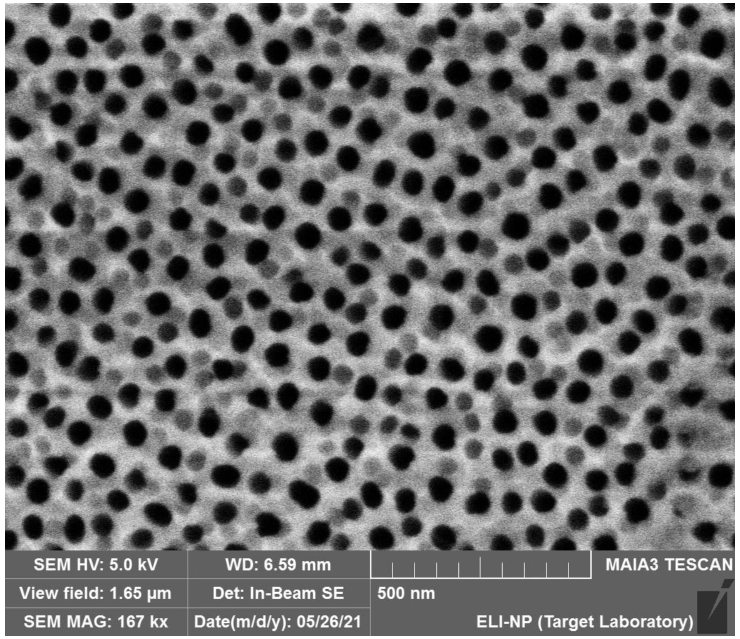
Open bottom pores



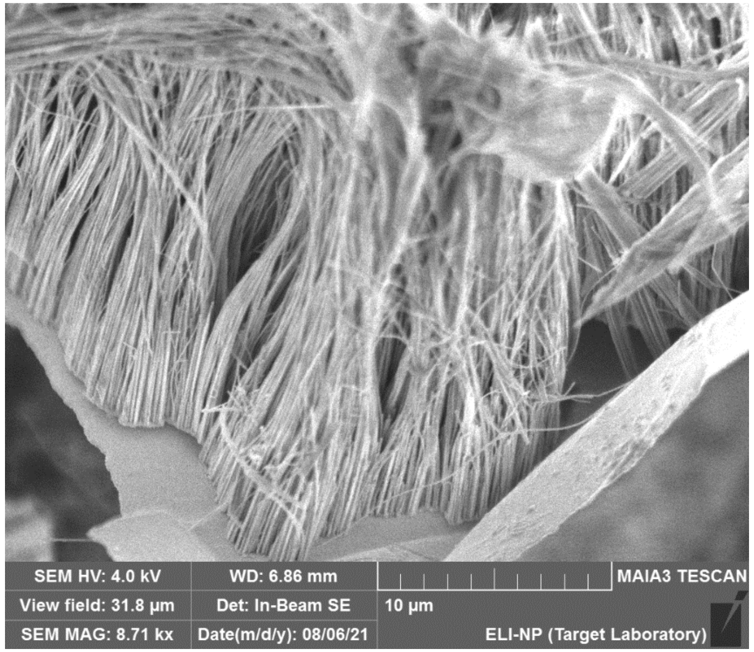


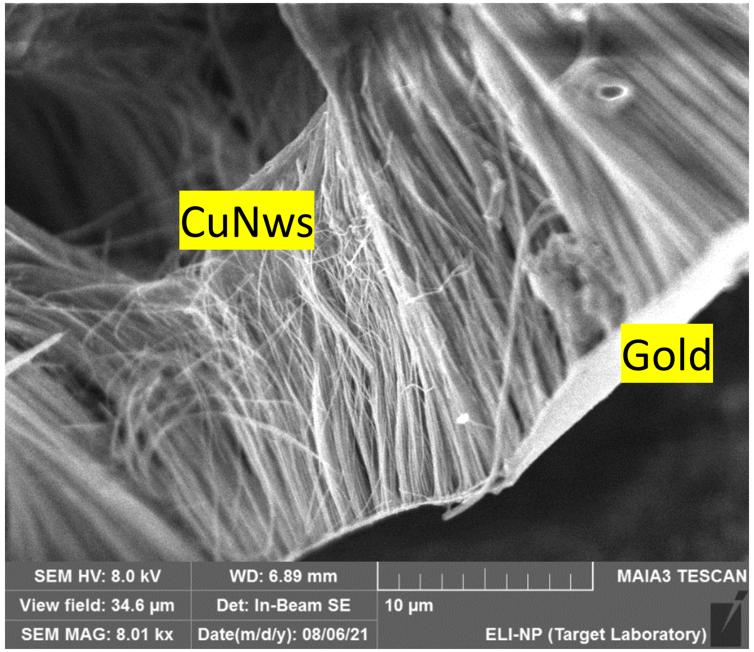
Top pores

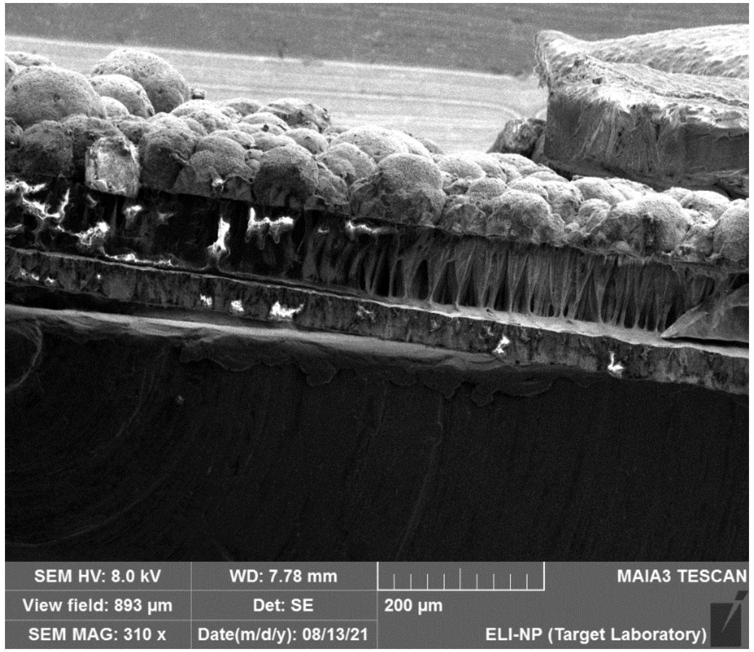


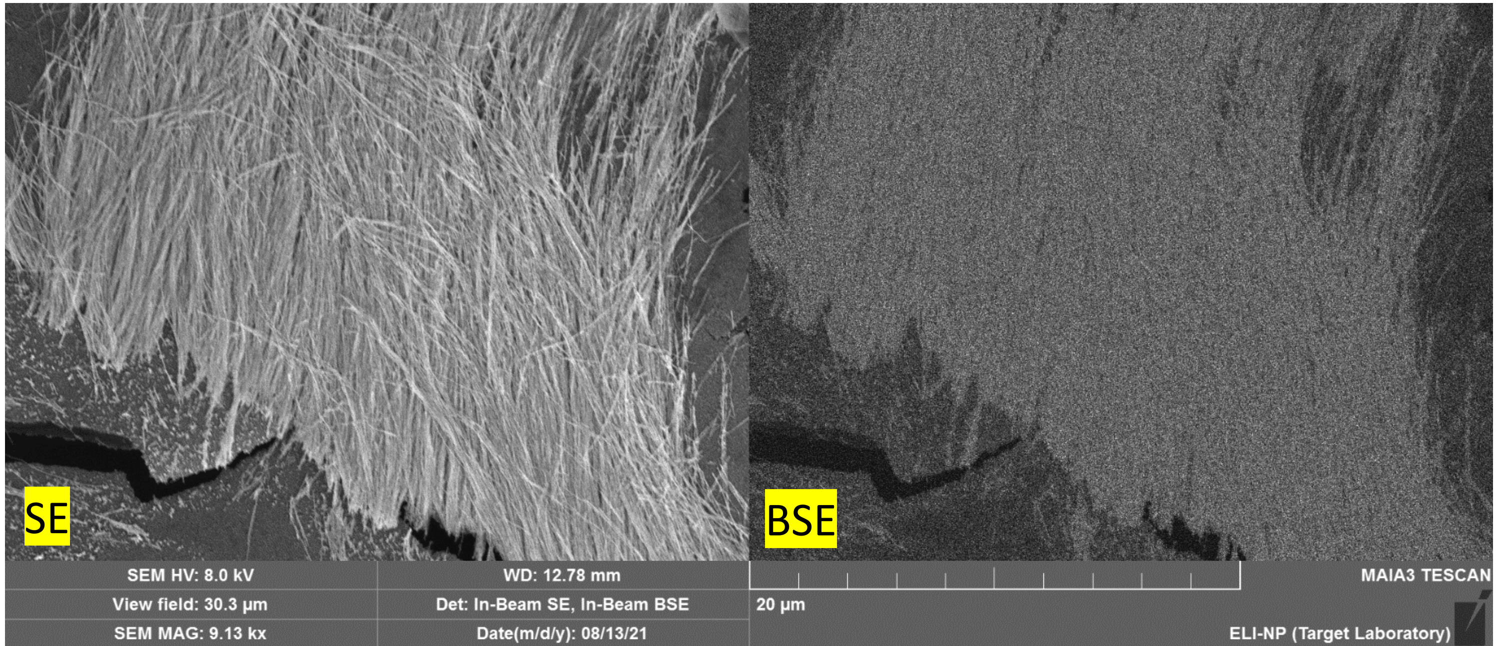


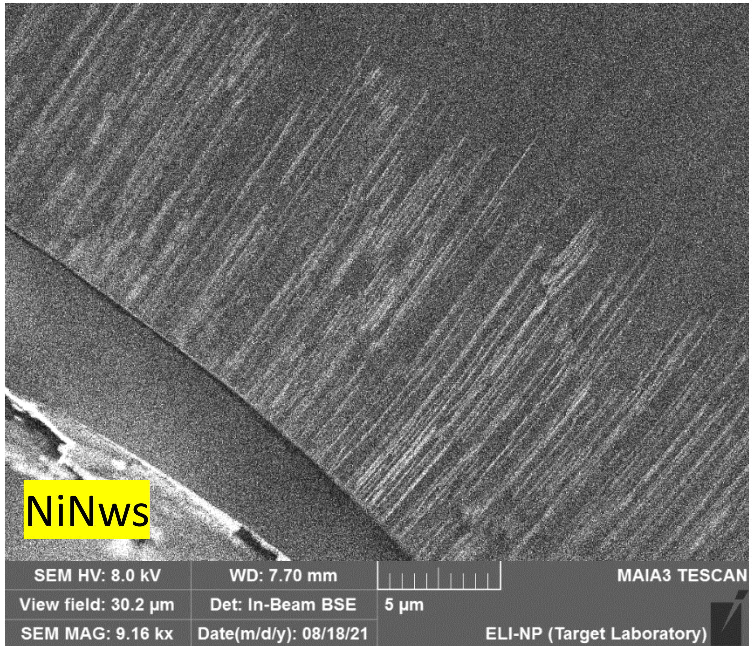
Freestanding copper nanowires On 150nm gold layer



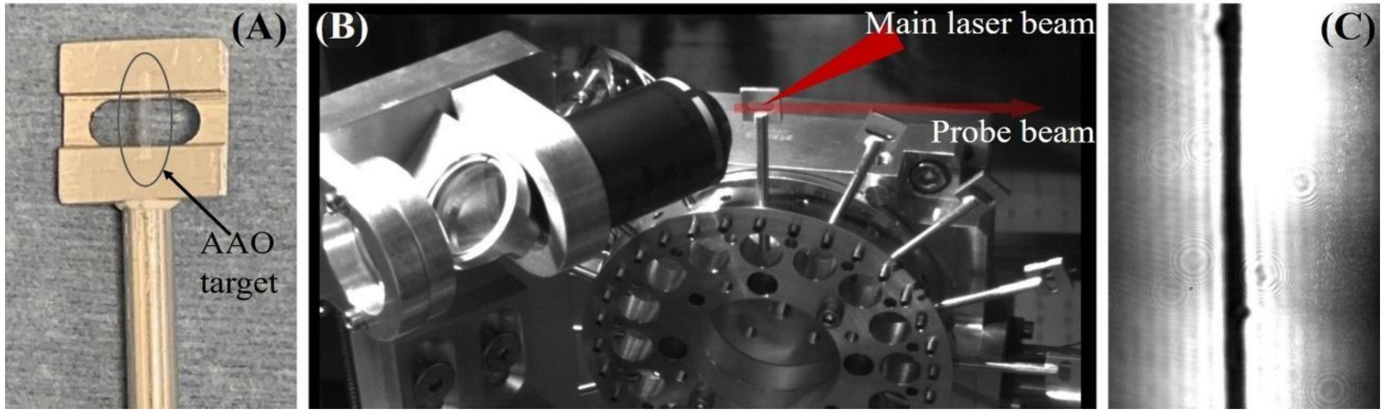






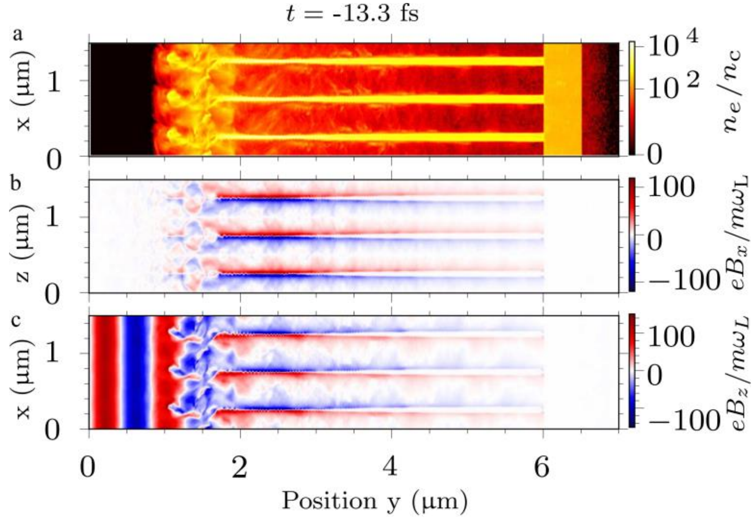


Laser Experiments and PIC Simulations



(A) Front side of the free-standing AAO mounted on a C-shaped Al-frame. (B) Experimental setup with target wheel and online imaging system used for laser focal spot optimization and target alignment. (C) Shadowgraph image of a 20 μm-thick AAO target.

See J.F.Ong presentation Today at 15:00



AAO targets

Free-standing AAOs mounted on a frame, prepared for the experimental setup.

* In **collaboration** with D.Doria, P.Ghenuche, M.Cernaianu

Nanowires Characteristics
Diameter: 40-500 nm
Height: 500 nm- tens of micrometers
Interpore distance: 40-100 nm
Material: **Cu, Ni, Co, Au** (Pd, Ag, Zn, Nd, Tb, combinations)

Acknowledgements

Team:

- Dr. Cristina Gheorghiu
- Dr. Victor Leca
- Daniel Popa
- Leonard Floarea
- Dr. Mihai Cuciuc
- Dr. Petru Ghenuche
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Thank you!

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