



Nanoplatfoms for Neuroscience— NeuroNanotech

Deliverable D2.1

First nanostructured metallic and conductive polymeric electrodes

Lead Beneficiary	IMDEA Nanoscience
Dissemination level	Public
Version	V1



**Funded by
the European Union**

Document Information

Grant Agreement Number	10116935
Project acronym	NeuroNanotech
Start date of project (Duration)	01/01/2025 (48 months)
Deliverable lead	IMDEA Nanoscience
Deliverable number	D2.1
Deliverable name	First nanostructured metallic and conductive polymeric electrodes
WP	WP2
Dissemination level	PU – Public

History of changes				
Version	Date	Author	Comments / Changes	Pages

EXECUTIVE SUMMARY

This document describes first prototypes of neural probes for stimulation developed at IMDEA Nanoscience. This work constitutes deliverable D2.1, First nanostructured metallic and conductive polymeric electrodes. Two types of probes are being developed, one based on metallic electrodes, mainly Pt, and the other based on laser induced graphene electrodes. In both cases, the electrodes are integrated over flexible polymeric substrates.

CONTENTS

1. Abbreviations and Acronyms.....	3
2. Description	3
Metallic-electrode probes	3
Probes with flat Pt electrodes	4
Probes with nanostructured Pt electrodes.....	5
Carbon-electrode probes.....	7
Laser-induced process over Kapton	7
Structural and chemical characterization of LIG substrates	7
Transfer of laser-induced graphene to PDMS	9
Design of photolithography masks for penetrating neural probes.....	10
3. Consortium	11

4. Contact data.....	11
----------------------	----

1. ABBREVIATIONS AND ACRONYMS

PI	Polyimide
NW	Nanowire
AAO	Anodized aluminium oxide
SEM	Scanning Electron Microscopy
RIE	Reactive ion etching
LIG	Laser induced graphene
PDMS	Poly(dimethylsiloxane)

2. DESCRIPTION

First prototypes of flexible neural probes for stimulation have been fully developed at IMDEA. Two types of probes are being developed: those with metallic electrodes and those with carbon-based electrodes. These prototypes integrate electrodes and conduction paths with diameters and widths in the range of tens to hundreds of micrometres and are, therefore, especially suitable for *in-vitro* experimentation. Designs for devices with elements in the range of a few micrometres, suited for *in-vivo* studies, are presently prepared as well.

Metallic-electrode probes

We fabricated probes with Pt electrodes over polyimide (PI) in which the conduction path has been passivated with a second layer of PI. The exposed Pt pads are either flat or nanostructured with a network of vertical Pt nanowires (NWs).

For the device base (Figure 1.1a), PI (PI-2611, HD MicroSystems) was spin-coated over glass slides following the manufacturer's specifications to obtain 5 μm thick films. These were then soft baked at 120 °C for 2 min on a hot plate and, subsequently, cured at 350 °C for 1 h under nitrogen atmosphere (furnace: YES-459PB6-2PE-CP, Yield Engineering Systems Inc., San Jose, CA, USA).

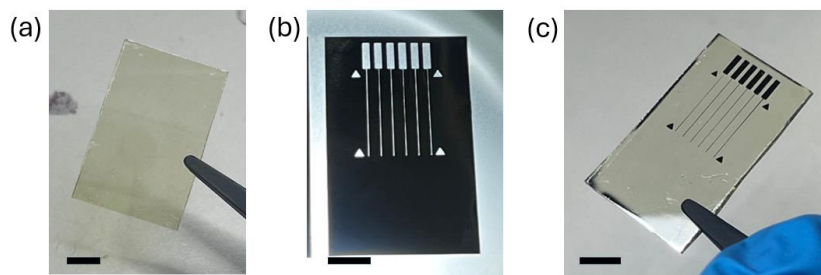


Figure 1.1. (a) 5 µm thick PI layer over a glass slide. (b) Al stencil with the circuit profile. (c) PI layer with Pt circuits. Scale bars: 5 mm in all the figures.

Probes with flat Pt electrodes

Stencils with open lines down to 80 µm in width were fabricated on 40 µm-thick Al foil using a laser marker (JLC MLFS 20 W) (see Figure 1.1b). The circuit geometry was optimized to match the external connectors (shown below) while enabling the smallest achievable features. These were used to define the Pt electrode circuits over the PI by sputtering of a 125 nm thick layer of Pt (see Figure 1.1c).

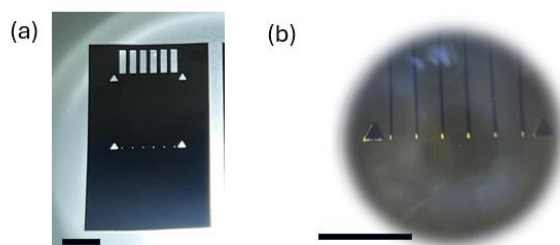


Figure 1.2. Photographs at different magnification of the Al stencil used to expose Pt pads by RIE through non-photosensitive PI. Scale bars: 5 mm in all the images.

A second layer of PI (PI-2610, HD Microsystems) was used to passivate the conduction paths, and small electrode pads were opened on it by reactive ion etching (RIE) in oxygen flow (5 min at 150 W) through a second Al stencil (see Figure 1.2). An alternative procedure, using photosensitive PI (PSPI-LTC 9310-E19, FUJIFILM) with an optical mask was also successfully explored. Electrode pads down to 20 µm × 145 µm were opened, as shown in the scanning electron microscopy (SEM) images of Figure 1.3.

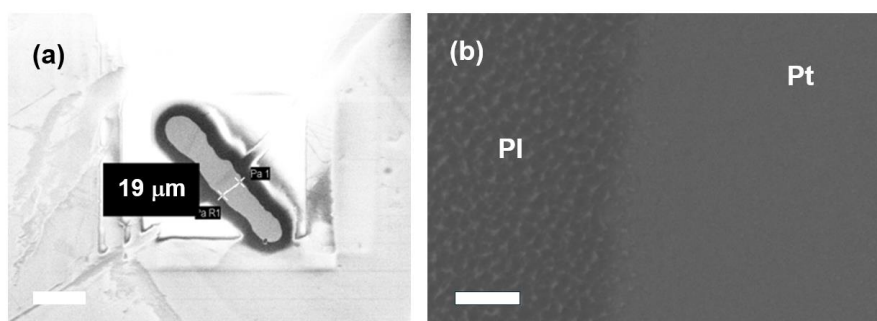


Figure 1.3. SEM images of the region open through PI leaving an exposed Pt pad on the circuits. Non-photosensitive PI was used in this case. The regions of exposed Pt and covered with PI are indicated in part (b). Scale bar: (a) 50 µm, (b) 500 nm.

Connectors were fixed to the circuits using silver paint with the help of an ad-hoc fabricated holder (Figure 1.4a). Epoxy resin (Epo-Tek) was used to passivated and further protect the connection area and then the whole device can be peel off from the glass slide just immersing it in water. Figures 4c and d show an image of the final flexible device ready to use.

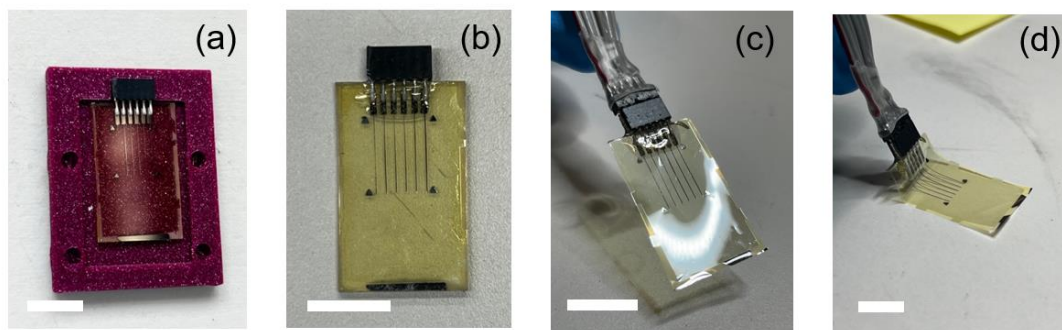


Figure 1.4. (a) Probe prototype on a holder that facilitates the alignment with the connectors. (b) Prototype with connectors fixed with Ag paste and protected with epoxy resin. (c) and (d) flexible probe prototype separated from the glass base. Scale bars: 1 cm in all images.

Cyclic voltammetry was performed in these probes in 0.5 M H_2SO_4 at 50 mV/s, with an Autolab potentiostat/galvanostat. We run 50 consecutive oxidation/reduction cycles without observing significant deterioration or delamination of the electrodes, as shown in Figure 1.5. The evolution of the peaks in the voltammograms evidence a restructuring of the Pt surface in the electrode pads.

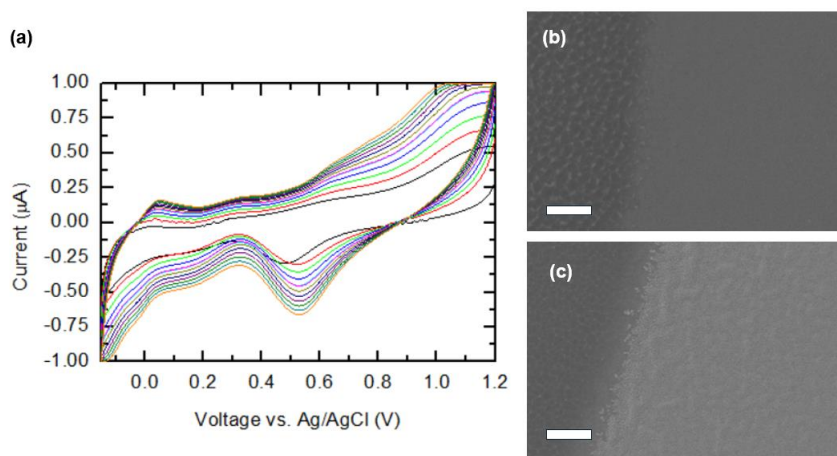


Figure 1.5. (a) Selected voltammograms out of 50 consecutive recorded cycles in H_2SO_4 at 50 mV/s for one of the electrode pads of a flexible neural probe prototype. (b) SEM image of the pad before the voltammetry. (c) corresponding SEM image after the voltammetry. No significant deterioration of the electrode is observed. Scale bars: 500 nm in all images.

Probes with nanostructured Pt electrodes

In this case, the electrode circuits were first defined over a nanoporous template (Whatman® alumina Anodisc filter discs 0.2 μm pore size), Pt NWs were grown by template-assisted electrodeposition¹ and then, the nanostructured circuits were transferred over the PI base.

¹ A. Domínguez-Bajo / B.L. Rodilla et al., [Adv. Biosys. 2020, 2000117](#); B.L. Rodilla / A. Arché-Núñez et al., [Sci Rep 14, 3729 \(2024\)](#)

Using an Al stencil prepared to fit the template shape (Figure 1.6a), the electrode circuits were defined over the anodized aluminium oxide (AAO) templates by sputtering (Figure 1.6b). Next, Pt NWs were grown by electrodeposition using a homemade Pt electrolyte. The electrodeposition time was chosen to ensure that all the nanopores of the template were fully covered leaving a homogeneous Pt layer on top of the template surface as shown in Figures 6e and f. In this situation, Pt filled the nanopores in the template forming NWs.

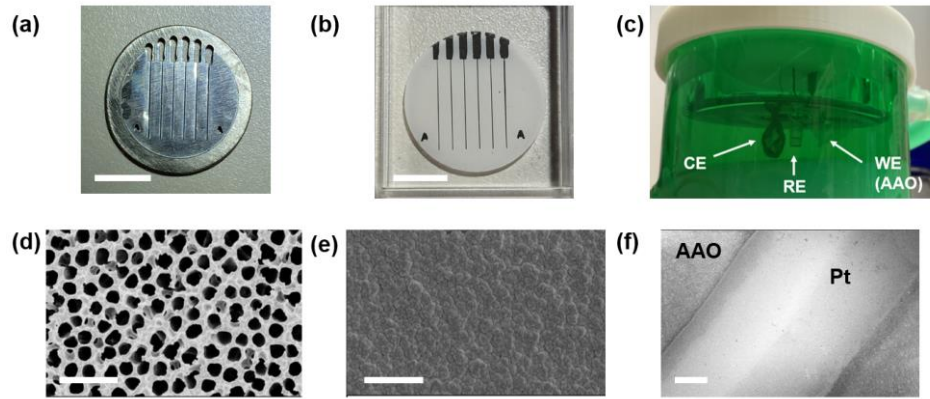


Figure 1.6. (a) Al stencil with the selected circuit profile. (b) AAO template after Pt sputtering through the Al stencil. (c) Double-jacket electrochemical cell used for electrodeposition. The reference electrode (RE), counter electrode (RE) and working electrode (WE) are indicated in the image. (d) SEM image of the AAO template after Pt sputtering shown open nanopores. (e) and (d) SEM images at two different scales of the Pt layer electrodeposited over the AAO template. Scale bars: (a) and (b) 5 mm, (d) and (e) 1 μ m, (f) 50 μ m.

The template is then transferred to the PI base described above, right after a short soft baking and then fully cured. Finally, the template is dissolved at 45 $^{\circ}$ C in a mixture containing chromium oxide (CrO_3 , 0.18 M) and orthophosphoric acid (H_3PO_4 , 4.2 %, 85 % purity), leaving a Pt circuit coated with Pt NWs on the surface of the PI base, as shown in Figure 1.7. In addition of the Pt circuits, the PI region around them is also nanostructured, as it has been cured in contact with the nanoporous template (see Figure 1.7e).

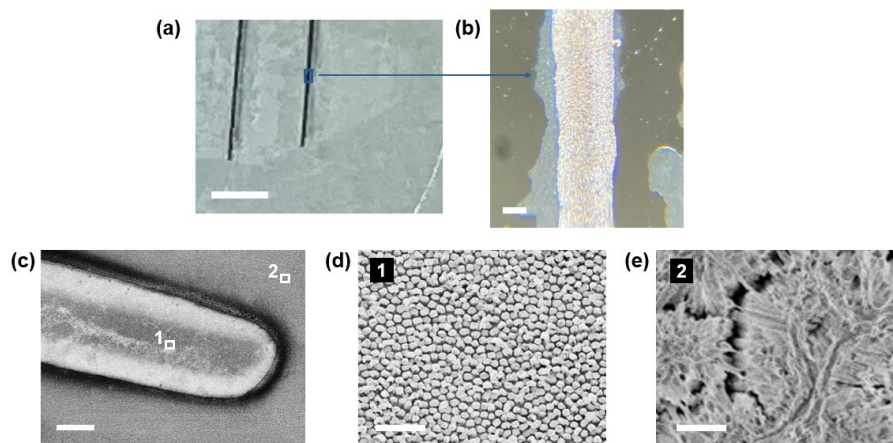


Figure 1.7. (a) and (b) Photographs at different magnification of the nanostructured Pt circuits transferred to the PI base of the neural probe. (c) SEM image at the end of one circuit. (d) Magnified SEM image of the region marked as 1 in part (c) corresponding to Pt NWs. (e) Magnified SEM image of the region marked as 2 in part (c) corresponding to nanostructured PI. Scale bars: (a) 1 mm, (b) and (c) 100 μ m, (d) and (e) 2 μ m.

Carbon-electrode probes

Laser-induced process over Kapton

Carbon-based electrodes were produced using laser-induced graphene (LIG), generated through laser photoablation and photothermal transformation of carbon-rich polymers such as polyimides. The irradiation causes the photothermal transformation of the substrate carbon-rich material as a direct route to obtain highly porous, electrically conductive, 3D graphene-like structures. This technique provides a highly scalable, cost-effective, and direct procedure for the fabrication of nanostructured graphene-like conductive architectures directly on flexible polymers.

In terms of substrate selection, commercial polyimide (Kapton) has been reported to provide some of the highest patterning resolution for LIG fabrication and was, therefore, chosen here. The LIG process was carried out using a 10.6 μm CO₂ laser engraver and cutter (Trotec Speedy 400). After generating the computer-designed layout, the laser was rapidly scanned across a 50 μm -thick commercial Kapton film at ambient conditions. The quality and resolution of the resulting LIG patterns produced were found to depend strongly on heat dissipation, which in turn is governed by the pattern geometry. Consequently, narrow interconnects and larger electrode areas required distinct laser settings to achieve uniform LIG formation:

- Interconnects: Power = 17%, Scan speed = 15%, Resolution = 1000 DPI, Z-offset = -2 mm.
- Contact pads: Power = 11%, Scan speed = 12%, Resolution = 600 DPI, Z-offset = -1 mm.

Figure 2.1 below shows an example of the LIG electrode patterns produced, comprising 1 mm diameter electrode pad areas, 200 μm wide interconnects, and 1 mm wide external contact regions.

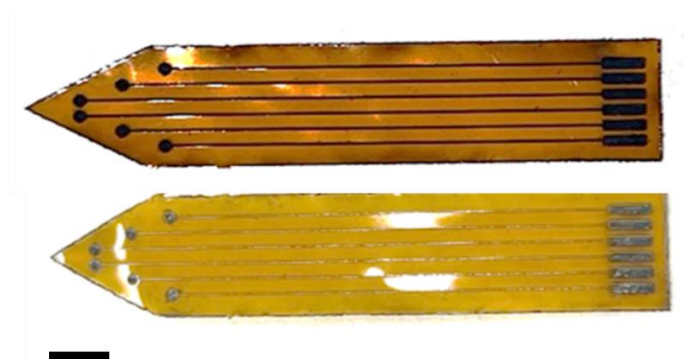


Figure 2.1. LIG electrodes fabricated on Kapton film: front side (up) and back side (down). Scale bar: 5 mm.

Preliminary resistance measurements of the LIG conductive patterns obtained using a voltmeter, yielded the following values:

- Contact pads: resistance in the order of a few hundred ohms.
- Interconnects: resistance in the 10 k Ω range in 34 mm.

Structural and chemical characterization of LIG substrates

The structural and chemical properties of LIG patterns on PI of both the contact pads and interconnects, were analysed using Raman spectroscopy. Raman spectra were acquired using a Bruker SENTERRA

confocal Raman microscope using a 532 nm green diode laser for excitation. Spectral collection was performed at 5 mW, using 10 accumulations with an integration time of 3 s per scan.

The Raman spectra obtained, shown in Figure 2.2, corroborates the graphitization achieved. The presence of a prominent G peak at $\sim 1580\text{ cm}^{-1}$ confirms the formation of sp^2 -hybridized carbon atoms, which is characteristic of graphitic materials. At the same time, the D peak at $\sim 1350\text{ cm}^{-1}$, suggests a high density of sp^2 structural defects, which is expected in LIG due to its photothermal formation mechanism. In addition, the appearance a broadened and less intense 2D peak at $\sim 2700\text{ cm}^{-1}$, also indicates the formation of multilayer graphene. The pronounced D peak, confirms that the material is not a pristine monolayer graphene but a multilayer graphene with high structural disorder, in agreement with previous reports.²

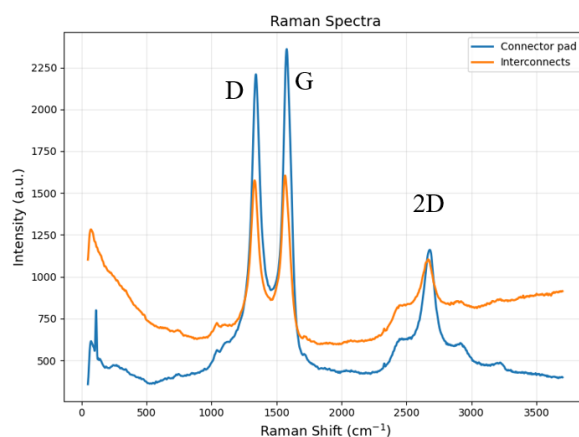
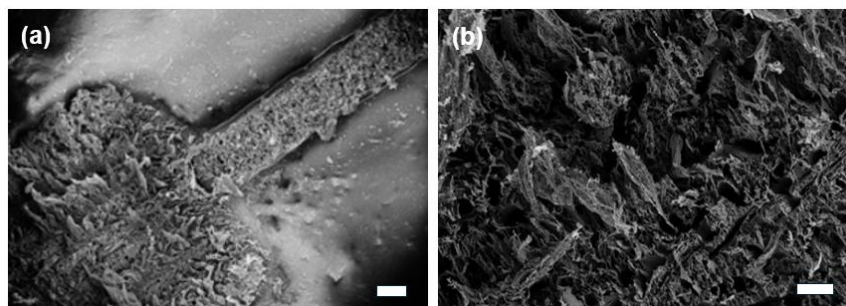


Figure 2.2. Raman spectra of the porous graphene network patterns in Kapton films created by LIG.

Structural characterization was performed using SEM to examine the surface morphology and graphitic microstructure of the laser-graphitized polyimide. Samples were imaged without conductive coating to preserve the native LIG microstructure. As shown in Figure 2.3, high-magnification SEM images reveal a porous graphene network at the electrode patterned sites. This porous architecture is expected to increase the effective surface area and enhance charge-transfer efficiency.



² Zhenhao Wu, Mirza Sahaluddin, Diala Bani Mustafa, Soumalya Ghosh, Mostafa Bedewy; Laser-induced graphene: from precursor chemistry to process control and throughput–resolution–performance trade-offs. *Nanoscale* 2026; 18 (13): 6647–6686. <https://doi.org/10.1039/d5nr04319k>

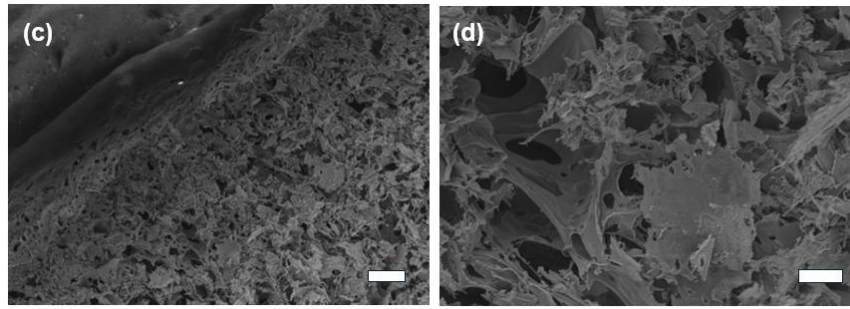


Figure 2.3. SEM images showing the porous graphene network formed by LIG. (a) Overview of the electrode structure; (b) contact pads; (c-d) interconnects at different magnifications at the electrode site. Scale bars: (a) 100 μm , (b) and (c) 20 μm , (c) 5 μm

Transfer of laser-induced graphene to PDMS

Aiming to obtain highly soft and compliant neural probes, the LIG patterns generated on Kapton were transferred onto the biocompatible polydimethylsiloxane (PDMS) elastomer.

The PDMS precursor (Sylgard 184, Dow Corning) was prepared at a 10:1 prepolymer- to-crosslinker ratio, mixed, degassed, and then cast over the LIG-patterned surface, allowing the uncured elastomer to infiltrate the LIG porous network. It was then cured at 70 $^{\circ}\text{C}$, promoting a mechanical interlocking of the porous carbon structures by the PDMS (see Figure 2.4a). Finally, the Kapton film was peeled off (Figure 2.4b), yielding a flexible probe with conductive carbon patterns embedded in PDMS (See Figure 2.4c).

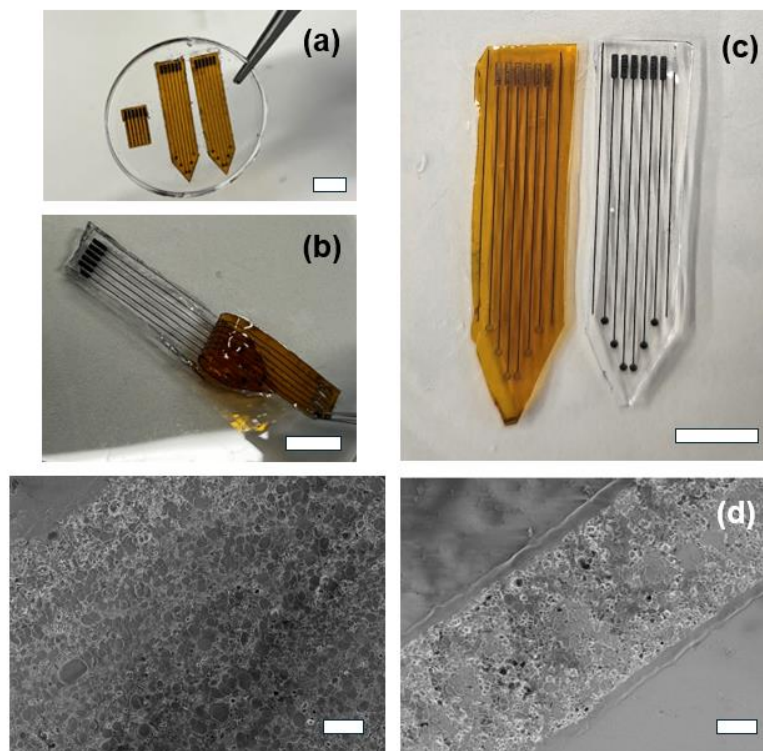


Figure 2.4 Transfer of LIG patterns to PDMS. (a) LIG electrodes on Kapton embedded within a cured PDMS block. (b) Kapton peeling step during LIG transfer to PDMS. (c) LIG electrodes fully transferred onto the PDMS substrate. (d) SEM images showing the porous graphene network embedded within the PDMS matrix. Scale bars: (a), (b) and (c) 10 mm, (d) 50 μm

High-magnification SEM images in Fig. 2.4d show the porous graphene network embedded within the PDMS matrix. In this case, the structure is still disorganized, but some circular features can be observed. These may be arising from blister based LIG formation during the photothermal transformation process. Resistance measurements of the LIG embedded patterns obtained using a voltmeter, yielded the resistance values in the 40 k Ω range.

Design of photolithography masks for penetrating neural probes

Several photolithography mask layouts were designed for penetrating neural probes, with geometries and electrode configurations optimized for stimulation of mouse neural tissue by the consortium partners. The corresponding probe designs, including dimensions, electrical interconnects, and stimulation sites, are shown below.

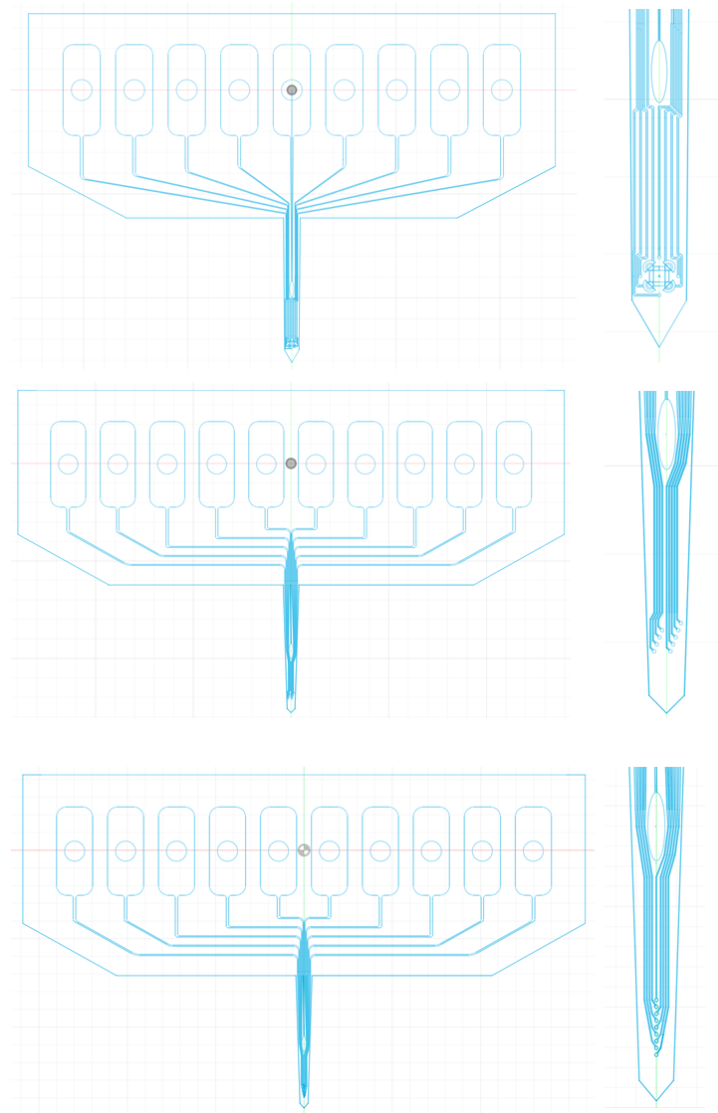


Figure 3.1. Photolithographic mask layout for the fabrication of flexible penetrating neural probes.

3. CONSORTIUM



HOST INSTITUTIONS

IMDEA NANOCIENCIA - Madrid (Spain)
CNRS-GREYC - Caen (France)
CEA - Paris (France)
TEL AVIV UNI - Tel Aviv (Israel)
ATLAS - Leuven (Belgium)
SISSA - Trieste (Italy)
UAM - Madrid (Spain)
CIMA - Pamplona (Spain)
RADBOUD UNI - Nijmegen (Netherlands)
UNIVERSITETET I OSLO - Oslo (Norway)



PARTICIPANTS

BOSTON SCIENTIFIC - Galway (Ireland)
MTTLAB - Trieste (Italy)
SILICON AUSTRIA LABS - Graz (Austria)
CSIC - Madrid (Spain)
UCM - Madrid (Spain)
NORWEGIAN UNI OF LIFE SCIENCES - Ås (Norway)
EUROPEAN BRAIN COUNCIL - Brussels (Belgium)
PARIS LODRON UNIVERSITY OF SALZBURG - Salzburg (Austria)
FIBIOHRC - Madrid (Spain)
UMC UTRECHT - Utrecht (Netherlands)
FUNDACIÓN JUAN XXIII RONCALLI - Madrid (Spain)
UNICAEN - Caen (France)
PARIS-SACLAY UNI - Paris (France)
U. NAVARRA - Pamplona (Spain)
KU LEUVEN - Leuven (Belgium)

4. CONTACT DATA

<https://neuronanotech.eu/>

Coordinator: Instituto Madrileño de Estudios Avanzados en Nanociencia (IMDEA Nanociencia)

Competitive Projects Office

C/ Faraday 9

28049 Madrid

Spain

neuronanotech.project@imdea.org

+34 91 299 87 00