

SYNTHESIS OF FREESTANDING POROUS ALUMINA AND METALLIC NANOWIRES BY ELECTROCHEMICAL METHODS

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Using electrochemical methods, porous alumina templates with thicknesses of tens of micrometers were synthesized from aluminum plates, on which copper nanowires were deposited, either on an aluminum substrate or on a thin gold film, to be used as targets for high-power laser experiments. Before the anodization process, the aluminum plates were treated by mechanical polishing to reduce the surface roughness to tens of nanometers, a process followed by thermal treatment for surface reconstruction. In order to obtain porous alumina with different characteristics (thickness, pore diameter, interpore spacing), the key process parameters (electrolyte solution composition, its temperature, and applied voltage) were optimized for the growth of nanostructures with a wide range of geometrical characteristics, which will allow the finding of a set of parameters that leads to an enhanced laser-target interaction. After the anodization process, the alumina film, obtained by detaching it from the aluminum substrate, has a thickness ranging from 7-100 μm , with a pore diameter of 15-400 nm, an interpore spacing of 30-100 nm and a density of 2-2.7 g/cm^3 . The synthesis of copper and nickel nanowires via two fabrication directions is presented: direct current electrodeposition on alumina, and pulsed alternating current electrodeposition on alumina grown on the aluminum substrate. At each stage of the fabrication process, the samples were characterized using electron microscopy.

Keywords: freestanding alumina, anodization, nanowires, electrodeposition, laser targets, laser-matter interaction

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1. Introduction

Extreme Light Infrastructure - Nuclear Physics is a research user facility with the scope of performing fundamental research (exotic nuclei and photo-fission,

vacuum properties and particle acceleration, nuclear structure and astrophysics studies) and applied research (materials for space science, management of nuclear materials, industrial tomography, brilliant positron source, radioisotopes for medical applications, among others). As equipment, two arms with 10 PW high-power laser system, commissioned, and a brilliant energy gamma-ray beam system, under construction, are available, among other laboratories and workshops [1, 2]. The interaction of the high-intensity, high-power laser pulse with solid matter creates a plasma from which particles are accelerated. For such experiments, two main parts are employed: the laser pulse, which is characterized by its pulse duration (usually in fs), energy (in J), focal spot (in μm) and temporal contrast, which shows how clean the laser pulse is (pre/ post-pulses, and the intensity of the pedestal); and the target, which can take multiple forms: gas, liquid, cryogenic, solid (flat, structured, multilayer) [3]. By tailoring the target and the laser pulse, the outcome - meaning the accelerated particles - can take different forms and configurations.

Nanostructured targets are materials with dimensions of the order of tens or hundreds of nanometers which can improve the interaction with a high-power, high-contrast laser pulse, in comparison with a flat target, by increasing the absorption of the laser energy due to a larger surface area and by volumetric heating [4], therefore the conversion efficiency of the laser energy to the accelerated particle energy is enhanced [5]. Studies about the ultra-high pressure formation and electron transport in the nanowire are made by PIC (particle-in-cell) simulations also at ELI-NP [6, 7]. Nanowires targets were reported in high-power laser experiments since the 2000s, with nanospaces that promote electron oscillation and ion collisions, low average density, and a high local-density structure [8], resulting in a 50-fold enhancement in X-ray emission compared with planar foils [9]. For their fabrication, electrochemical methods are the most versatile option, as they allow easy control of the structure parameters (diameter, interpore spacing, length, and substrate thickness, as well as the material). To deposit metals electrochemically, a template is prepared by aluminum anodization in acidic electrolytes [10]. The deposition of the structures can be performed directly in the alumina template grown on the aluminum substrate, or in a freestanding alumina template, detached from the aluminum substrate, an option that will allow for a thinner substrate.

2. Materials and Methods

Nickel nanowires were fabricated by electrodeposition in a porous alumina template obtained by aluminum anodization, by two routes, as shown in Figure 1 [11]. To change the parameters of the targets, investigate different configurations, and find an optimum, several fabrication parameters, such as the acidic electrolytes and different anodization voltages, have been explored to allow for wider geometric

characteristics of the final targets. Porous alumina is obtained by electrochemical anodization of aluminum. In this work, a high-purity 99.999% aluminum plate of 20 mm × 50 mm × 1 mm was used as base material, and as the anode for the electrochemical cell. The setup consists of a water-cooled double-jacketed electrochemical cell with a two-electrode system, a DC power supply (PSU 400–3.8, GW Instek) of 400 V maximum voltage output and 3.8 A maximum current output, 2 bench-top multimeters (Peaktech 4075) for current and voltage monitoring connected to a PC interface, a four-channel oscilloscope (Siglent SDS2204X PLUS) for AC pulsed electrodeposition, and a refrigerated heating circulator bath (Huber CC508) for temperature control.

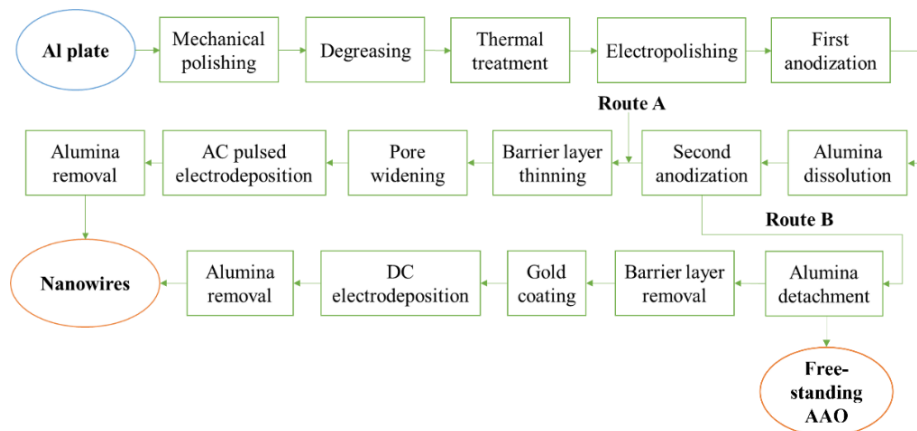


Figure 1. General block diagram of the process

Table 1. Anodization process parameters and resulting pore diameters.

Electrolyte	Anodization				BLT ¹	Detachment, V	Diameter, nm		Current density, mA/cm ²
	Voltage, V	Type ²	Time, h	Temp., °C			Front	cell size	
H ₂ C ₂ O ₄ 0.3 M	40	2 s	11.5	15	–	20-26	30	100	6.25
	40	2 s	2	15	–	18-23	25	80	8.75
	60	2 s	6	0	–	18-32	80	140	19
	90	M-H	6	0	yes	5-23	25	50	12.5
	90	M-H	4.2	15	–	45-53	70	225	23.13
	90	M-H	6	0	yes	5-27	30	50	7.5
	90	M-H	6.3	15	yes	5-9	35	30	18.75
H ₃ PO ₄ 0.4 M	110	M-H	4.5	0	–	–	25	290	18.75
	40	1 s	2	0	–	–	30	–	0.5
	80	2 s	3	0	–	–	100	–	–
	120	M-H	4	0	–	20-26	110	370	5
	140	M-H	6	0	–	22-68	140	400	2.5
H ₂ SO ₄ 0.4 M	170	M-H	6.6	0	–	30-37	200	400	3.13
H ₂ SO ₄ 0.4 M	20	1 s	2	0	–	10	15	40	9.88

¹ BLT - Barrier Layer Thinning. ² Anodization type: 1 s for 1 step, 2 s for 2 steps, M-H for Mild-High.

The cell contains the acidic electrolyte in which the anode and cathode (usually an inert material; in this work, titanium covered with platinum, a rod of 5 cm length and 7 mm diameter) are immersed.

For pre-treatment of the aluminum plate, we followed the steps shown in Figure 1: mechanical polishing to reduce roughness, degreasing in acetone in an ultrasound bath, and thermal treatment at 500°C for 3 h. Electropolishing was performed in a perchloric acid mixture with ethanol in a 1:4 ratio, at 0°C, 10 V, for a few minutes, until a mirror-like polish was obtained.

In this work, the anodization parameters that give the characteristics of the porous alumina are:

- the electrolyte: oxalic acid, phosphoric acid, sulphuric acid;
- electrolyte concentration;
- electrolyte temperature, which was controlled to be under 15°C to avoid the burning of the sample;
- anodization voltage, which gives the pore diameter; with increased voltage (varied between 20-170 V), a larger diameter can be obtained;

After the anodization of aluminum, the porous alumina layer can be detached from the plate by changing the electrodes, as the anode becomes the cathode and vice versa, and a step-like voltage procedure is applied. The starting voltage depends on the anodization voltage, as for higher anodization voltages the sample has more resistance and, therefore, a higher starting voltage must be used, which is why this method is not entirely reproducible and may lead to the detachment of the template in multiple pieces or with holes. After detachment, the pores can be opened on the bottom side by chemical etching in sodium hydroxide or phosphoric acid. The steps are presented in Figure 1. All the process parameters are given in Table 1, where cell size refers to the dimension given by both the walls and the diameter of the pore, representing the morphological unit of the porous alumina template. Interpore spacing is the distance measured between the margins of two pores. The Mild-High approach [12] was used to conduct the anodization in one step, initially at a lower voltage for 10 minutes, after which the voltage is slowly raised by 1 V/s, to reduce the burning of the samples and to allow for higher voltages, which result in bigger diameters.

The pore-widening is performed by etching in a 1 M phosphoric acid solution, at room temperature for 20 minutes to 2 h, depending on the thickness of the alumina, the pore diameter, and the desired final result.

The freestanding porous alumina is used as a template for nanowire deposition, but first a metallic thin layer is deposited on one side of the open pores to serve as an electrode, in the Watts bath, which is a mixture of metallic salts of the metal wanted to be deposited. After deposition of the nanowires inside the template, the structures are liberated by dissolving the alumina template in NaOH

solution. The resulting nanowires have various diameters and lengths, according to the template characteristics.

For nanowires with diameters smaller than 200 nm and lengths longer than a few microns, the structures tend to cluster and break from the support during the drying process due to the liquid surface tension. As possible solutions to the clustering problem, alumina can be grown with larger pore diameters or by optimizing the drying process of the nanowires after template dissolution, by using methods like freeze drying (lyophilization) or supercritical drying.

The samples were characterized after each step of the fabrication process using a scanning electron microscope (SEM Tescan Maia 3), operated at 3-10 kV beam accelerating voltage, with an in-beam secondary electron detector and a working distance of 6-7 mm. The elemental analysis was performed with a Flash 6/30 Bruker Nano GmbH EDS detector (integrated in the SEM equipment), at a working distance of 6 mm (sample-pole piece), at 4 kV.

3. Results and Discussion

The parameters of anodization in different electrolytes, together with the resulting pore diameters, are given in Table 1. The obtained nanochannels (porous alumina - also used as a template) have pore diameters of 40 – 400 nm and interpore distances of 50 - 350 nm. The nanowires will have the same diameters and pore distances as the template, and are made of copper or nickel, with heights from hundreds of nanometers to tens of micrometers. They are vertically aligned, homogeneously distributed on large areas (cm²), and sit on a substrate made of either aluminum or copper, with controllable thickness starting from 300 nm, as depicted in Figures 2 and 6.

3.1 Synthesis of porous AAO and copper NWs on Al substrate

A highly pure aluminum plate of 1 mm thickness is partially anodized on one side (inset in Figure 2(b)), in phosphoric acid, to create a layer of porous aluminum oxide of a few micrometers thickness, with pores of several hundred nanometers. A barrier layer thinning process [13] is performed at the end of the anodization by gradually reducing the anodization voltage, which creates small channels at the bottom of the pores, reaching the aluminum substrate. For this, the voltage is reduced by 1 V each minute until 5 V, when the process is stopped.

The electrodeposition of copper nanowires was performed with the pulsed AC method at 200 Hz, sine wave, and ± 15 V [13], to ensure that the copper ions enter the pores and form the structures. The solution was made with copper sulfate 0.5 M and boric acid 0.57 M, with a pH of 3-3.5. The alumina template is dissolved in an NaOH solution, and the nanowires are liberated on the aluminum substrate. In Figure 2 (a), SEM image showing the top view of the aluminum oxide template

is depicted, and images of the copper nanowires are presented in (b), (c), (d), and (e) for longer deposition durations.

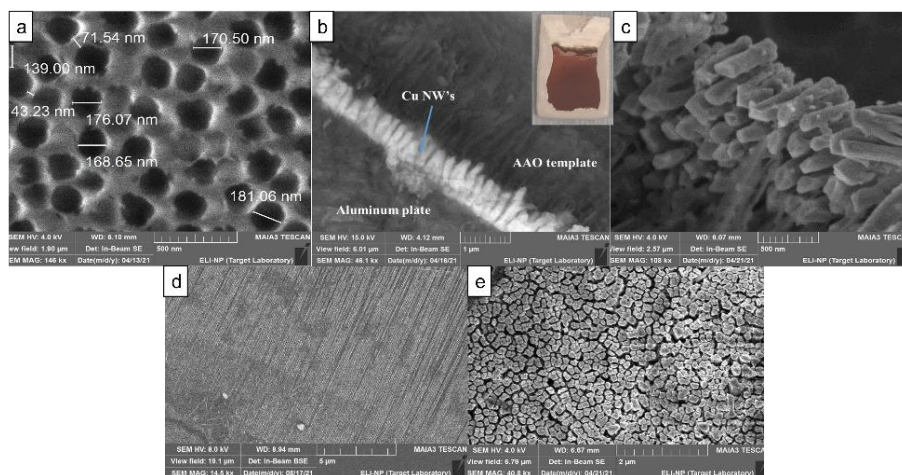


Figure 2. One-step anodization and pulsed AC electrodeposition. SEM images of (a) open pores of alumina template, (b) lateral view of the Cu NWs embedded in the alumina template, (c) liberated CuNWs on the aluminum substrate, (d) cross-section of porous alumina template after a long duration copper deposition, (e) top view of liberated CuNWs.

Figure 3 shows the elemental analysis by EDS (energy-dispersive X-ray spectroscopy) with the spectra of one sample after anodization (orange), where we notice that only aluminum and oxygen from the alumina template are present, with some contaminants from phosphor (from the electrolyte) and carbon from the environment; and with blue, the sample after electrodeposition, with copper from the nanowires and oxygen and aluminum from the template.

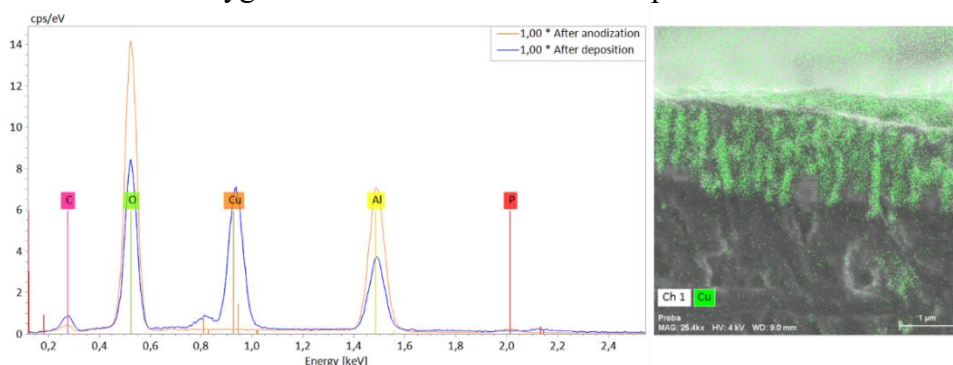


Figure 3. EDS analysis of one sample at 4 kV: (left) elemental spectra after anodization (orange) and after electrodeposition (blue); and (right) mapping after copper electrodeposition showing the copper channel for nanowires with green

On the right of Figure 3 we depict the elemental mapping of the sample, where we can observe the electrodeposited copper nanowires inside the template (top), placed on the aluminum plate (bottom). The percentages for each sample are given in Table 2.

Table 2. EDS measurements for samples after anodization and after copper deposition

Element	Atomic percent (at. %)	
	After anodization	After electrodeposition
Al	36.84	27.41
O	59.52	49.75
Cu	-	16.17
C	1.31	6.17
P	2.33	0.51

3.2 Synthesis of free-standing porous AAO template

The anodization process is performed in two steps to form a highly ordered template, in 0.3 M oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) electrolyte, at 40 V. As cathode, a cylindrical Ti electrode covered with Pt is used, and the anode is the aluminium foil of 1 mm thickness, on which the oxide will form, on all the parts in contact with the electrolyte, meaning on the front, back, laterals, and bottom, with respect to the counter electrode.

Table 3. Parameters and results for the freestanding alumina fabrication

Sequence	First anodization		Second anodization		AAO thickness, μm
	Time, h	Temperature, $^{\circ}\text{C}$	Time, h	Temperature, $^{\circ}\text{C}$	
1	6	10	11.5	10	60-63
2	6	15	2	15	7-8
3	6	15	3	15	20-23

To obtain a freestanding alumina template by detachment, the electrodes are interchanged after the anodization process, with the aluminum plate as the cathode (-), and the Ti/Pt counter electrode as the anode (+), after which stair-like reverse biases are applied starting at 18 V up to 26 V by 1 V every few minutes, with the current limit set to 2 A, until the template detaches [14].

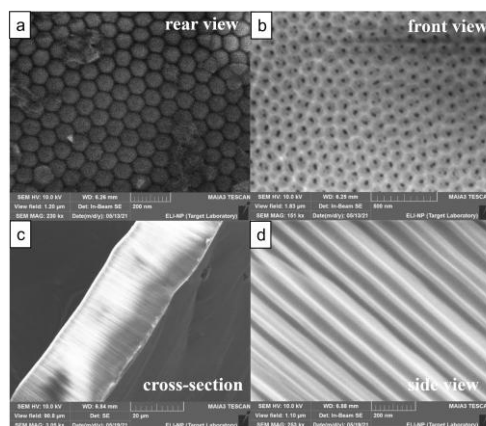


Figure 4. SEM images of freestanding porous alumina (a) rear view, (b) front view, (c) cross-section, (d) side view.

The procedure is reproducible for samples anodized in oxalic acid at 40 V, while when different acids and voltages are used, the detachment process conditions change and have been tested, as presented in Table 1. The resulting template thickness related to the anodization time is presented in Table 3. In Figure 4 the SEM images of the freestanding porous alumina template are shown. The aluminum plate can be reused multiple times, as shown in Table 3, where 3 sequences are presented using the same aluminum plate.

3.3 Free-standing copper nanowires by DC electrodeposition

Using the freestanding porous alumina template presented in the previous section, copper nanowires were grown. The template is initially treated in a phosphoric acid solution to widen the pores and reduce the barrier layer thickness, followed by etching of the barrier layer by floating on 4 M sodium hydroxide, on the back part of the template, until all the pores are opened, usually for 2 to 10 minutes, at room temperature, with the results presented in Figure 5 (c,d). The template is characterized with the SEM, and if the pores are not completely open, the etching is continued for a few more minutes. A seed layer of 100-200 nm of gold is sputtered on one side of the template, and DC electrodeposition is performed at room temperature in a cell with 2 electrodes, where the counter electrode is a copper foil, in potentiostatic mode, at 3 V. The electrolyte for electrodeposition is the same as in Section 3.1, and the deposition time is of a few minutes, enough to grow nanostructures of several micrometers length. The obtained copper nanowires on a very thin substrate are presented in Figure 5.

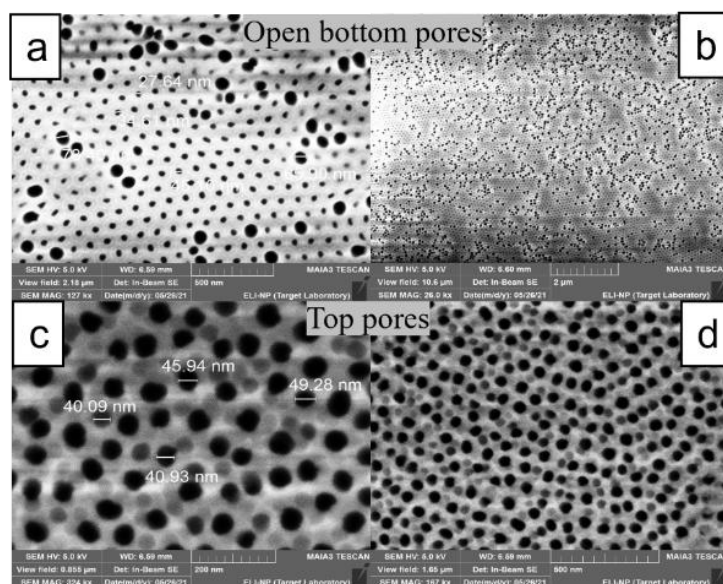


Figure 5. SEM images of (a, b) open bottom pores, and (c,d) open top pores of a free-standing porous AAO template used for nanowires growth

By using the same method, but a different deposition bath with nickel salts (NiSO_4 , NiCl_2), nickel nanowires were grown [15], as shown in the cross-sectional SEM image in Figure 6 (d).

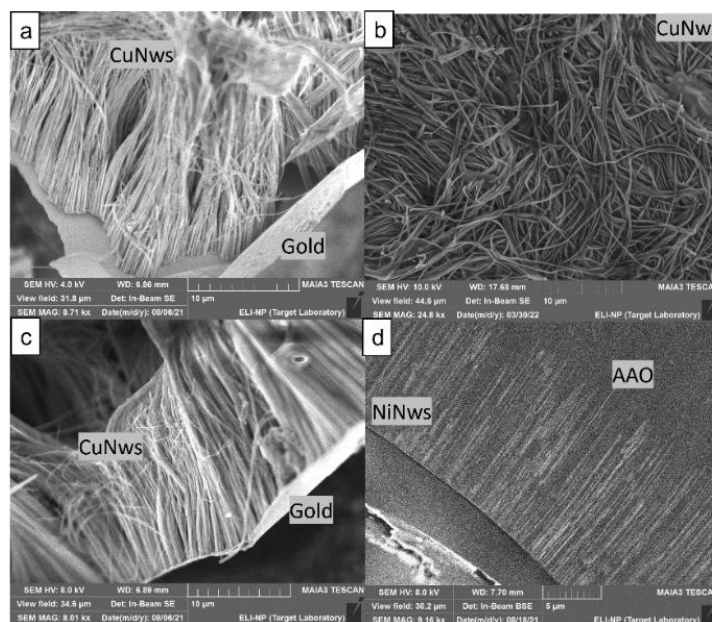


Figure 6. SEM images of (a,b,c) CuNws on a gold substrate, (d) NiNws in a porous alumina template.

4. Conclusions

The synthesis of porous alumina on an aluminum plate, using several electrolytes, and the detachment of the template by using stair-like reverse biases, have been presented. The obtained template was used for nanowire fabrication, either by DC or AC electrodeposition, resulting in structures with diameters from tens of nanometers to several hundred nanometers and lengths of a few microns. Such materials, which offer a bigger surface area compared with flat foils, are further used in high-power laser experiments as targets in order to improve the interaction with the laser pulse.

Author Contributions

Conceptualization, S.I., C.G. and V.L.; methodology, S.I., C.G.; formal analysis, S.I.; investigation, S.I., D.P., L.F., C.G.; writing—original draft preparation, S.I.; writing—review and editing, S.I., V.L., C.G.; supervision, V.L.; funding acquisition, V.L. All authors have read and agreed to the published version of the manuscript.

Data Availability Statement

Data supporting the findings of this study are available upon reasonable request from the corresponding author.

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Conflicts of Interest

The authors declare no conflict of interest.

Artificial Intelligence Disclosure

No artificial intelligence-based or generative tools were used in the preparation of this manuscript.

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