Fabrication of metallic nanowire targets for high power laser experiments

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Young Researcher and Young Engineer Days

10-12 January 2022

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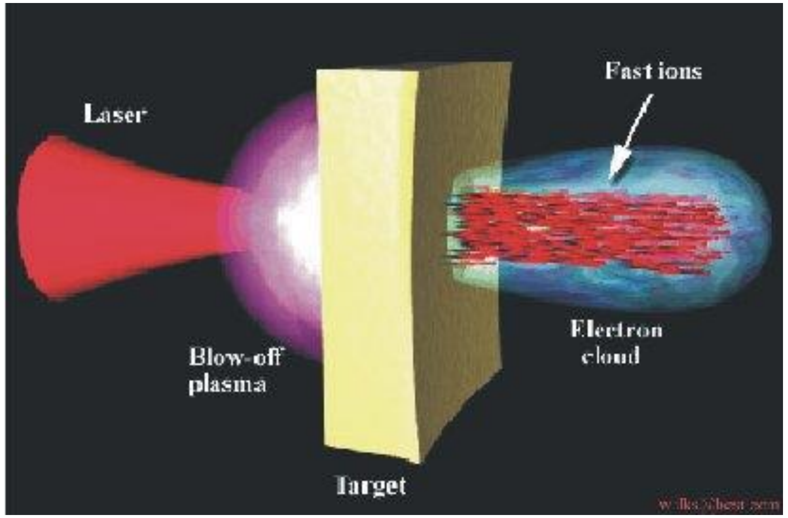
Short introduction

Laser Targets



E. Brambrink et al, Proceedings of EPAC, 2002

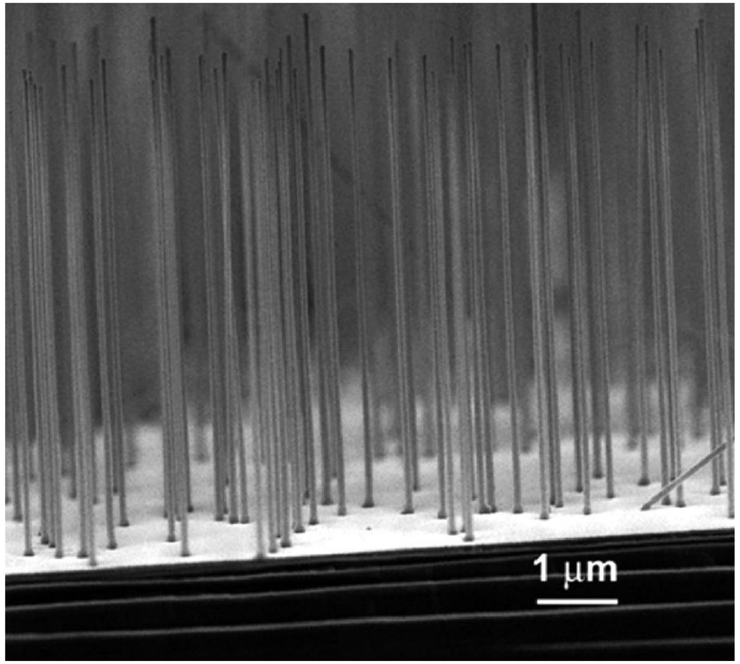
Types of targets:

- 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)

→ investigation of light-matter interaction phenomena *(particle acceleration, dynamic compression, radiation source...)*

→ structures with high aspect ratio with unique **magnetic, optic and electric properties**



R. Elnathan et al, Nano Today, 2014

NW targets for laser experiments may increase:

✓ **Maximum resulted particle energy**

✓ **Laser absorption on the target**

✓ **Conversion efficiency**

A. Macchi et al, Review of modern physics, 2013

C. Bargsten et al., Science Advances, 2017

S. Vallieres et al, Nature Scientific Reports, 2021

Methods for NW synthesis

Bottom-Up

- Vapor-Liquid-Solid
- Chemical Vapor Deposition
- Hydrothermal
- Molecular Beam Epitaxy
- Electrodeposition

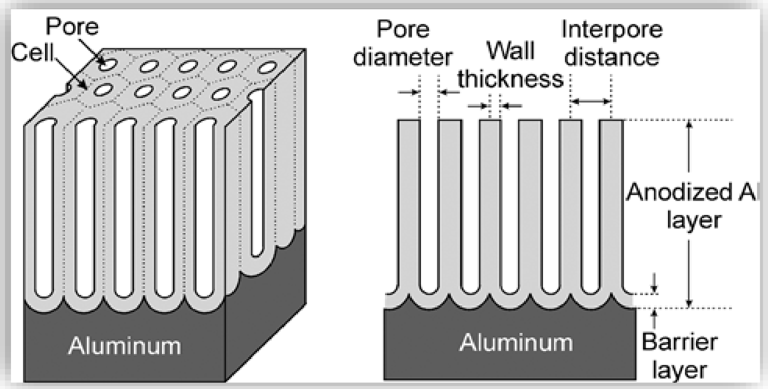
→ **Advantages**:
- wide range of tunable parameters
- low-cost equipment and materials

Top-Down

- Reactive Ion Etching
- Metal Assisted Chemical Etching
- Lithography

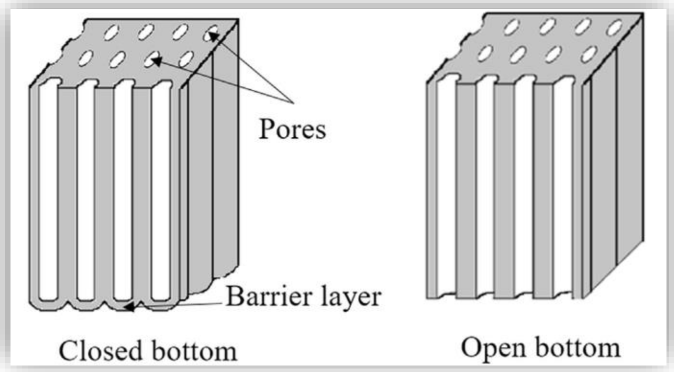
Anodization process

- Al anodization transforms Al in porous alumina, in an electrochemical cell.

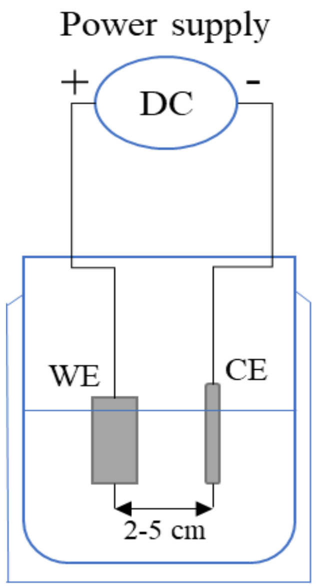


G. Sulka, Nanostructured Materials in Electrochemistry, 2008

- Free-standing alumina: by detaching the anodized layer from the aluminum substrate.



C. Gheorghiu et al., Frontiers in Physics, 2021



Electrochemical cell

Reactions for the anodization of Al in **phosphoric acid** electrolyte:

- **Electrolyte reactions:**

$H_2O + PO_4^{3-} \rightarrow OH^- + HPO_4^{2-}$

$H_2O + PO_4^{3-} \rightarrow O^{2-} + H_2PO_4^-$

- **Cathodic reactions:**

$6H^+ + 6e^- \rightarrow 3H_2$

$2H_2O + 2e^- \rightarrow 2OH^- + H_2$

- **Anodic reactions:**

Aluminum dissolution: $2Al \rightarrow 2Al^{3+} + 6e^-$

Metal/ Oxide interface: $2Al + 3O^{2-} \rightarrow Al_2O_3 + 6e^-$

$2Al + 6OH^- \rightarrow Al_2O_3 + 3H_2O + 6e^-$

Oxide/ Electrolyte interface: $2Al^{3+} + 3H_2O \rightarrow Al_2O_3 + 6H^+$

Overall anodization equation: $2Al + 3H_2O \rightarrow Al_2O_3 + 3H_2$

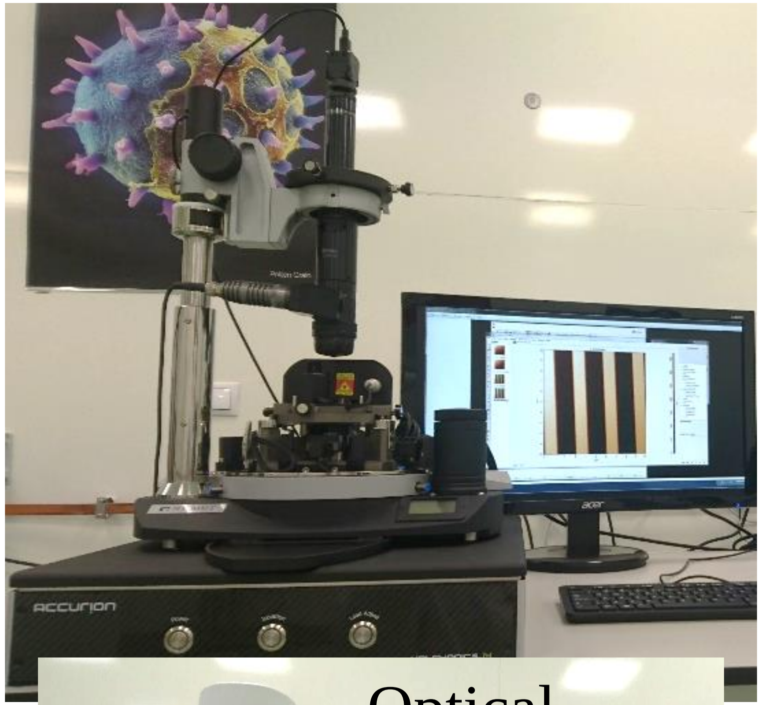
G. Absalan et al, Materials Chemistry and Physics, 2017

Electrochemical set-up

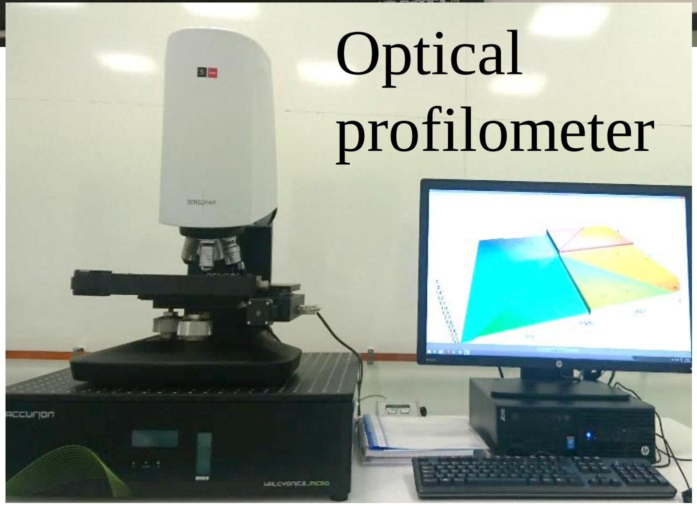
Electrochemical set-up used for aluminum anodization includes a double wall glass electrochemical cell connected to a chiller, a DC power supply, 2 bench-top multimeters for current and voltage monitoring, connected to a PC interface, and a stirring and heating plate. For pulsed electrodeposition, an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

Characterization techniques

Atomic force microscopy (AFM)

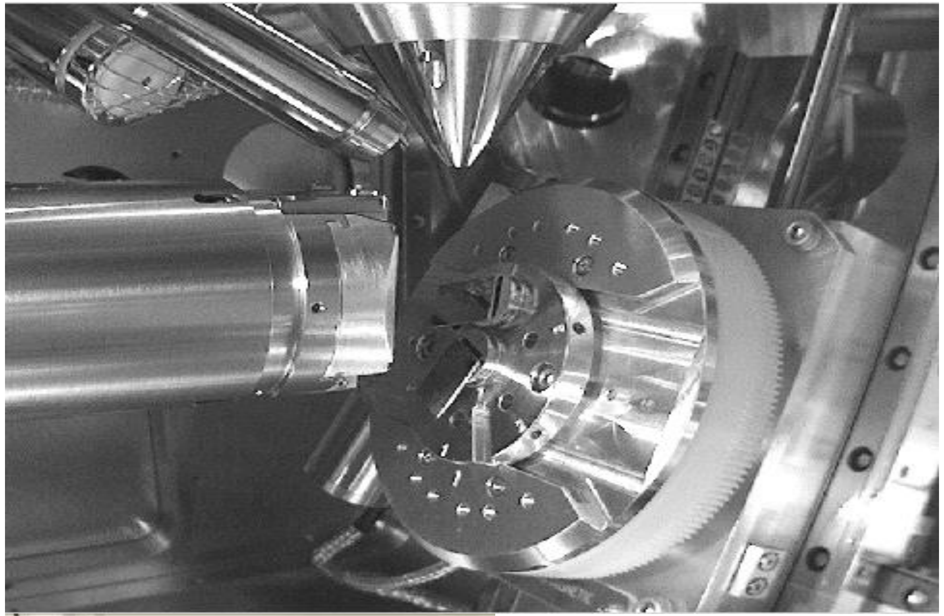


Optical profilometer



Scanning electron microscopy (SEM)

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



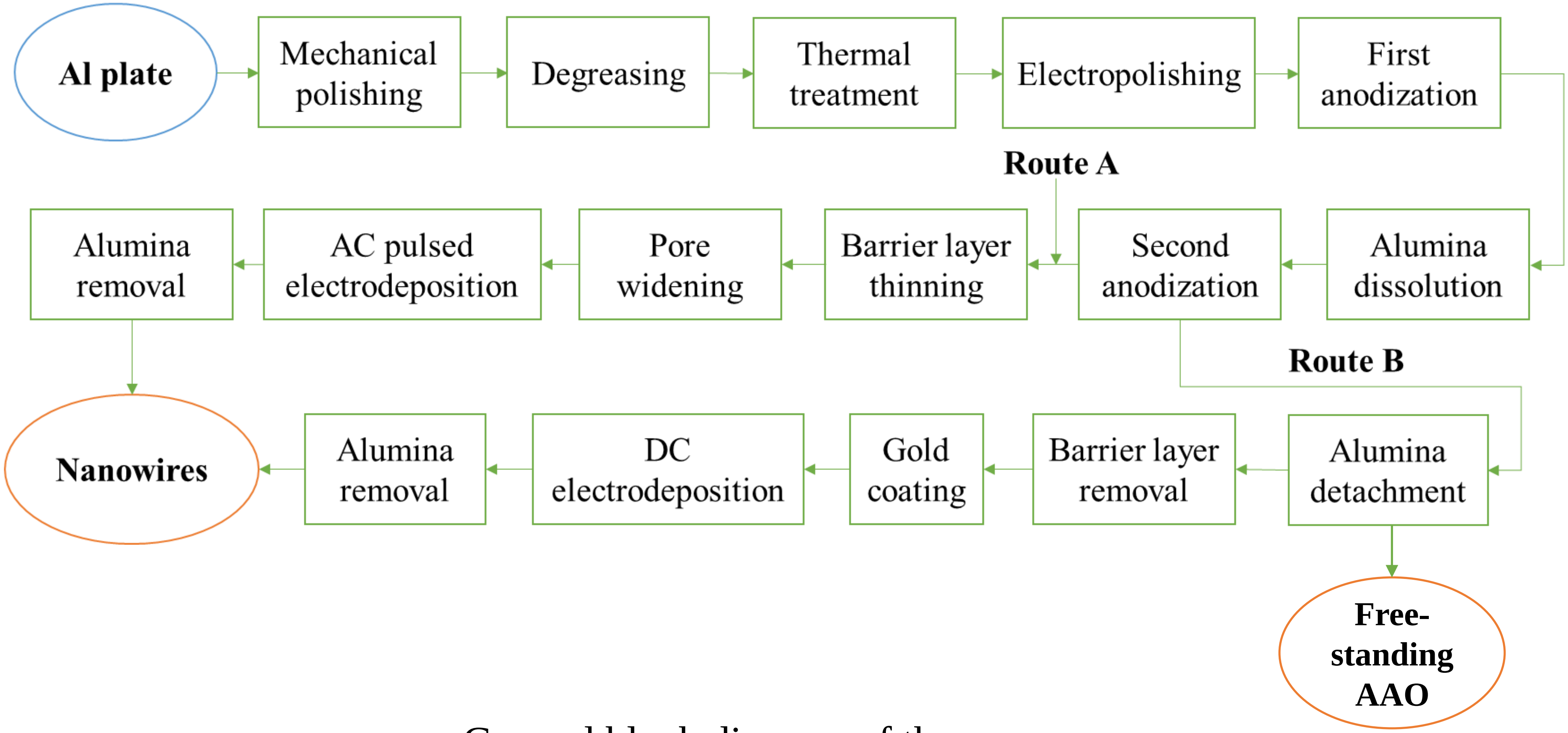
Optical microscope

Results (on-going research)

The work is focused on two routes:

A) NWs on aluminum substrate synthesized by AC pulsed electrodeposition;

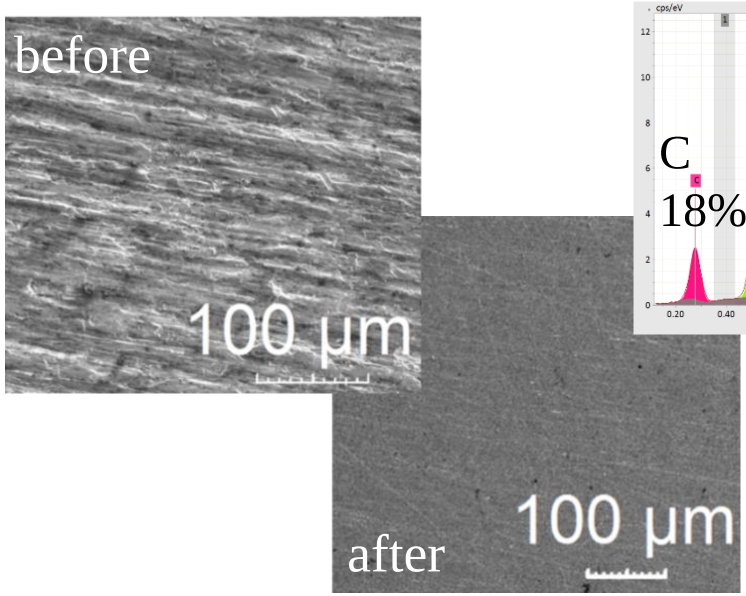
B) freestanding AAO (anodic aluminum oxide) by electrochemical detachment & free-standing NWs by DC electrodeposition.

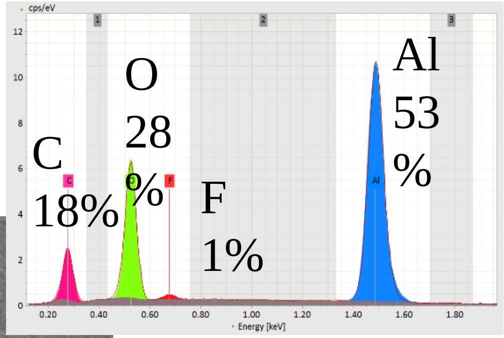


General block diagram of the process

Aluminum foils pre-processing

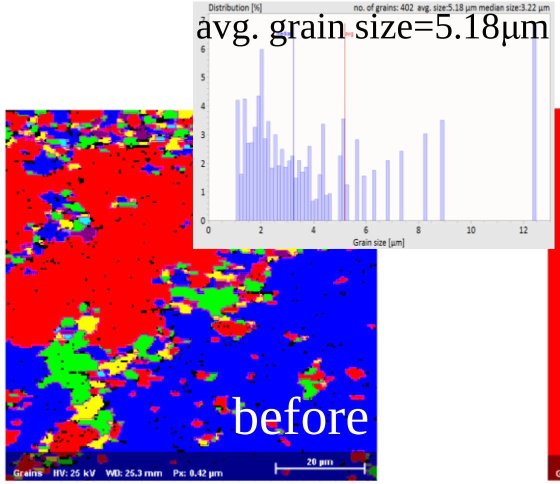
Mechanical polishing

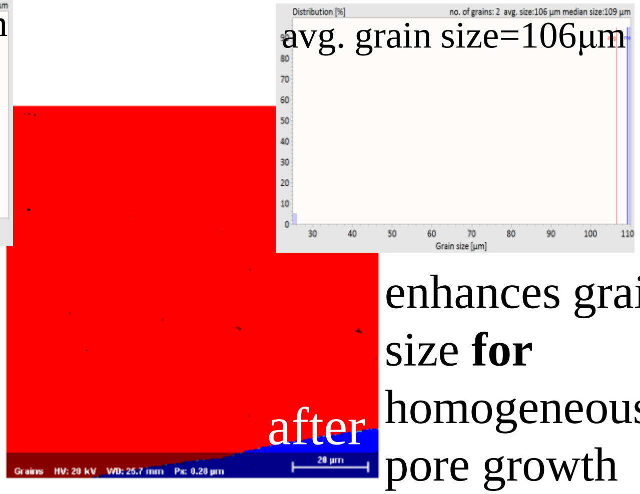




reduces roughness;
but increases
surface contaminants

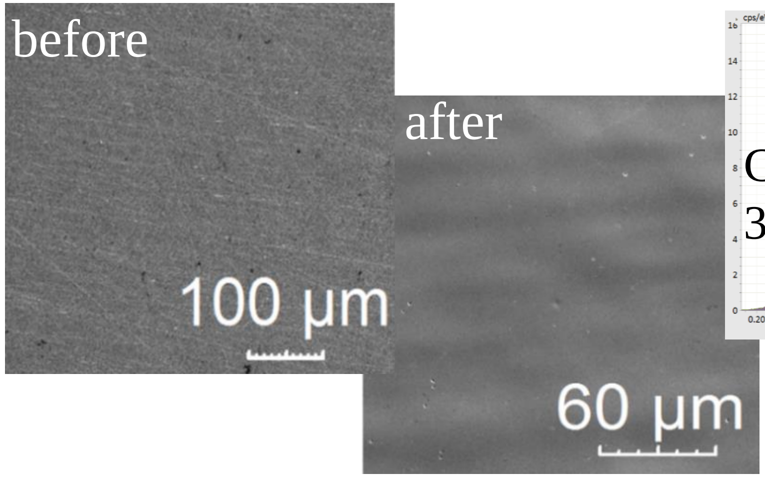
Thermal treatment

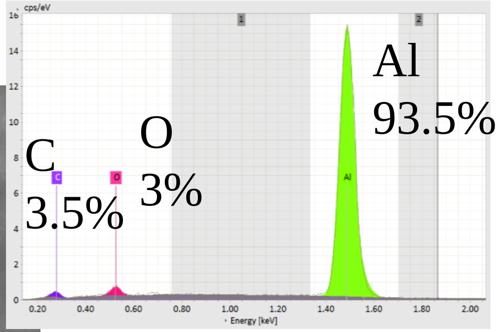




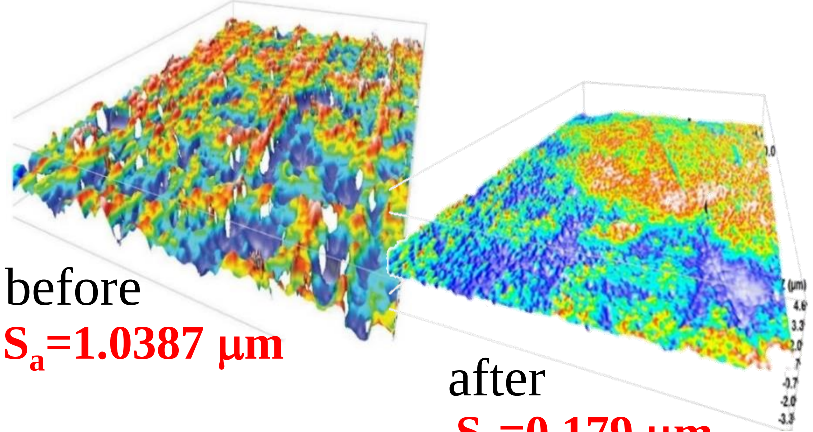
enhances grain
size **for**
homogeneous
pore growth

Electropolishing





improves roughness;
reduces surface contaminants



before
S_a=1.0387 µm

after
S_a=0.179 µm

Synthesis of porous AAO and copper NWs on Al substrate

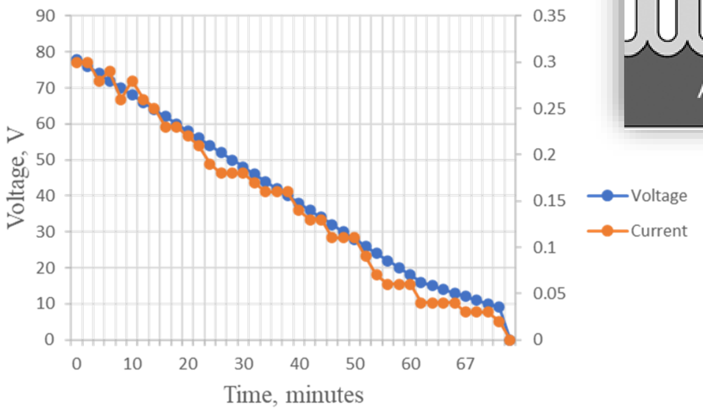
One-step anodization and pulsed AC electrodeposition

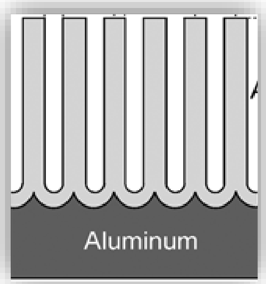
Conditions:

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





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.

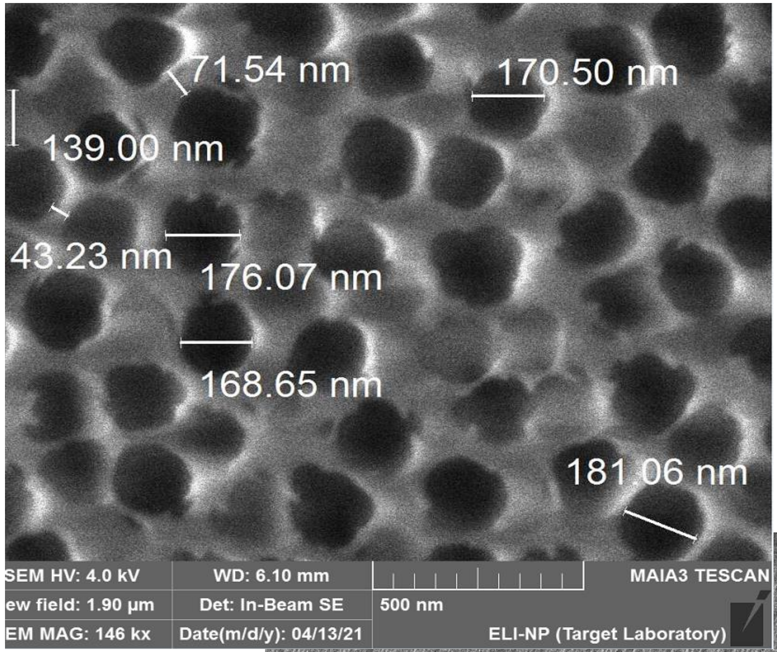
Alumina dissolution

Liberation of NWs: 5 min, 1M NaOH at 40C.

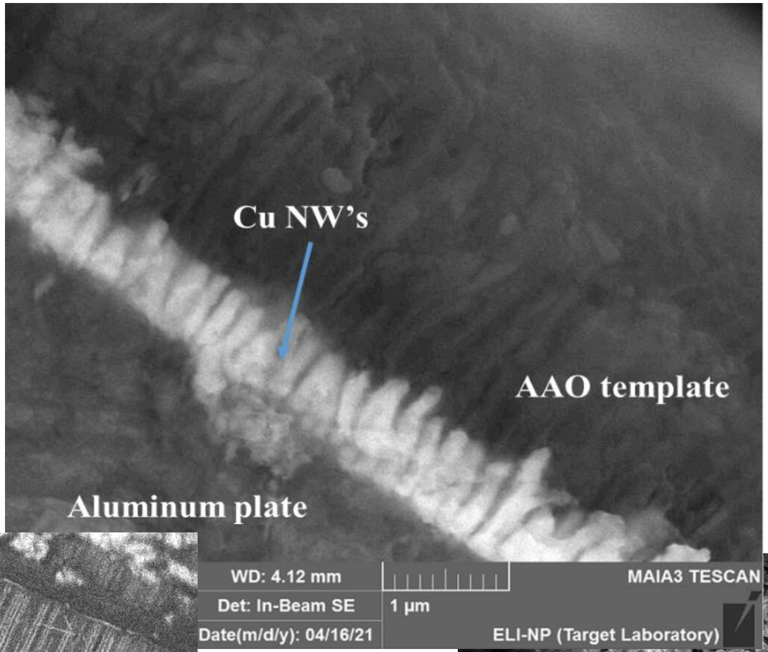
Synthesis of porous AAO and copper NWs on Al substrate

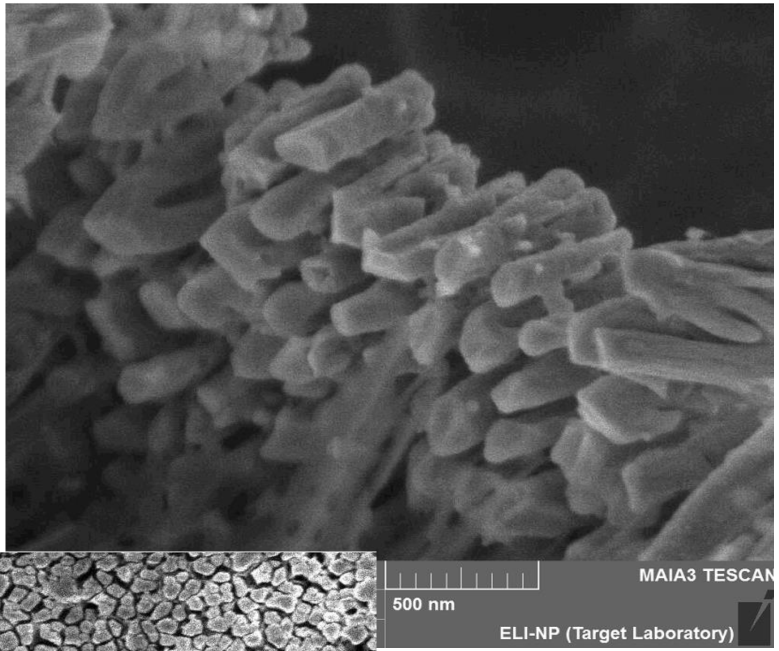
One-step anodization and pulsed AC electrodeposition Results

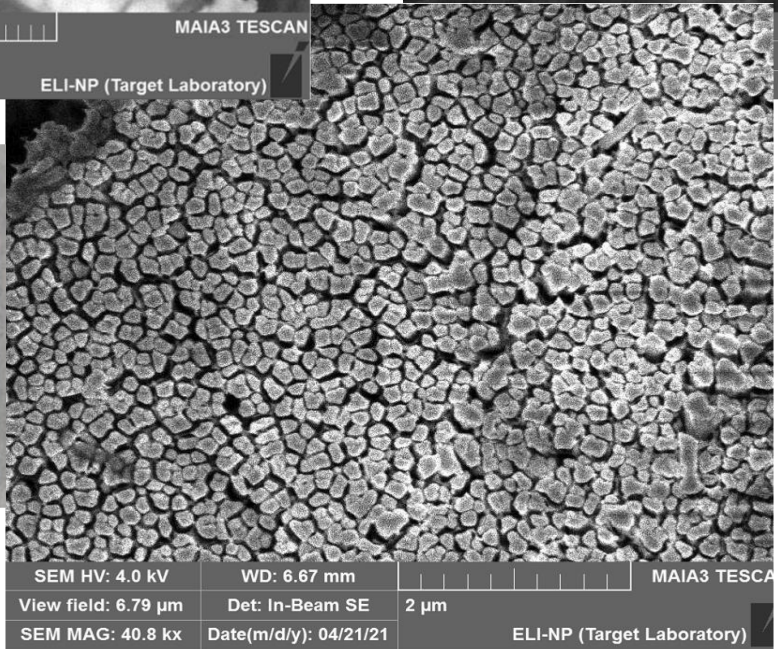
Anodization



Pulsed AC electrodeposition and dissolution







Synthesis of free-standing porous AAO template

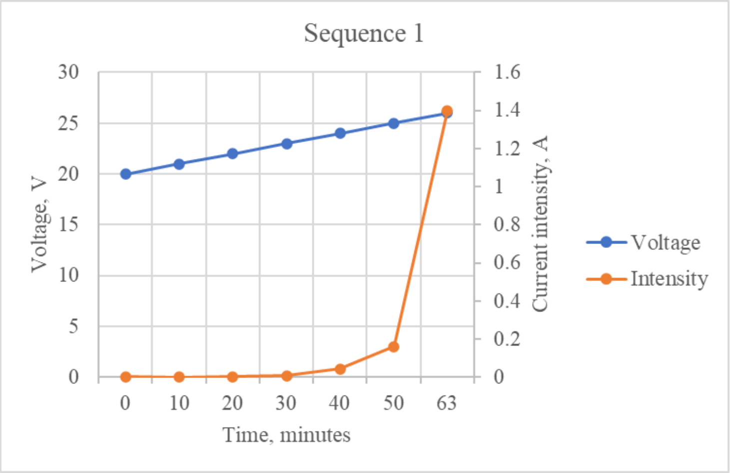
Conditions:

Anodization process

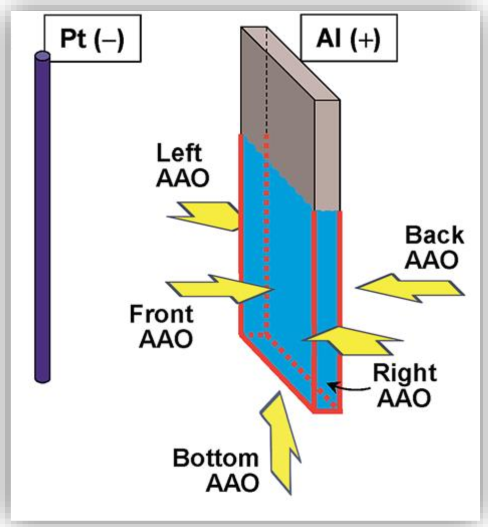
0.3 M **oxalic acid** $H_2C_2O_4$ electrolyte, **40V**. Cathode: cylindrical electrode Ti covered with Pt.

Alumina detachment

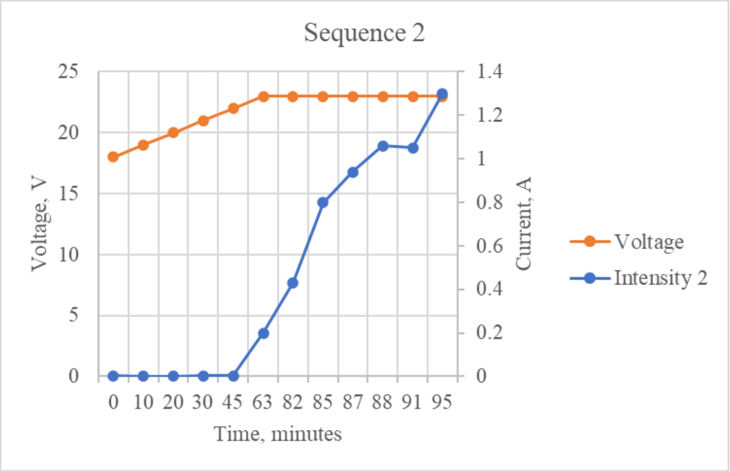
The electrodes are changed:
- aluminum plate becomes the cathode (-)
- Ti/Pt counter electrode becomes the anode(+)
- Stair-like reverse biases are applied starting at 18V up to 26V.



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

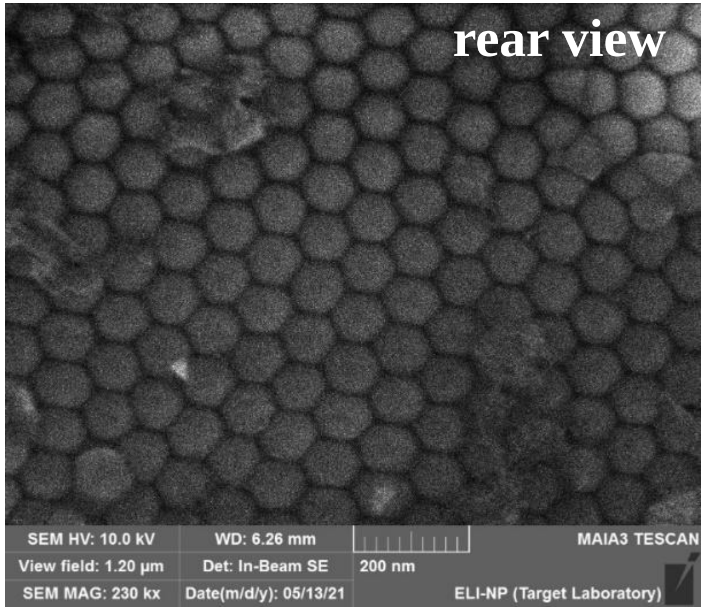


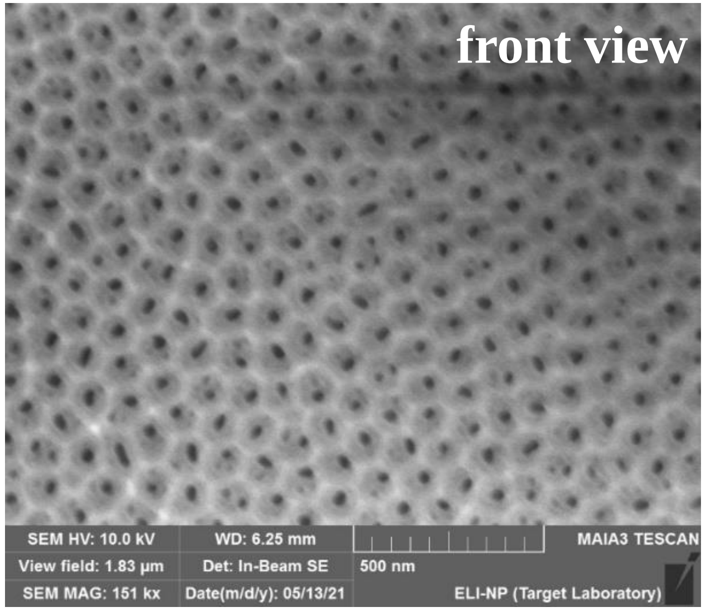
Y. Hong et al, RSC Advances, 2015

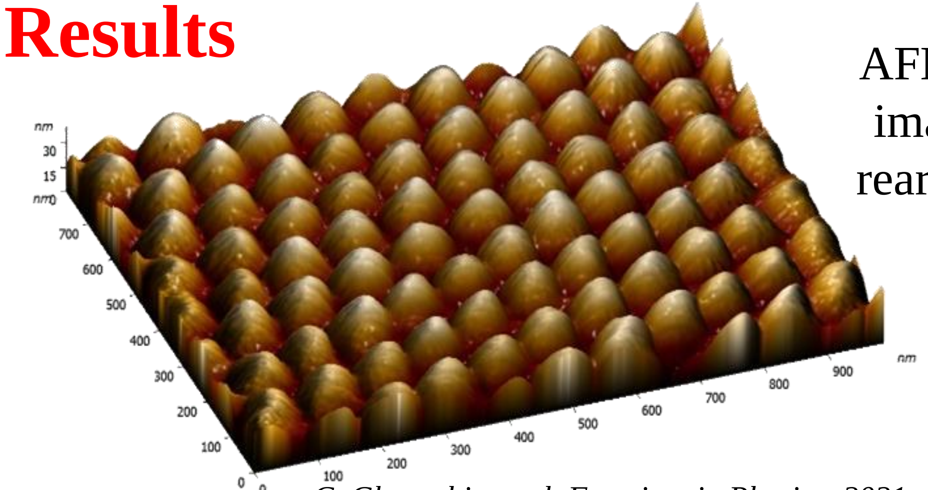


Synthesis of free-standing porous alumina template

Optimized thick AAO template Results

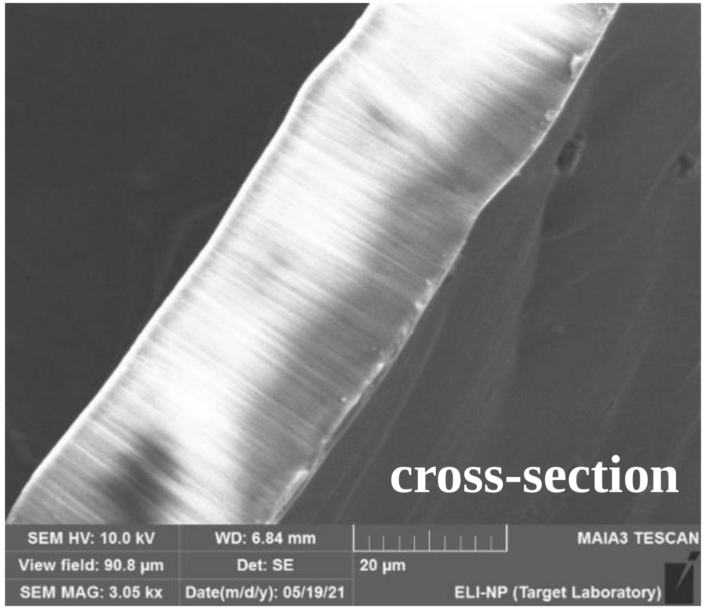


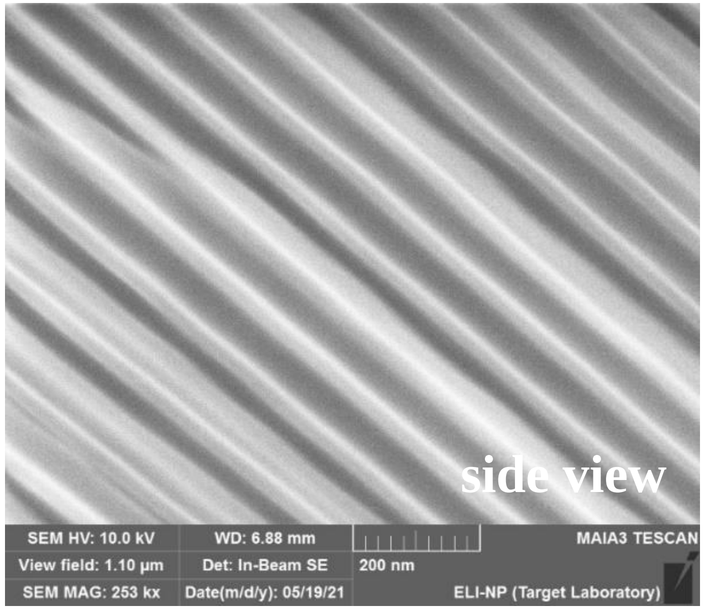


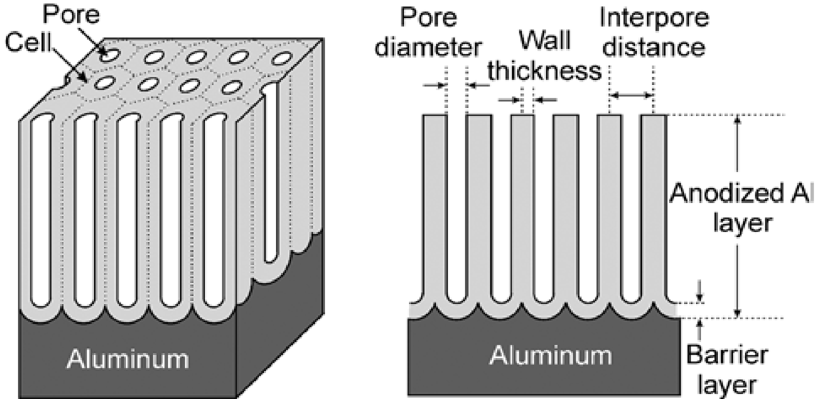


AFM 3D image - rear view

C. Gheorghiu et al, Frontiers in Physics, 2021



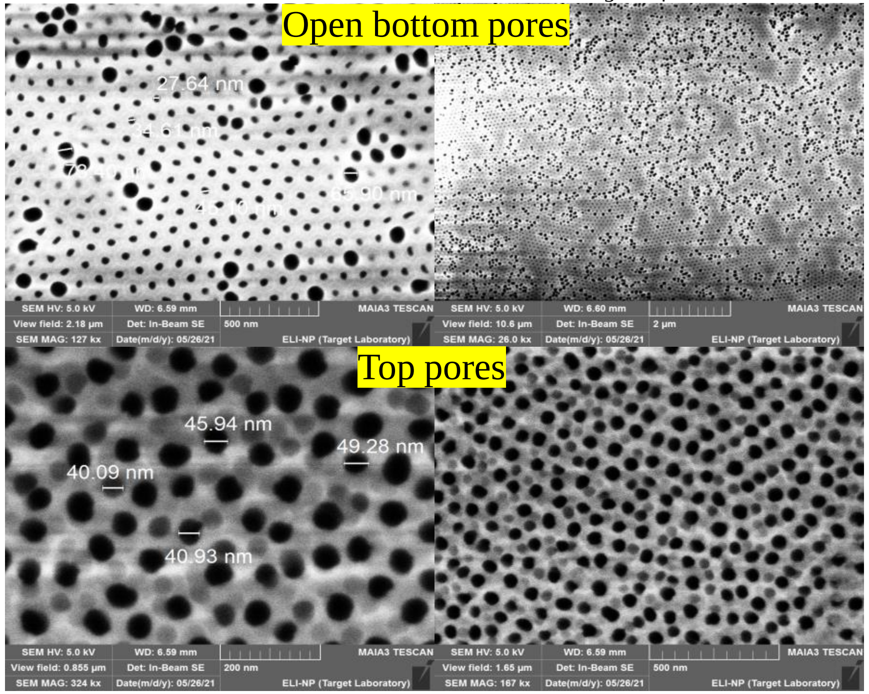




Free-standing copper nanowires by DC electrodeposition

Open pores on both sides

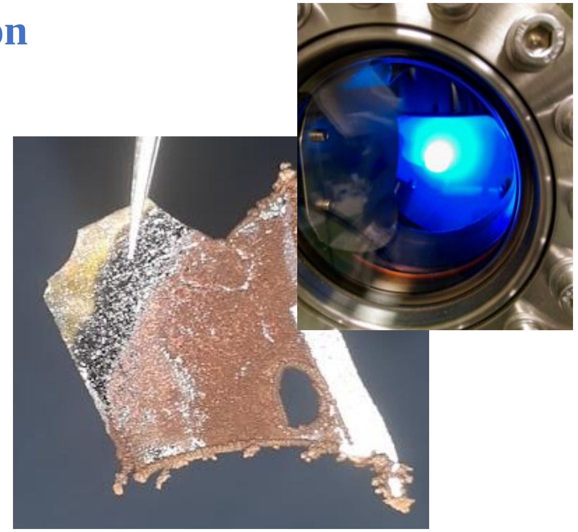
Chemical etching in:
-NaOH 2M
-H_3PO_4 0.5M

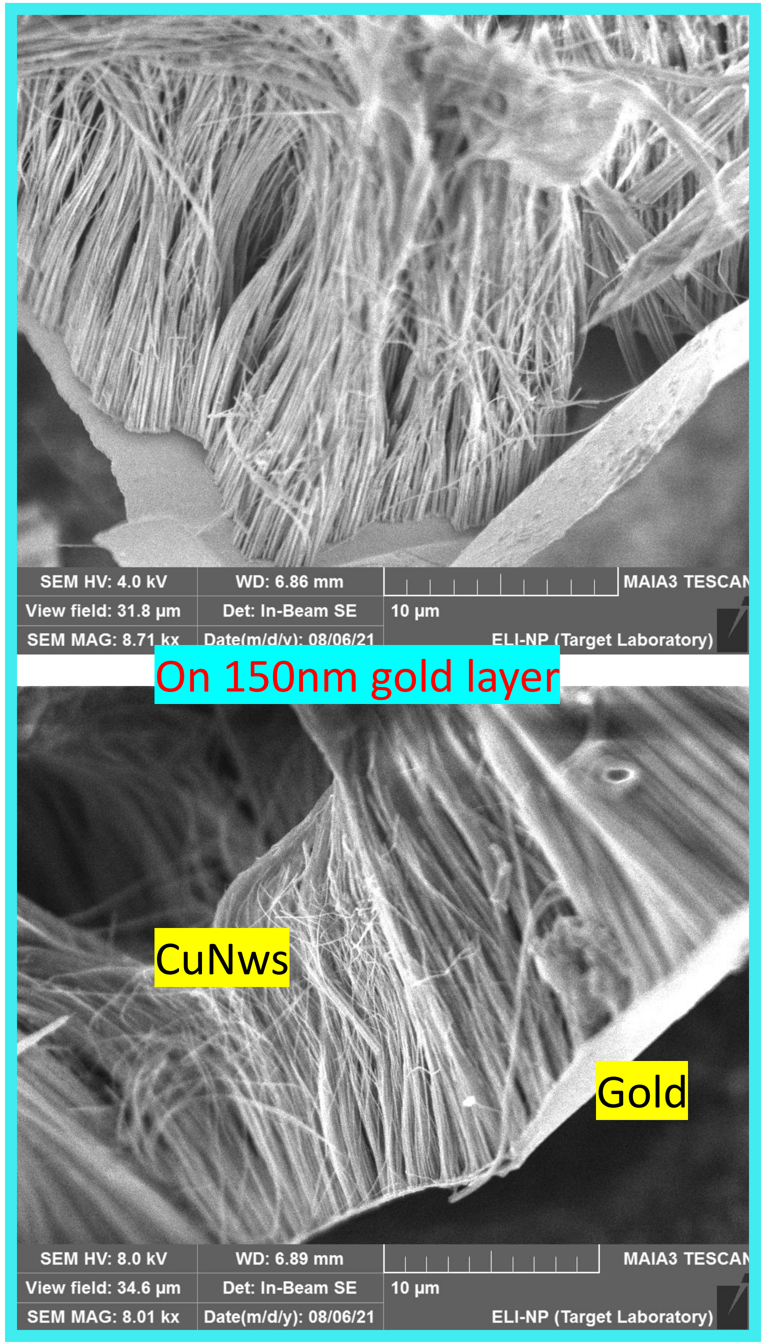


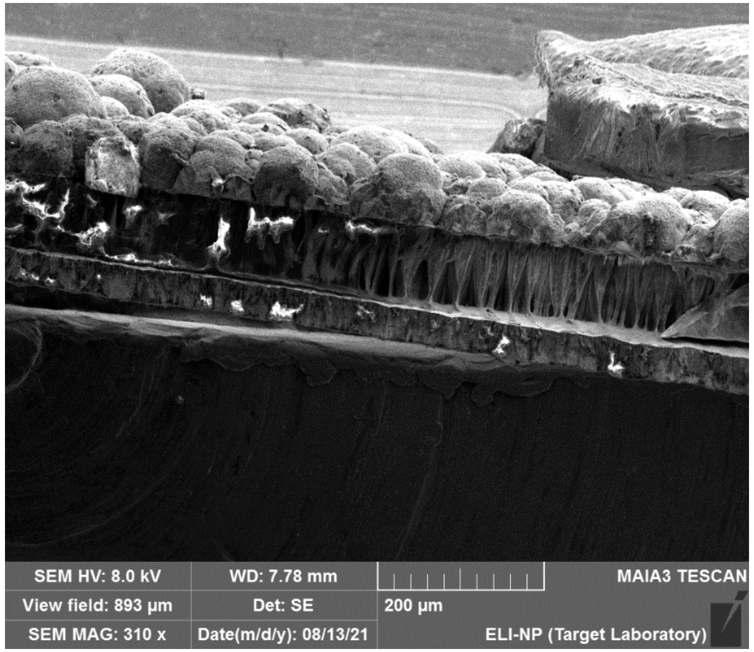
DC Electrodeposition

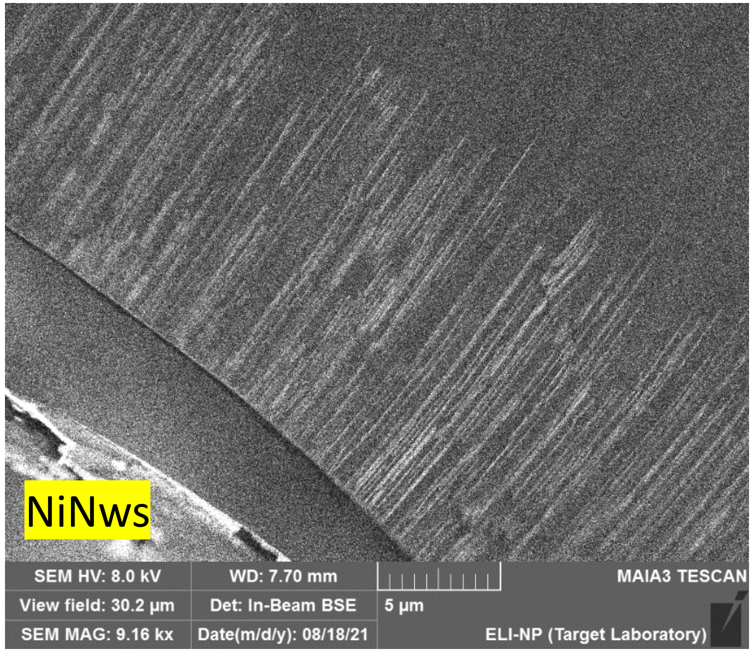
Conditions:

- W.E. (AAO) -
- C.E. (Cu) +
- 0.5M $CuSO_4$+H_3BO_3
- pH 3-3.5
- 1-4 V
- 1-2 h

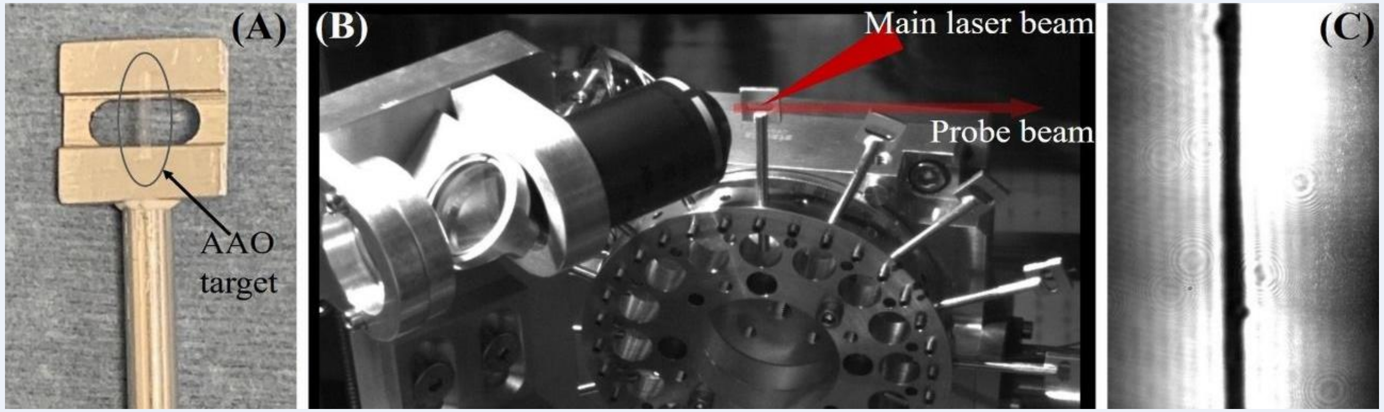








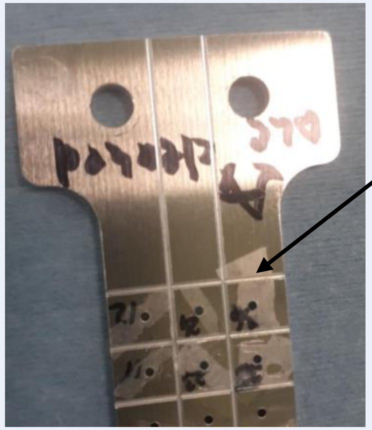
Preliminary laser experiments

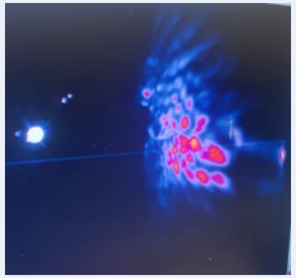


(A) Front side of the free-standing AAO mounted on a C-shaped Al-frame. (B) Experimental setup with target wheel and online imaging system used for laser focal spot optimization and target alignment. (C) Shadowgraph image of a 20 µm-thick AAO target.

C. Gheorghiu et al., Frontiers in Physics, 2021

AAO targets





First shots with plasma mirror:

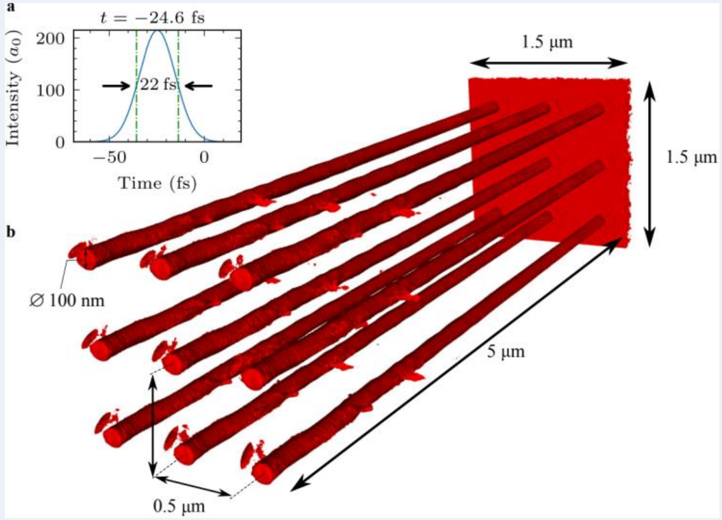
2x AAO 19 µm: open TOP / both ends open

Laser pulse duration 23 fs FWHM

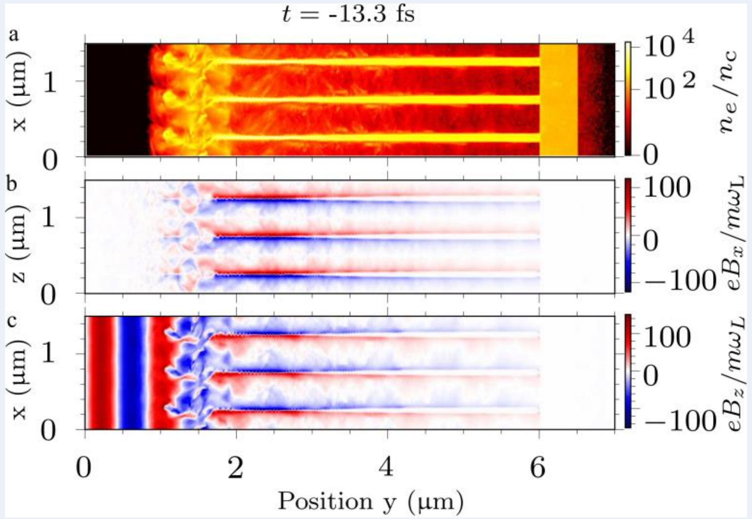
Beam intensity 1e21 W/cm^2

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

PIC Simulations



* In **collaboration** with J.F. Ong, A. Berceanu



Conclusions

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)

The following has been achieved:

- ✓ Obtaining free-standing alumina of 7-100μm thickness
- ✓ Synthesis of Cu NWs on Al substrate & on Au thin film
- ✓ Laser experiments & PIC Simulations (ongoing)

Regarding further work, next directions are of interest:

- ➢ Synthesis of AAO <5 μm thick
- ➢ Freestanding metallic NWs (Co, Ni, Au, Pd, or mix)
- ➢ Si NWs by RIE+ EBL (Top-Down method)
- ➢ Target testing in high power laser experiments
- ➢ PIC simulations for NWs & AAO targets

Acknowledgements

Team

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- Dr. Petru Ghenuche
- Dr. Mihail Cernaianu
- Dr. Domenico Doria
- Dr. Jian Fuh Ong
- Dr. Andrei Berceanu

Thank you!

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