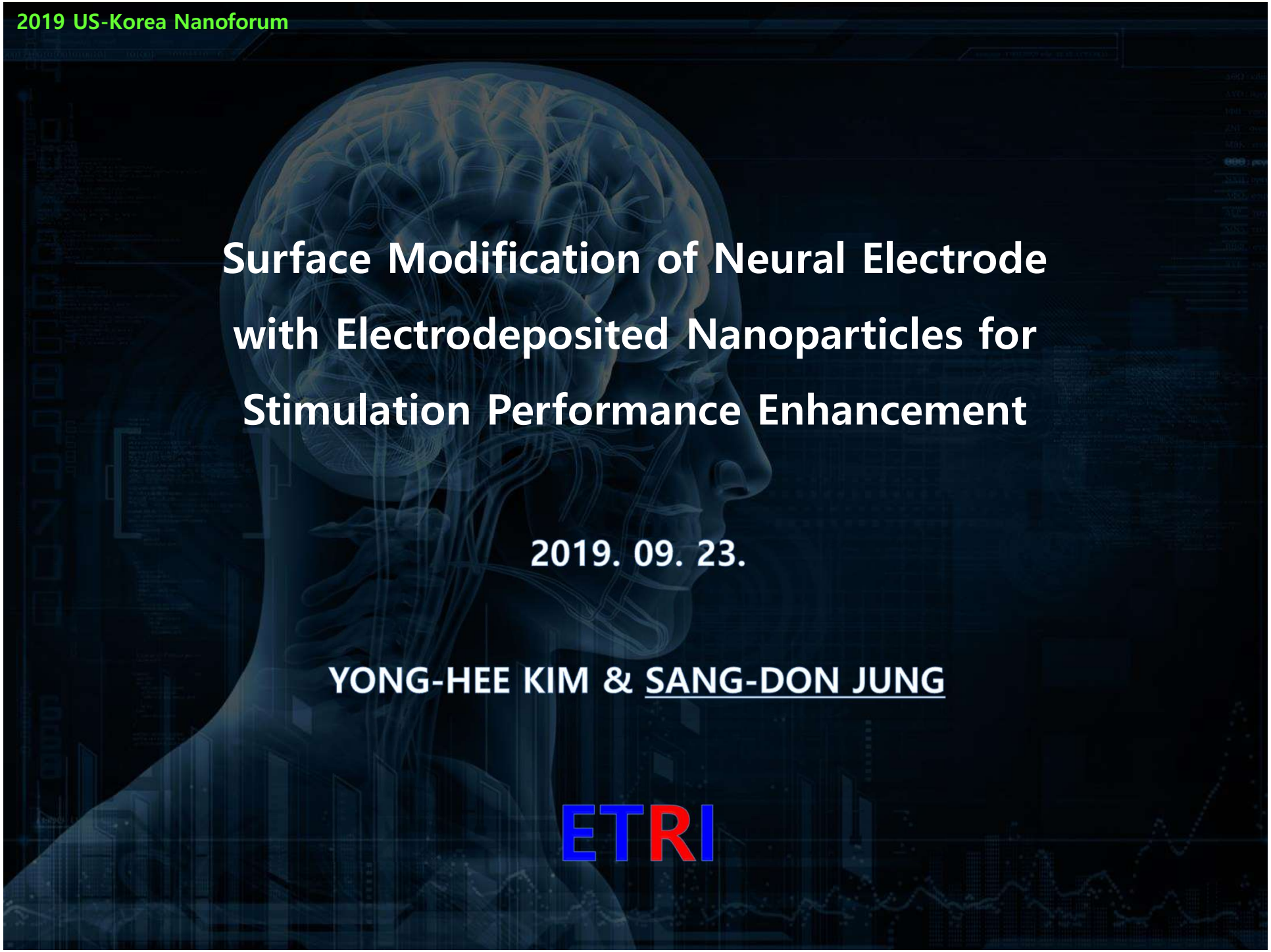


Surface Modification of Neural Electrode with Electrodeposited Nanoparticles for Stimulation Performance Enhancement

2019. 09. 23.

YONG-HEE KIM & SANG-DON JUNG

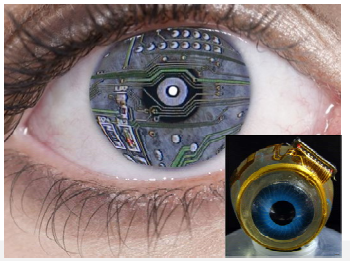
ETRI

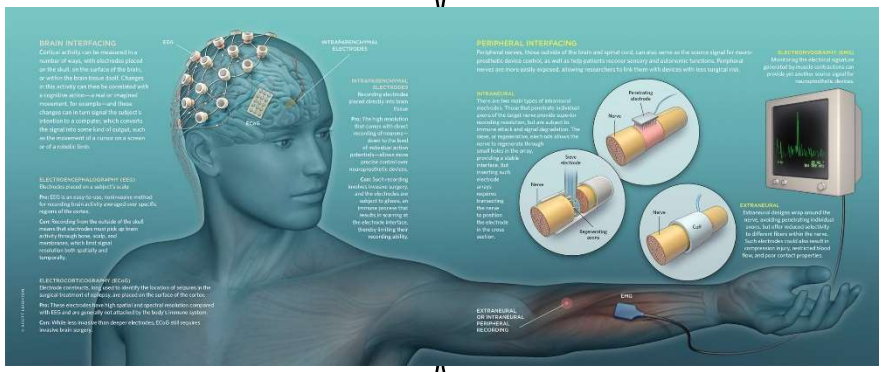
Application Potentials

- **Applications for neurological disorders**

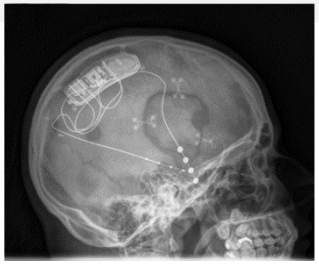
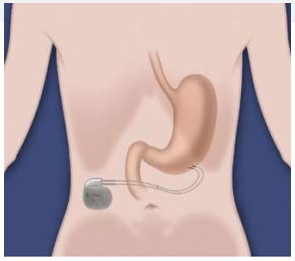


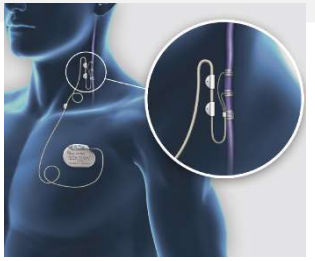








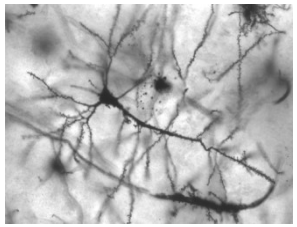



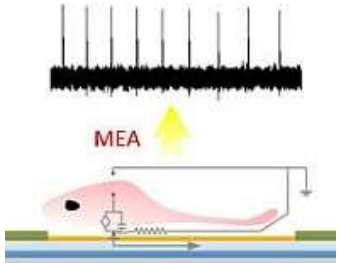


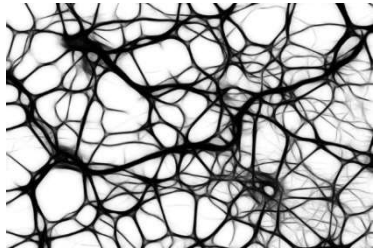
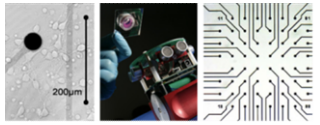
Multi-electrode Array (MEA)

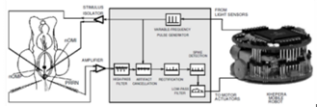
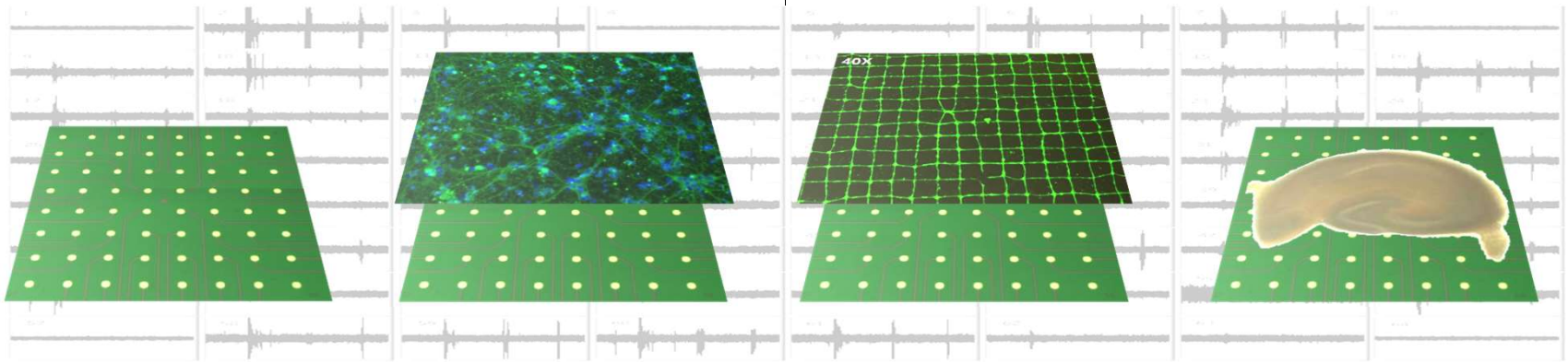
- **Definition**
  - **MEAs** or microelectrode arrays are devices that contain multiple micro plates
  - **Extracellular recording & stimulation**
  - 1st MEA by Thomas Jr. (1972) & commercialized by Multi-Channel Systems® (1996)
- **Application potential**

Disease model	Drug screening	Cell-based sensors	Network modelling	Learning



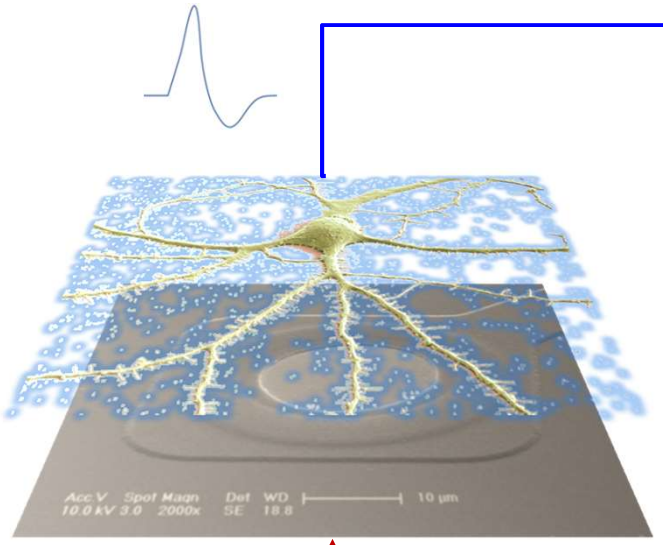


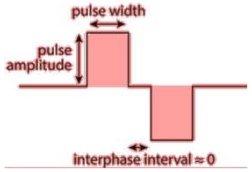



Specifications

- **Requirements for neural-computer bi-directional interface**





Recording
- High SNR: » 5
- Low noise
- Long-term viability
- Temporal resolution
- Electrode size: <30 μm
  – Single unit recording
- High # of channel: ~256 CH
- Intimate contact
- Spike sorting

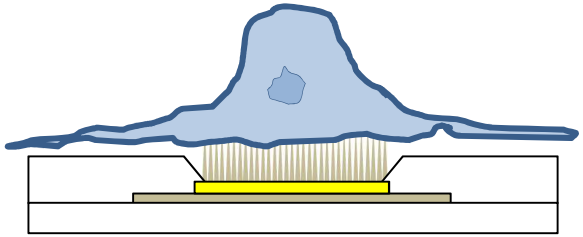
Decoding

Stimulation
- Charge injection capacitance: »
- Charge injection limit: » 1 mC/cm^2
- Charge injection efficiency: ~1
- Damage threshold: 1 mC/cm^2
- Long-term durability
- Field focality

Encoding

Extracellular Recording & Stimulation

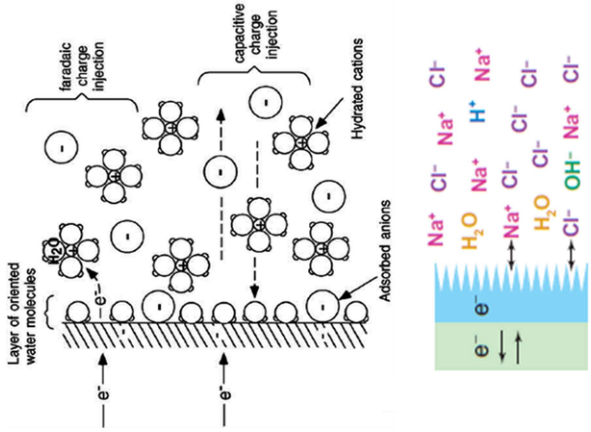
- **Recording issue**
  - Impedance control for reduction of interfacial noise



$$N_{e.e} = \sqrt{4\,k\,T\,Re(Z_e')\,\Delta f}$$

$$Z_e' \propto 1 / A_s^2$$

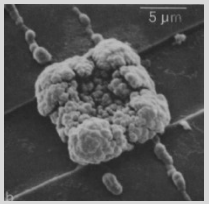
- **Stimulation**
  - Charge storage capacitance
  - Charge injection limit
    - Electrochemical window: -0.64 ~ 0.75 V (vs. SCE)*
    - Safety limit: ~ 1 mC cm^{-2} **
  - Material dependence



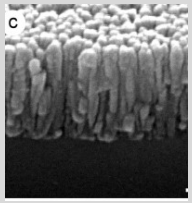
$$q_c = C_{dl}\,\Delta V$$

- **Common requirement**
  - Increase in surface area
- **Long-term reliability issues**

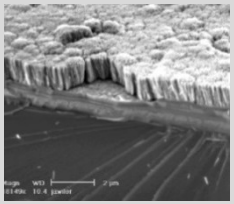
Nanomaterials for surface modification



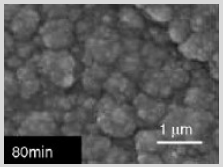
Pt black
1980 Pine



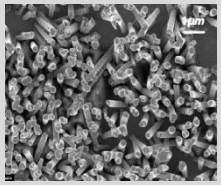
TiN
1998 Egert



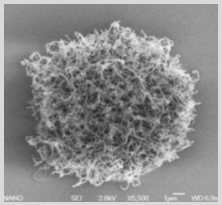
IrOx
2002 Weiland



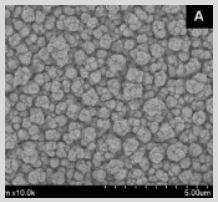
PEDOT
2004 Xiao



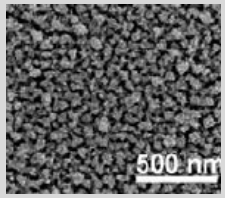
Au NW
2007 Yoon



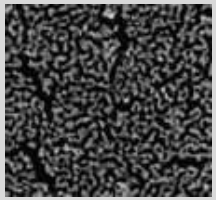
CNT
2009 Shein



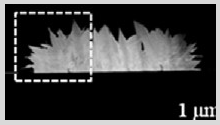
NP Pt
2010 Park



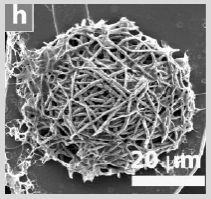
Layered Au NPs
2012 Zhang



NPG
2010 Seker

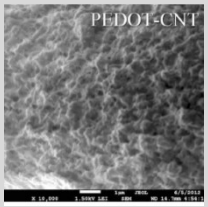


Au nanoflake
2010 Kim

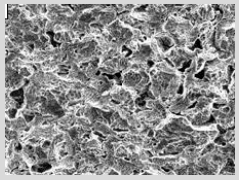


CP
2010 Abidian

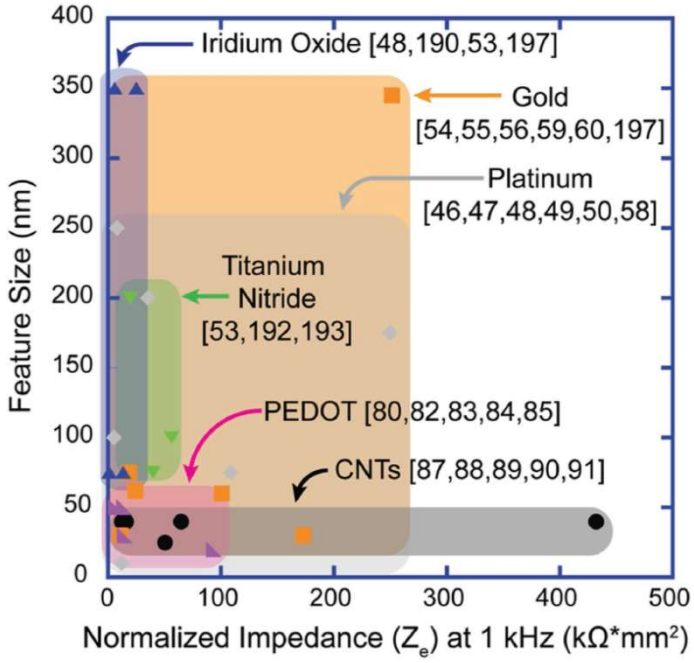
CNT + Au NP
2014 Zhang



PEDOT + CNT
2014 Castagnola



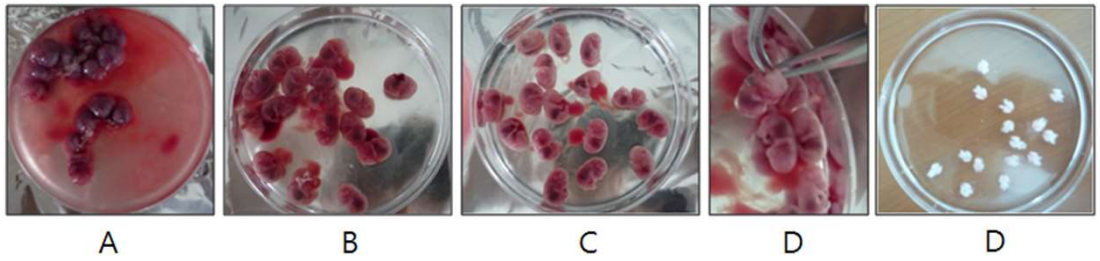
Ir-NiO
2014 Stilling

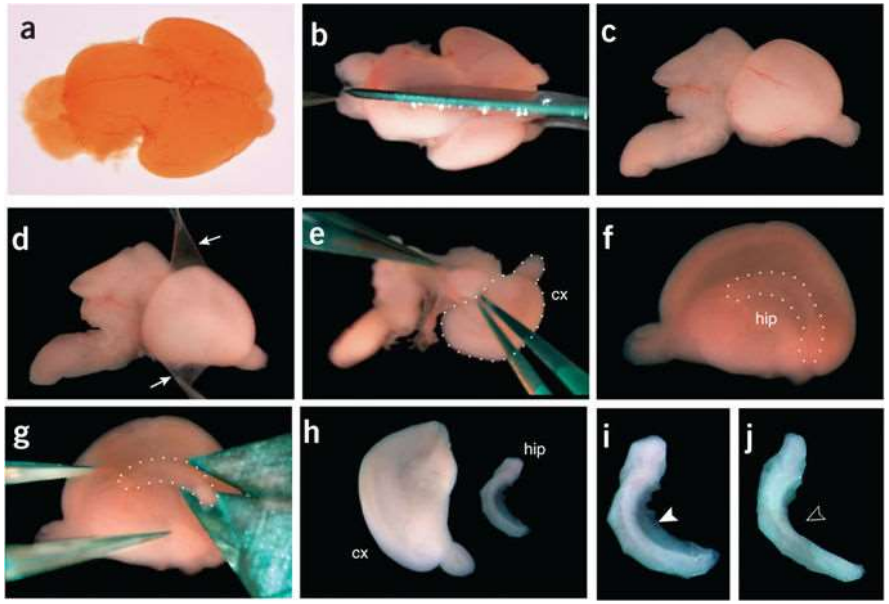


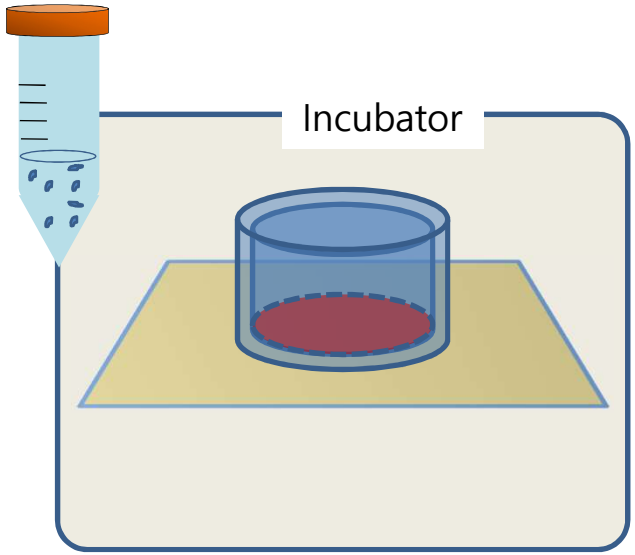
from Chapman & Seker (2017)

Primary neuronal cell culture

- **SD rat**







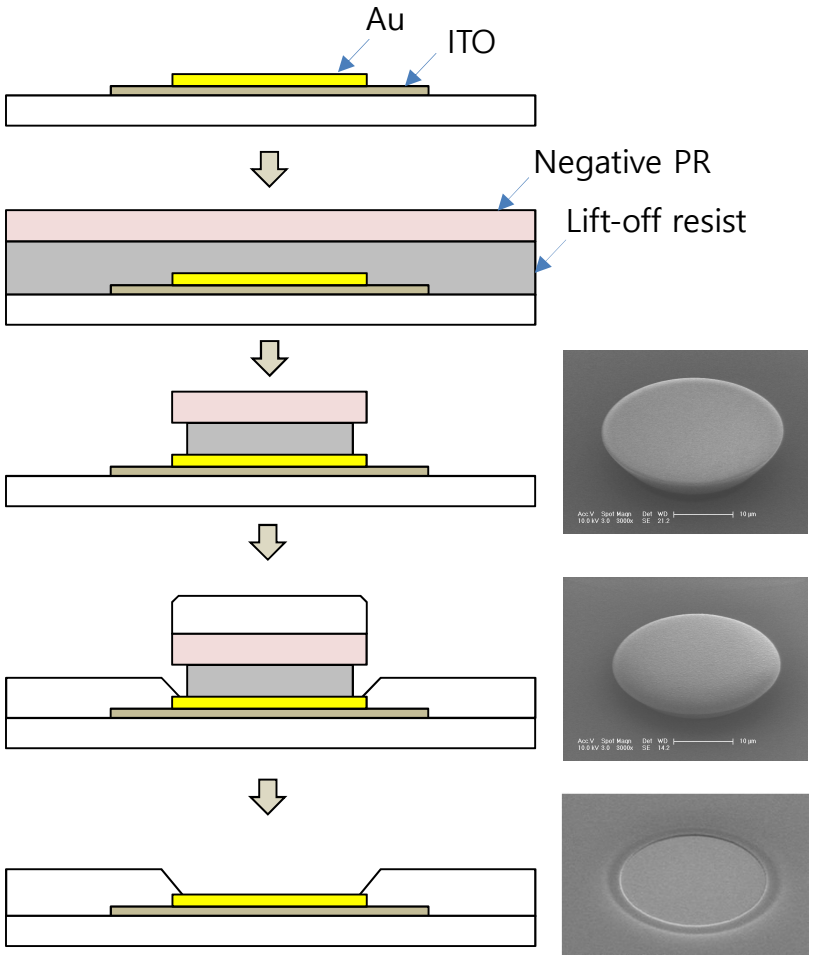
Electrode Materials for Neural Interface

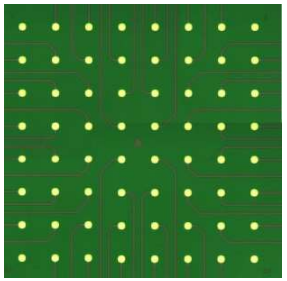
- **Five criteria***
  - Tissue response
  - Allergic response
  - Electrode-tissue impedance
  - Charge injection capability
  - Radiographic visibility
- **Metals**
  - List of biocompatible metals
    - Au, Pt, Pt-Ir, stainless steel, Pd, W, Pt-Rh, Cr-Mo, Au-Ni-Cr, Au-W, Ti, IrOx,
  - List of improper metals
    - Fe, Cu, Ag, Co, Zn, Mg, Mn, Al, Bi, Cd, Ni
  - Hierarchy of allergenic metals
    - Be > Hg > Cu > Au > Ag
  - **Best candidates as implants**
    - Au, Pt, W, Rh, Pd, Ti
  - **Choice for stimulating electrodes**
    - Pt, Pt-Ir, Au, W, Rh
- **Non-metals**
  - Organic materials
    - CNT, conducting polymers
  - Inorganic materials
    - ITO, IrOx,

- **Nanostructures**
  - Nanoparticles
  - Nanorods
  - Nanowires
  - Nanoflakes
  - Nanoporous structures

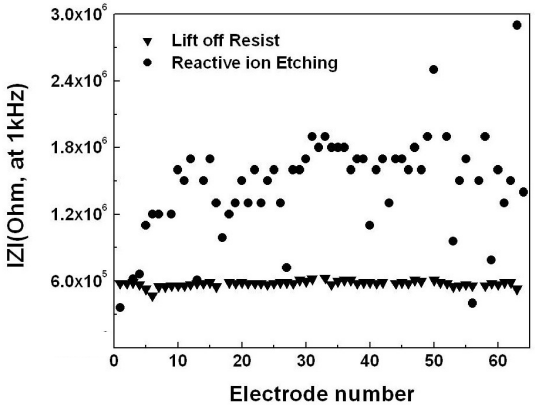
Fabrication of MEA

- **Bi-layer lift-off resist technique**
  - Lift-off resist + Negative photoresist + Sputter deposition of SiO_2





Excellent uniformity in impedance

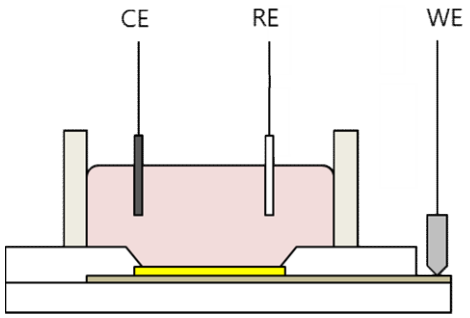


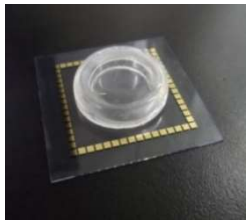
Y.H. Kim et al., Fabrication of multi-electrode array platforms for neuronal interfacing with bi-layer lift-off resist sputter deposition, J. Micromech. Microeng. 23, 097001 (2013)

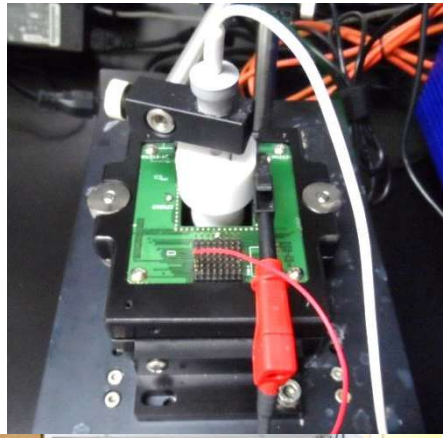
Y.H. Kim et al., Optimization of bi-layer structure formation and SiO_2 sputter-deposition process for fabrication of gold multi-electrode array, RSC Advances 5, 6675 (2015)

Electrodeposition of metallic nanoparticles

- **Typical 3-electrode configuration**
  - MEA electrode (working), Pt foil (counter), Ag|AgCl (reference)

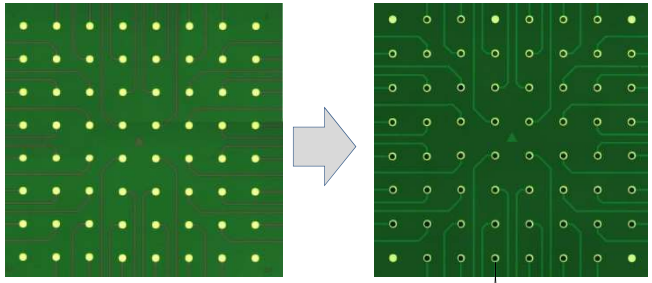


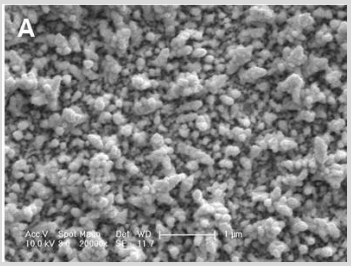




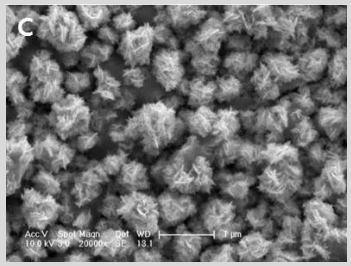
- **Electrochemical characterization**
  - Electrodeposition
  - Electrochemical impedance spectroscopy (EIS)
  - C-V
  - voltage transient

- **List of electrodeposited metallic nanostructures**

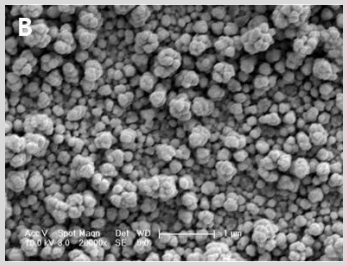




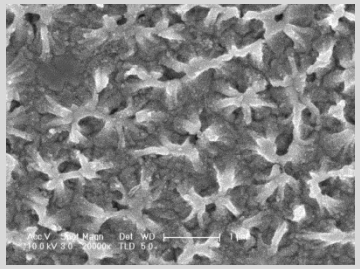
Au NPs



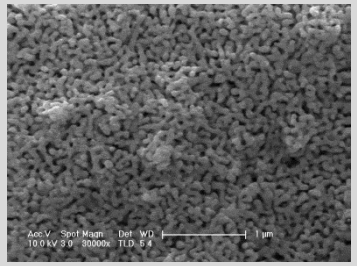
Pt NPs



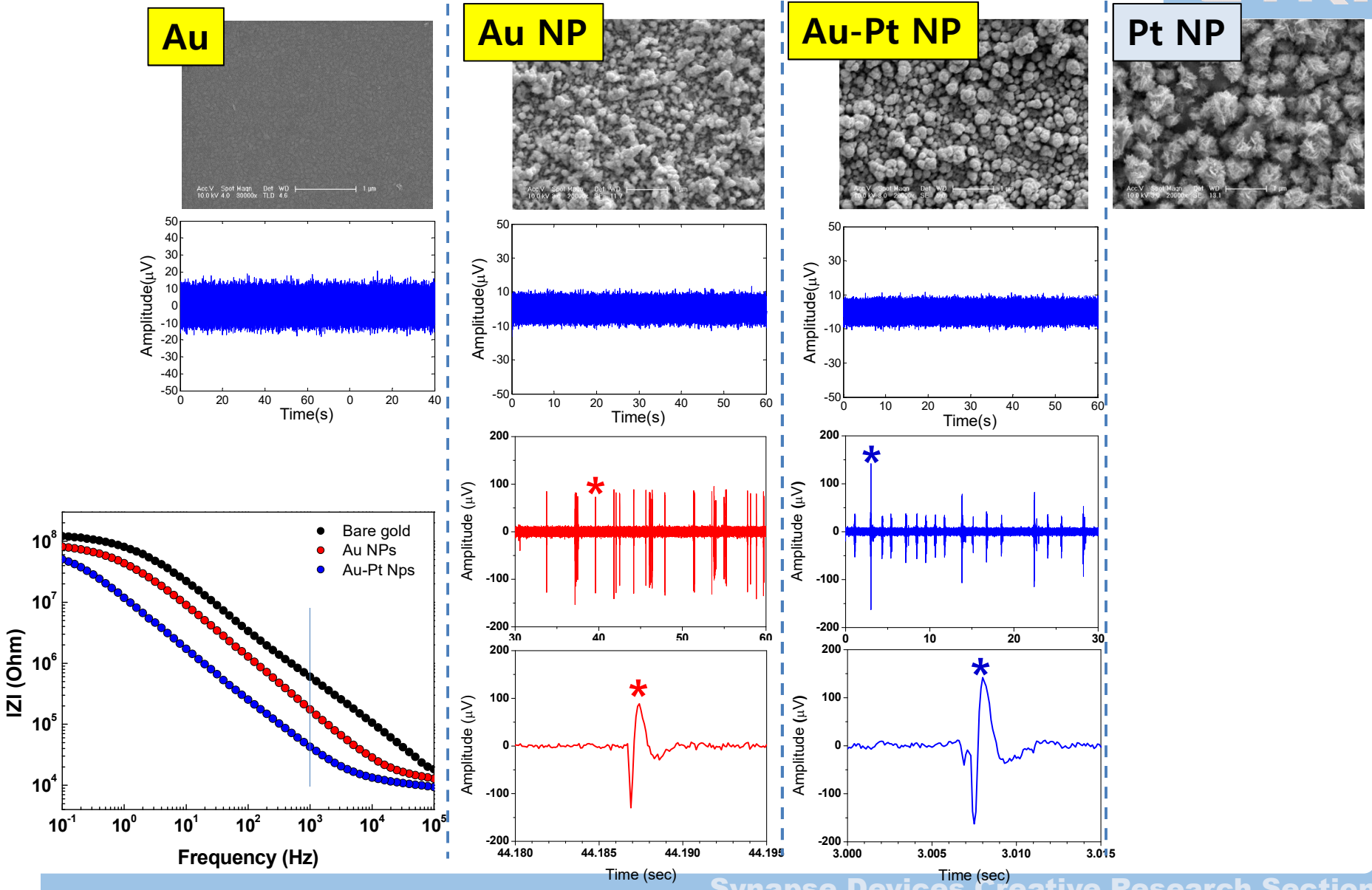
Au-Pt NPs



Pt black

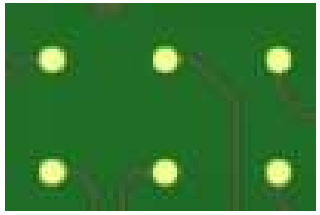


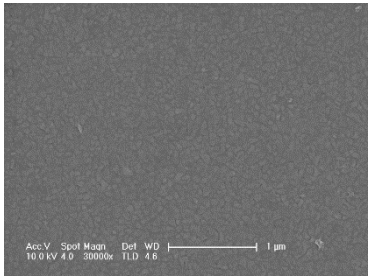
NPG


Au
Au NP
Au-Pt NP
Pt NP
Amplitude(μV)
Time(s)
Bare gold
Au NPs
Au-Pt Nps
IZI (Ohm)
Frequency (Hz)
Amplitude (μV)
Time (sec)

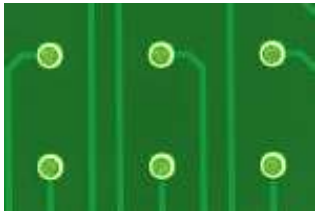
Modification with nanoporous Au (NPG)

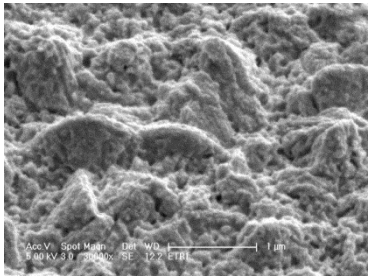
Au MEA



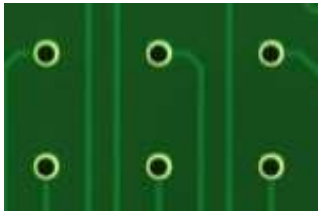


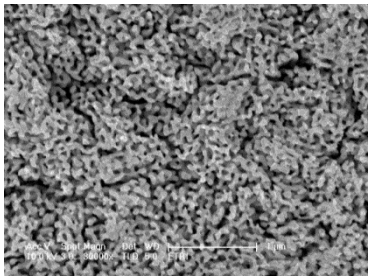
Electro-co-deposition of Ag:Au on Au

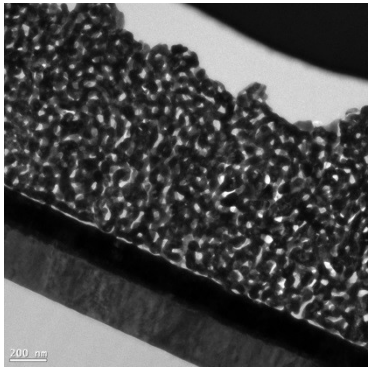




Chemical leaching of Ag in conc. Nitric acid @ 70 ℃

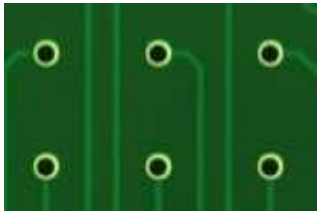


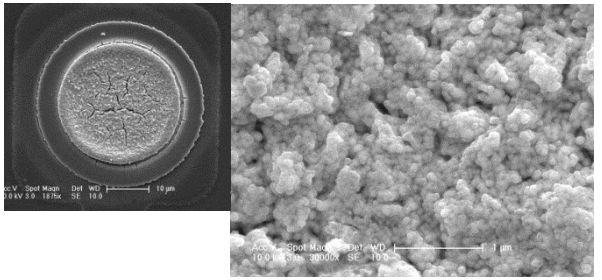


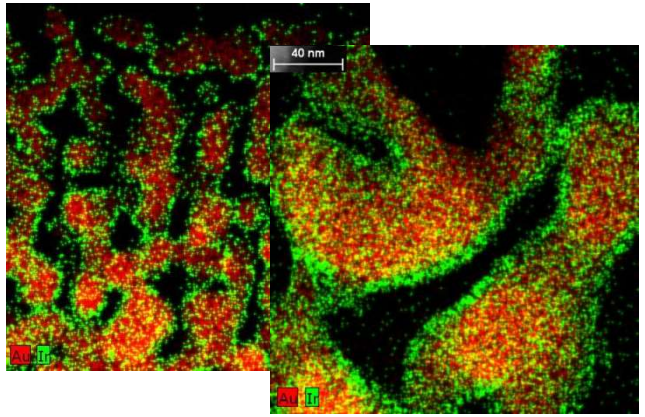


NPG

Electrodeposition of IrOx on NPG

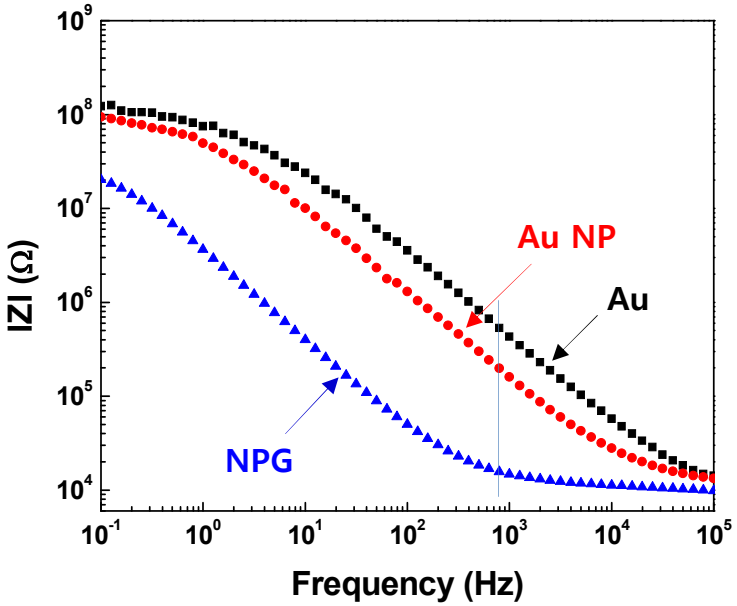


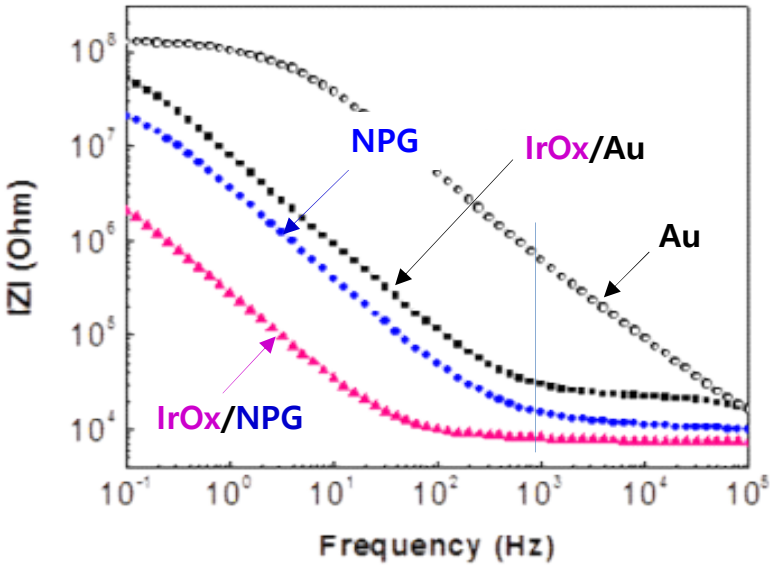


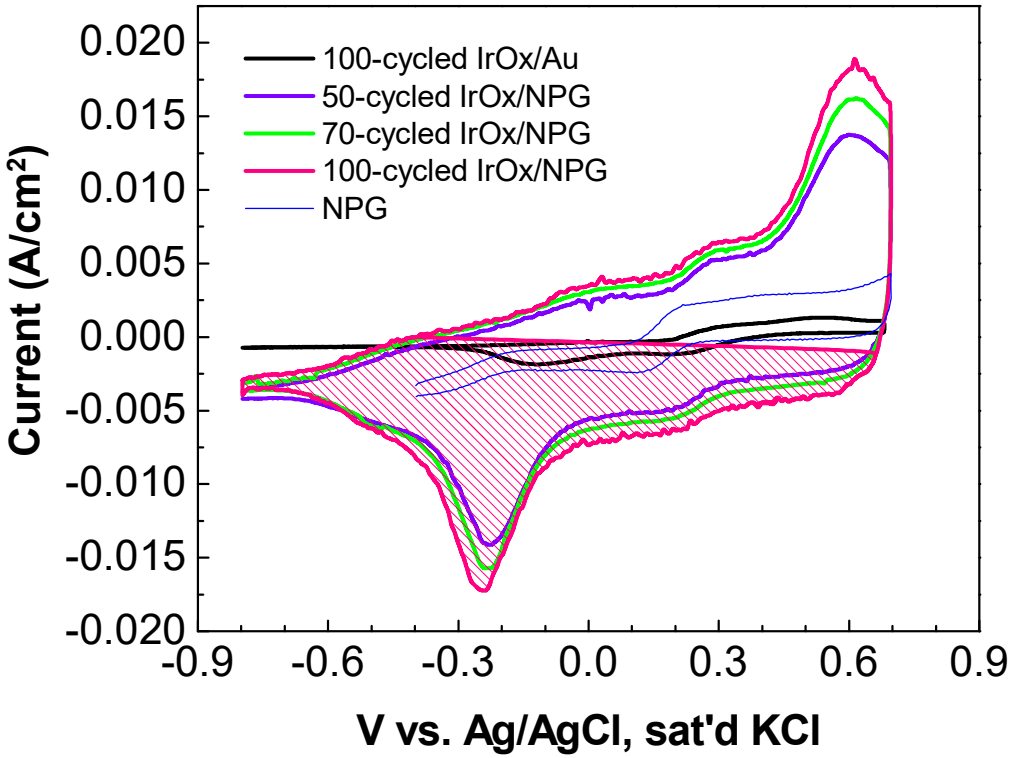


Y.H. Kim et al., *In vitro* extracellular recording and stimulation performance of nanoporous gold-modified multi-electrode arrays, J. Neural Eng. 12, 066029 (2015)

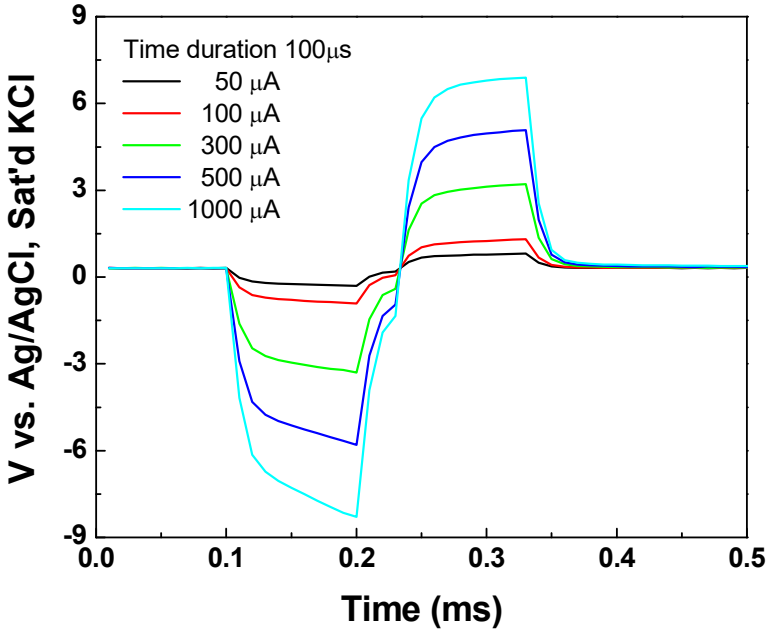
Y.H. Kim et al., Iridium oxide-electrodeposited nanoporous gold multielectrode array with enhanced stimulus efficacy, Nano Lett. 12, 066029 (2016)


|Z| (Ω)
Frequency (Hz)
Au NP
Au
NPG


|Z| (Ohm)
Frequency (Hz)
NPG
IrOx/Au
Au
IrOx/NPG


Current (A/cm²)
V vs. Ag/AgCl, sat'd KCl
100-cycled IrOx/Au
50-cycled IrOx/NPG
70-cycled IrOx/NPG
100-cycled IrOx/NPG
NPG

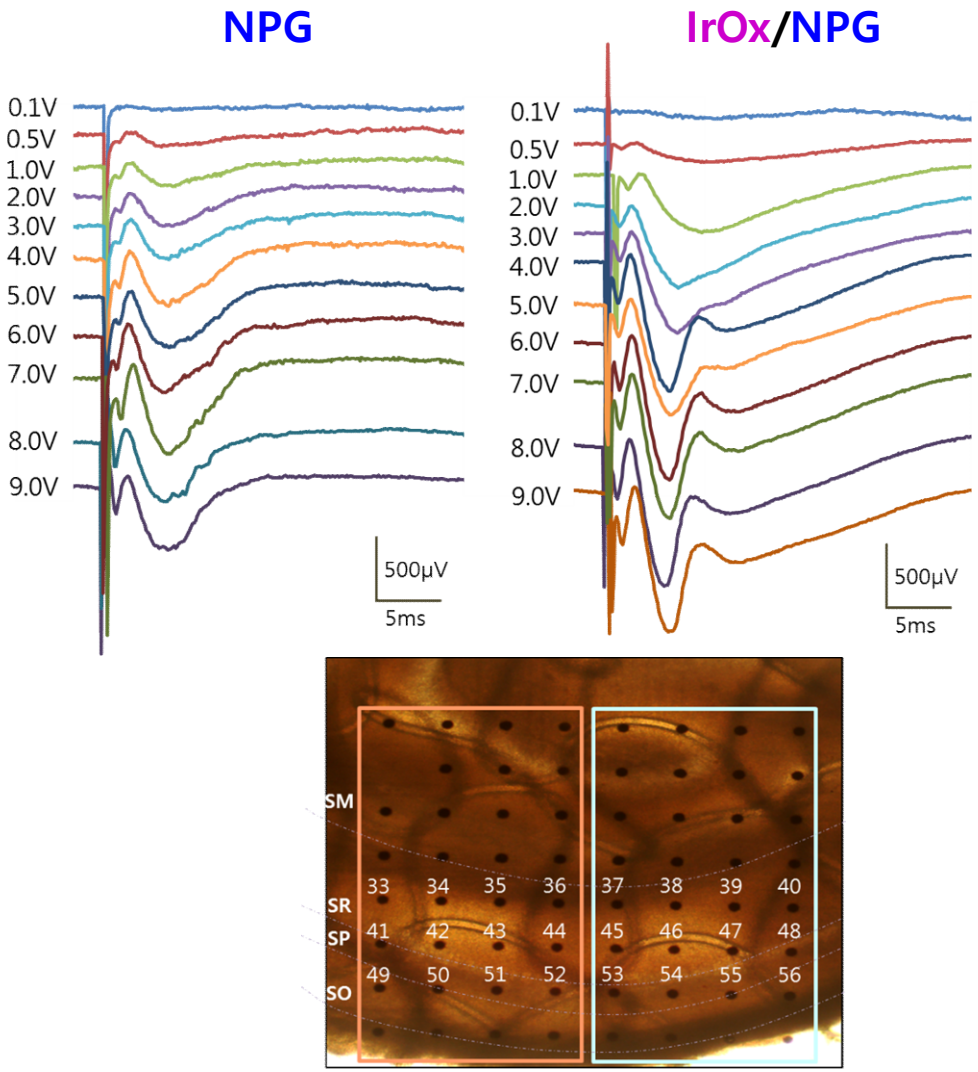
- **Cont'd**
  - Cathodic charge storage capacitance (cCSC) vs. charge injection limit
  - Derived from voltage transient measurement
  - Water window, -0.6 V

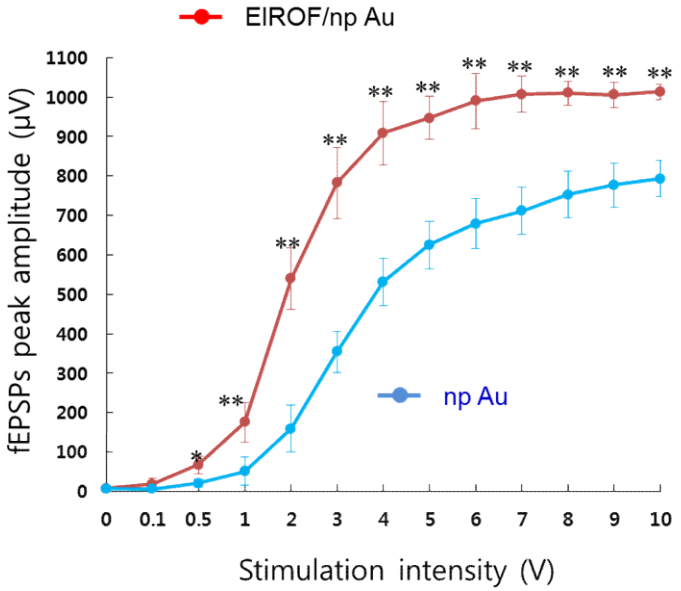


Material	cCSC (mC/cm²)	Charge injection limit (mC/cm²)	Efficiency (CIL/cCSC)
Pt		0.1-0.35, 0.05-0.15	
Au	0.27		
Pt black	16		
TiN		0.87	
PEDOT		2.3±0.6	
Roughed Pt	>8.9	1.0	
CNT	1.6	1-1.6	
EIROF	**23.54**, 16, 25	**1.27**	**0.054**
SIROF	36.15, 54, 31.5±6.6	2-3, 4.6±1	**0.13**
NPG	**1.0**	**0.98**	**~1**
IrOx/NPG	**8.8**	**2.3**	**0.26**

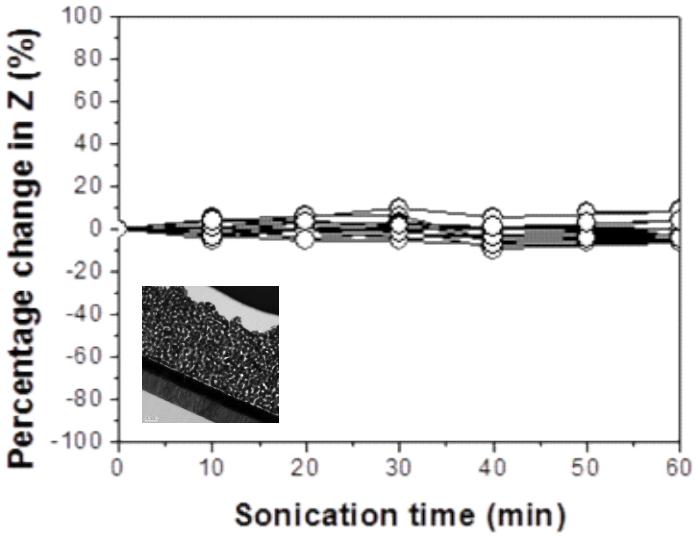
  - The charge injection limit is defined as the maximum quantity of charge that an electrode can inject before reaching the water electrolysis potential

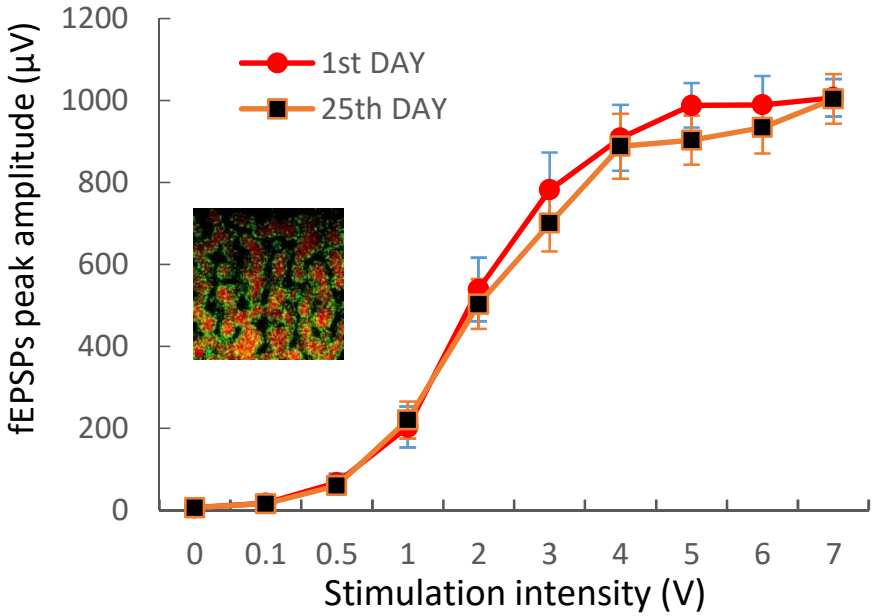
- **Stimulation performance**





▪ Durability test

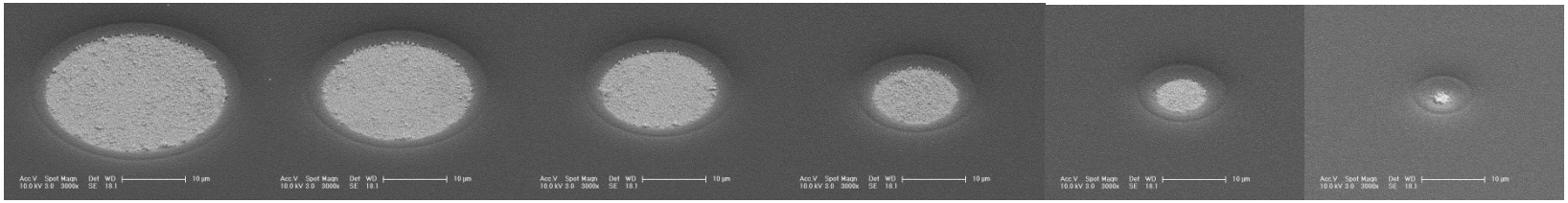


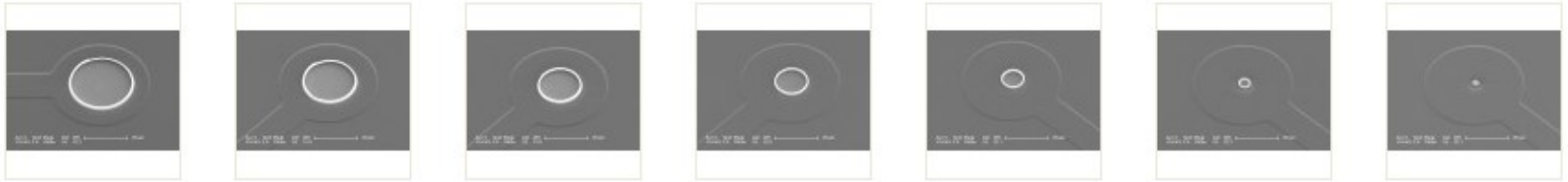


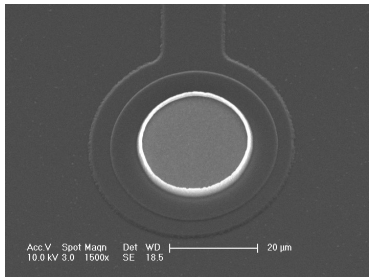
- Excellent mechanical durability
- Some MEA manufacturers recommend 'Do not apply sonication'

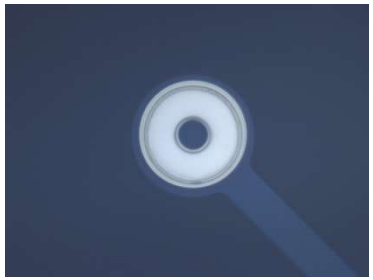
- 8 hours a day, 25th day of use
- Excellent anti-corrosion ability ?

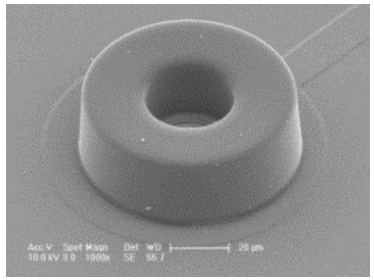
- **Flexibility of LOR passivation technique**





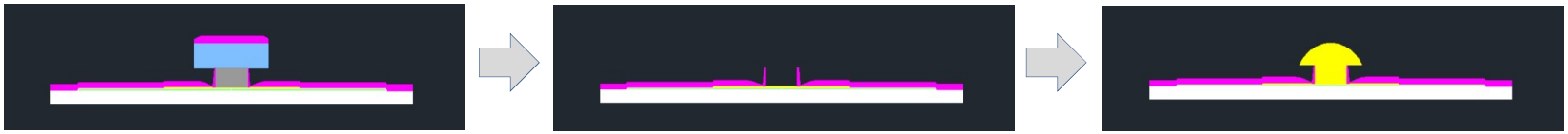


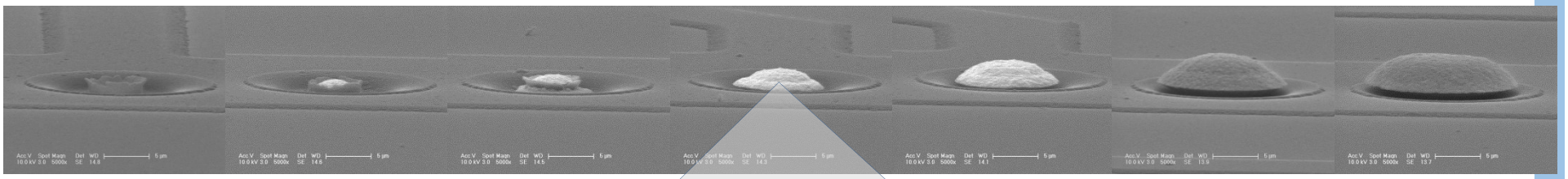


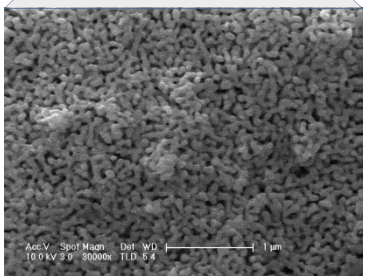


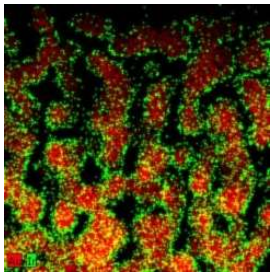
NEXT

- **Mushroom-type MEA for slice tissue interfacing**
- **LOR passivation + electro-co-deposition of Ag:Au alloy**



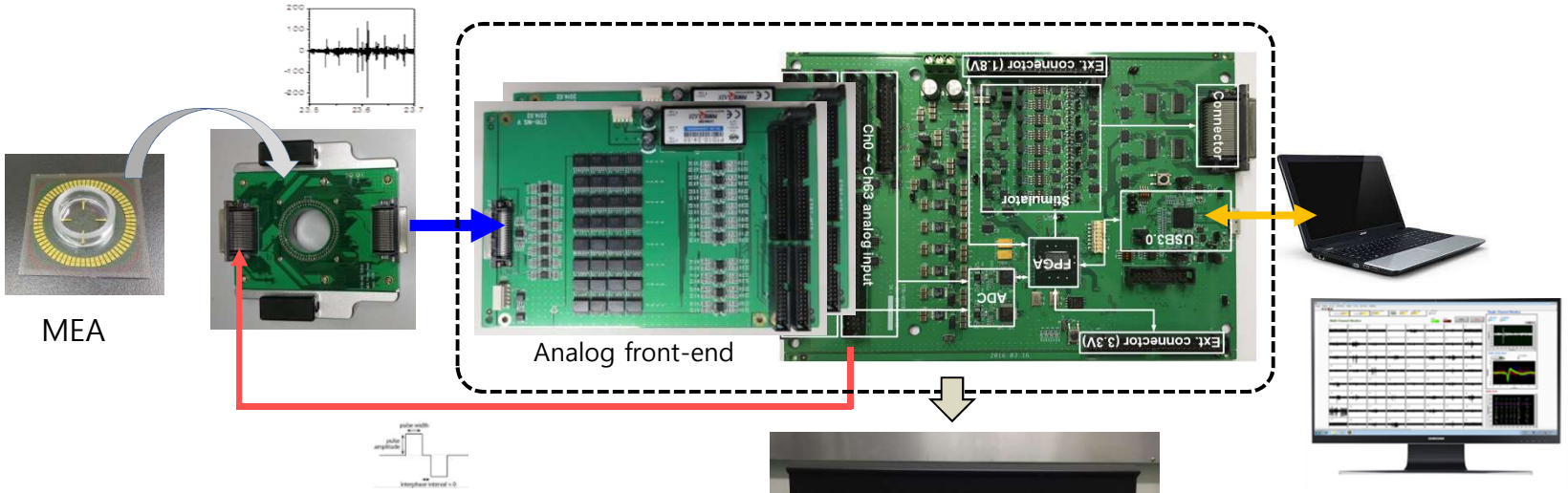




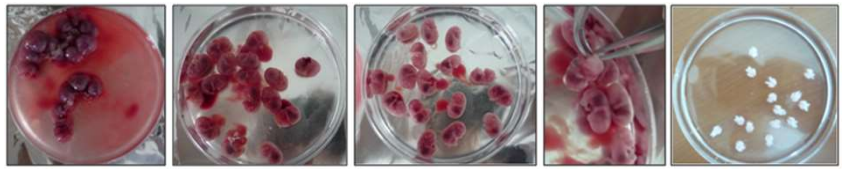


64 & 128 CH MEA System

- **FPGA-based 128 CH bi-directional MEA system**

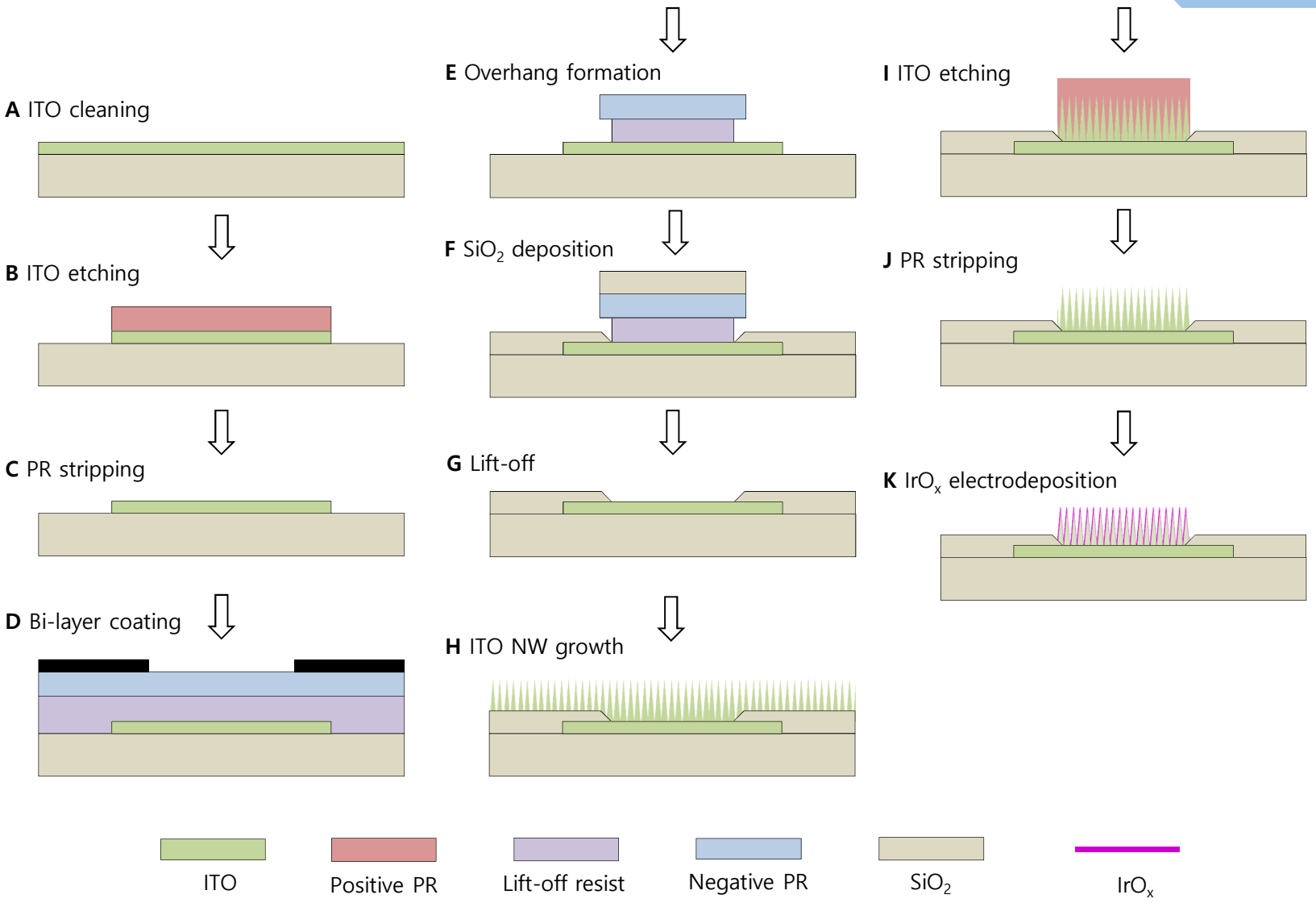


- 128 channel recording (4.16 MSamples/s)
- Real-time online spike sorting (feature learning & extraction capability)
- 8 channel arbitrary voltage and current stimulation



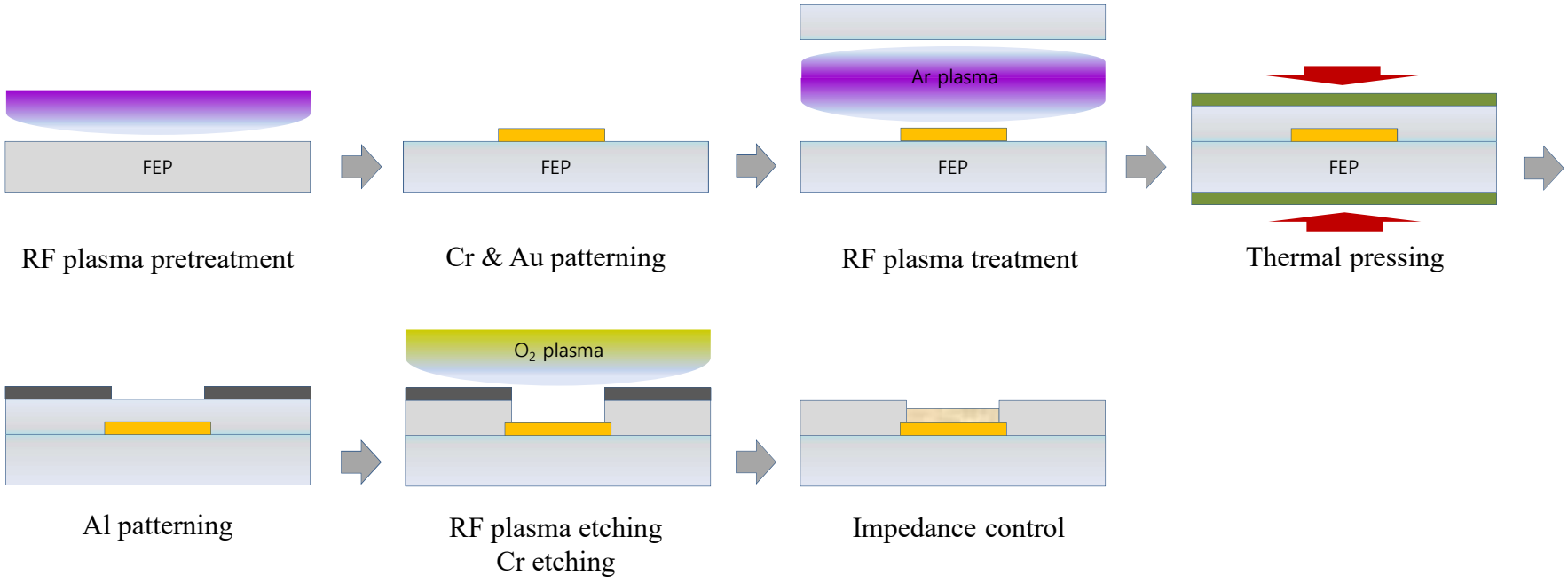
J. Park et al., A 128 channel FPGA-based Real Time Spike Sorting Bidirectional Closed-loop Neural Interface System, IEEE Transactions on Neural Systems & Rehabilitation Engineering, Vol. 25, 2227-2238 (2017).

All metal-oxide-based MEAs



Fabrication of flexible electrode

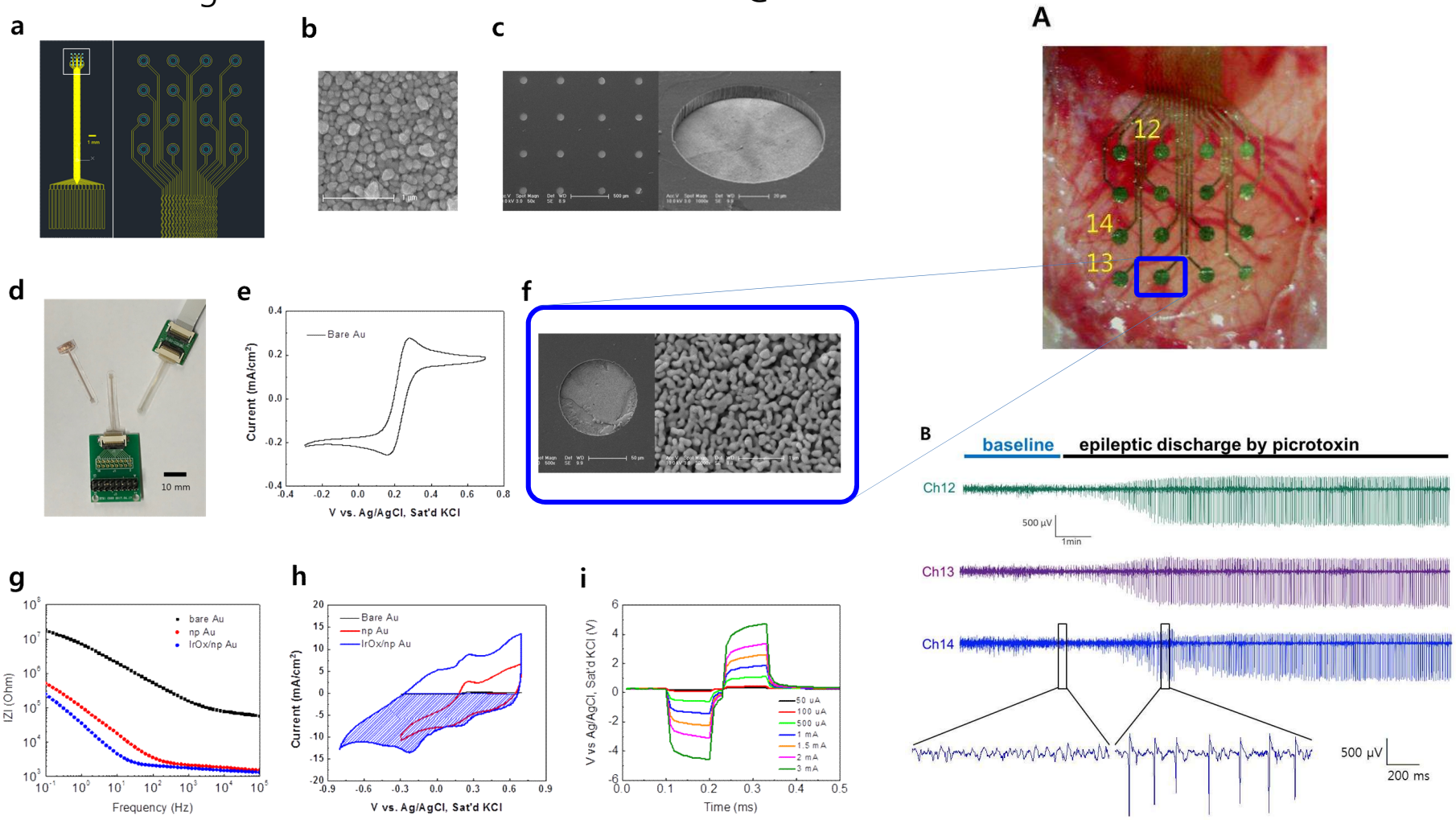
- **Fluoropolymer-based flexible electrode**
  - **Fluorinated ethylene propylene (FEP): m. p. ; Tg**
  - **FEP plasma treatment and thermal pressing** beyond the meting temperature
  - Solely composed of FEP and Au without adhesion metal



Y.H. Kim et al., Fluoropolymer-based flexible neural prosthetic electrodes for reliable neural interfacing, ACS Appl. Mater Interfaces, Vol. 9,43420-43428 (2017).

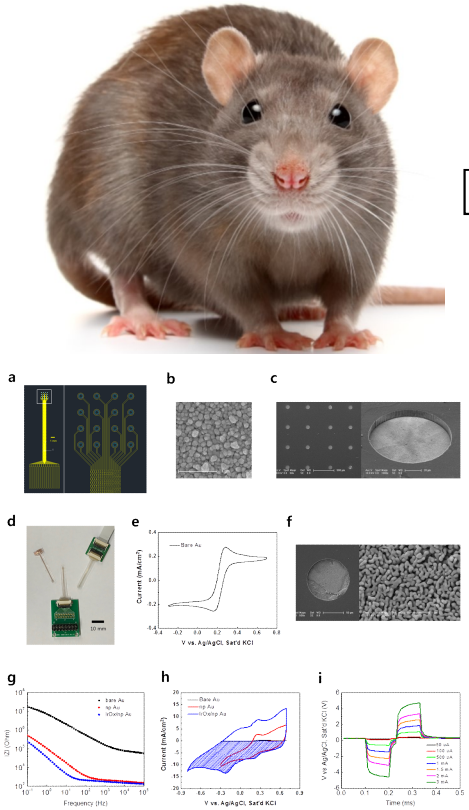
16-CH ECoG electrode array

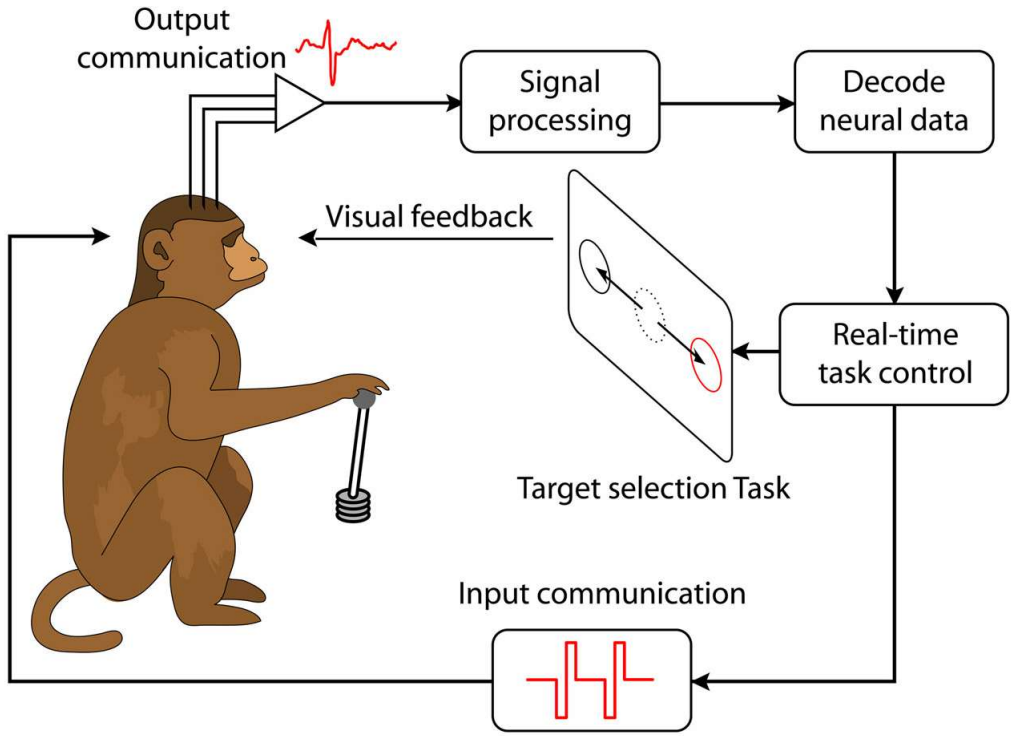
- **Excellent chemical stability**
  - Longer than an hour in conc. Nitric acid @ 70 ℃



Co-work Program

- **Reliability test with primate platform**






THANK YOU
FOR ATTENTION!