MoS$_2$ growth and device technology
Towards integration with multiplexed graphene sensors arrays

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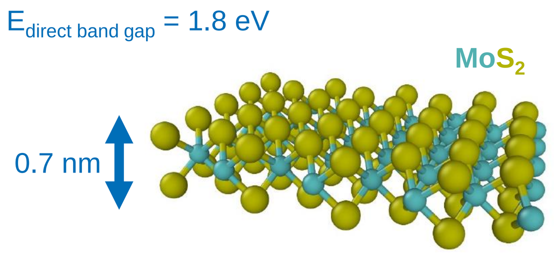
Motivation

1. More Moore – Beyond Si CMOS scaling

- Si nanosheets present charge scattering
- MoS$_2$ has higher intrinsic mobility than similar scale Si nanosheets.

Atomic-thin semiconductor
$E_{direct\ band\ gap}$ = 1.8 eV



2. More than Moore – New functionalities such as flexible electronics

- This work: MoS$_2$ Field Effect Transistors (FETs) on flexible biocompatible probes for a multiplexed sensing read-out.

Graphene based solution-gated FETs are the only technology that has been demonstrated to record ultra-slow brain electrophysiological activity.

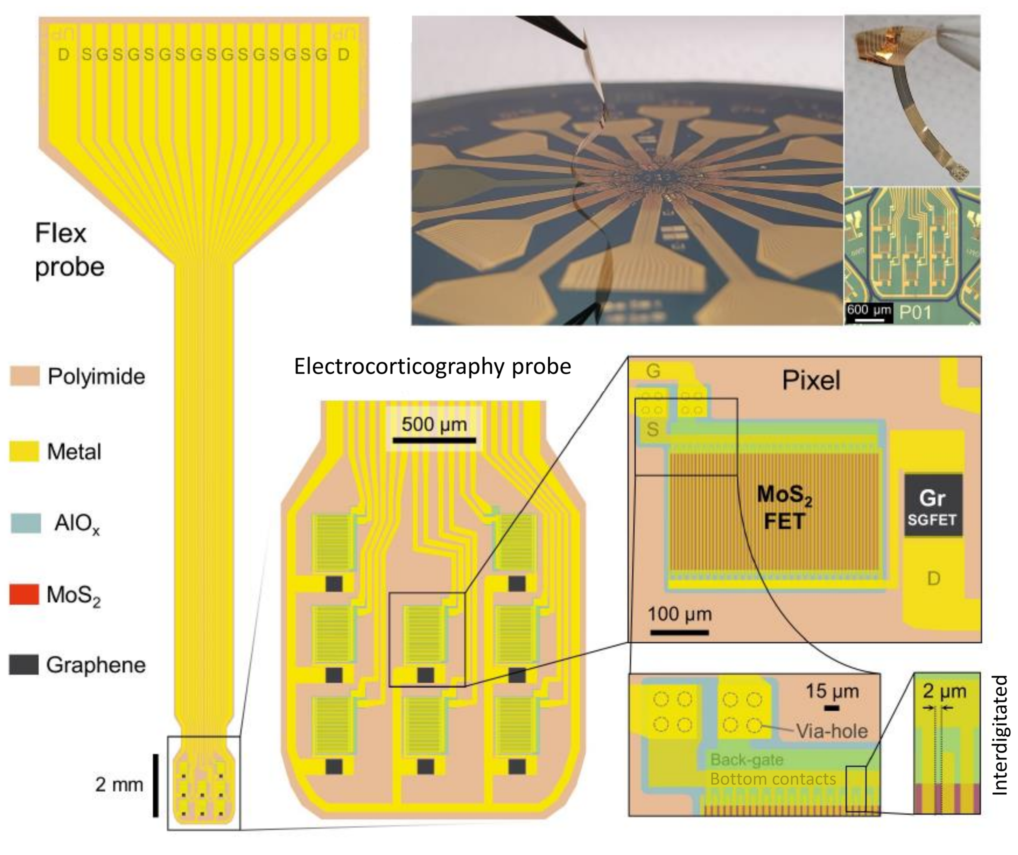
Need: Large matrix of sensors with a reduced cable footprint.

Solution: Monolithic integration of MoS$_2$ FETs.

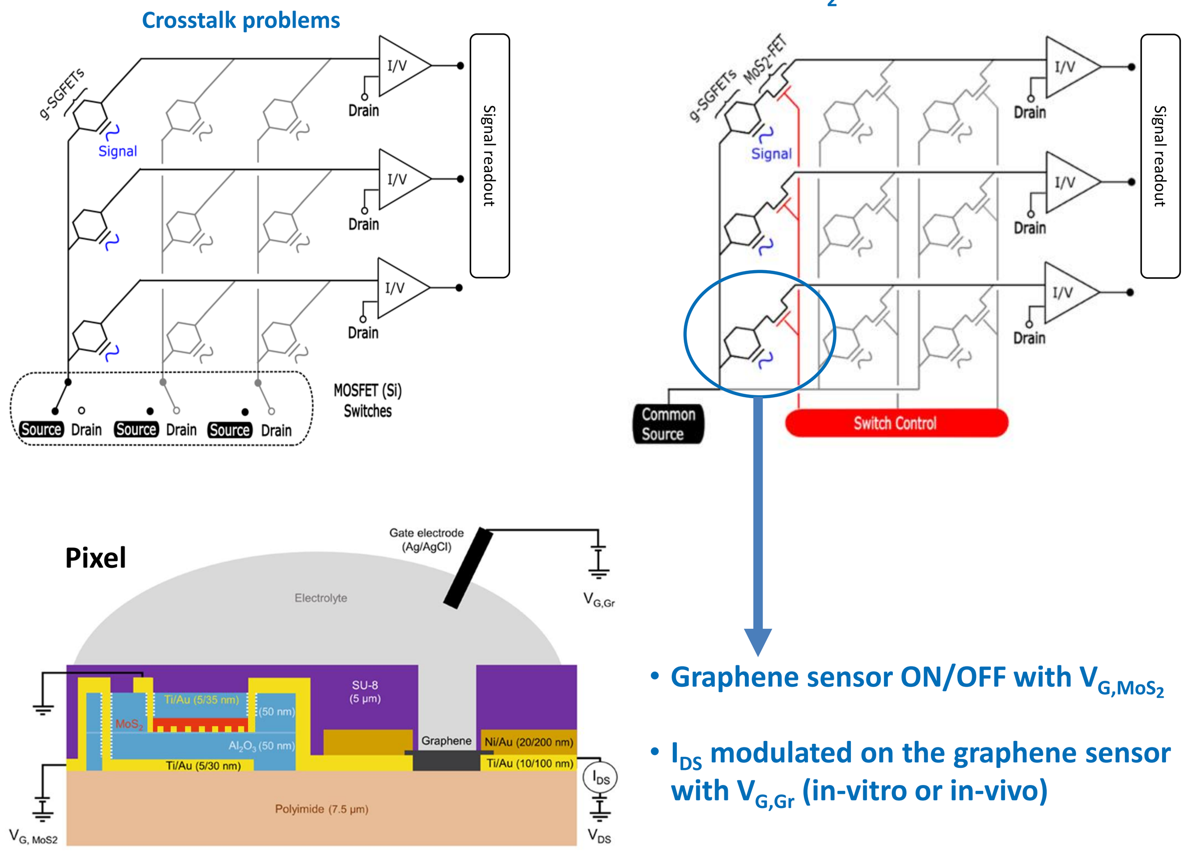
MoS$_2$ technology requires advancements to reach manufacturing readiness, covering everything from growth to device integration.

Multiplexed graphene sensors

Monolithic integration of MoS$_2$ FETs and graphene solution-gated FETs for high density neural recordings



Column-wise time-division multiplexing



- Graphene sensor ON/OFF with V_{G,MoS_2}
- I_{DS} modulated on the graphene sensor with $V_{G,Gr}$ (in-vitro or in-vivo)

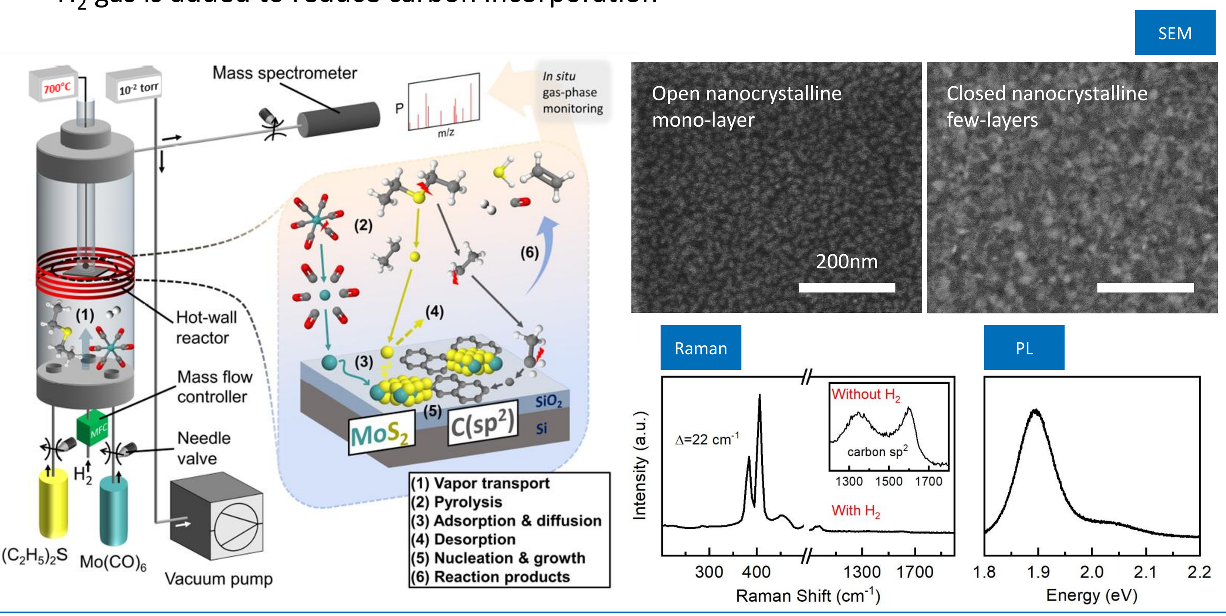
MoS$_2$ Field Effect Transistors Nanofabrication Challenges

MoS$_2$ growth by Metal-Organic CVD

- Homogeneous large-scale synthesis (2x2 cm^2) without toxic H_2S gas
- Molybdenum hexacarbonyl and diethyl sulfide as gas phase precursors
- Mono- and few-layers nanocrystalline MoS$_2$ on SiO$_2$/Si

x Diethyl sulfide is prone to carbon incorporation, impacting the nucleation density, lateral growth and electrical properties.

✓ H_2 gas is added to reduce carbon incorporation



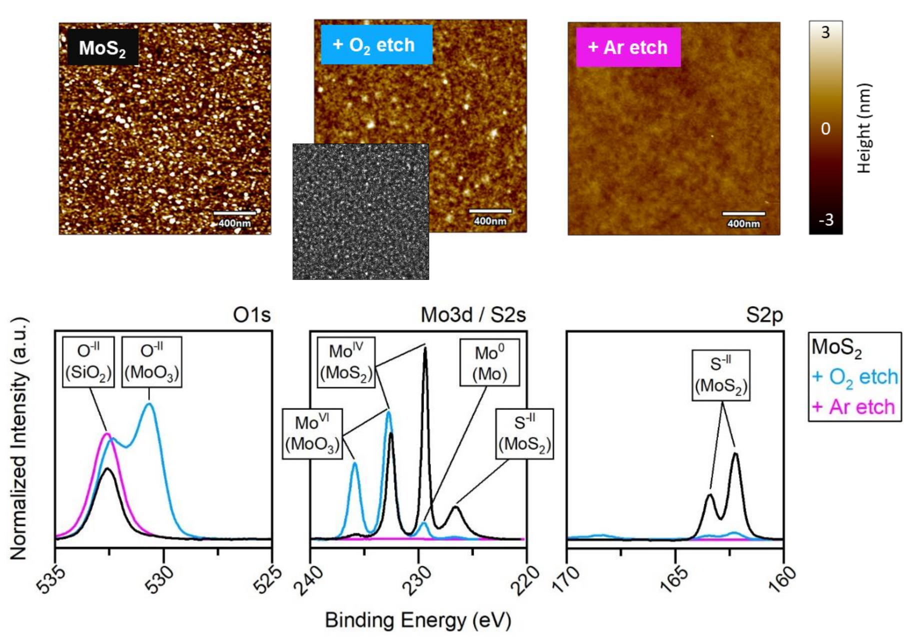
Induced Coupling Plasma - Reactive Ion Etching

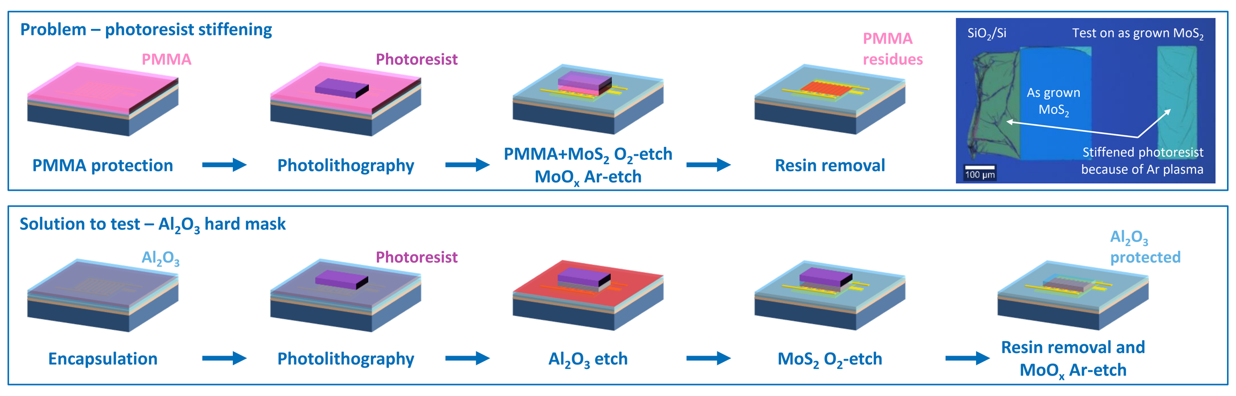
2

- MoS$_2$ easily oxidizes to MoO$_x$ with O$_2$ plasma
- MoO$_x$ residues are hard to remove with O$_2$ plasma and may not be noticed by eyesight

✓ Solution: MoO$_x$ can be removed with Ar plasma

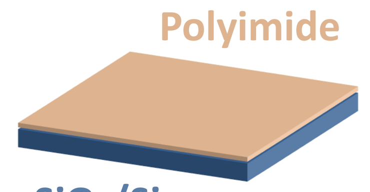
x Ar plasma stiffens the photoresist even for a decreased plasma power





Process flow

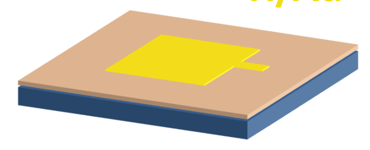
Polyimide



Flexible substrate

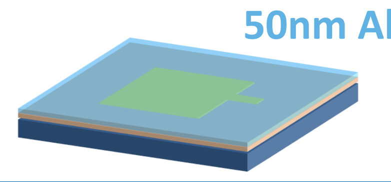
SiO$_2$/Si

Ti/Au

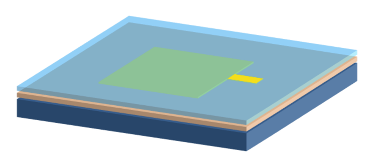


Gate electrode

50nm Al$_2$O$_3$

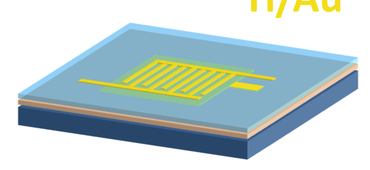


Gate dielectric (1)



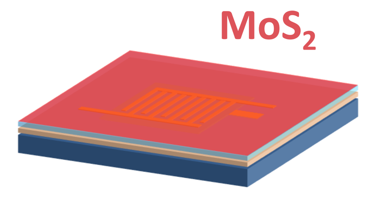
Via-hole etching

Ti/Au

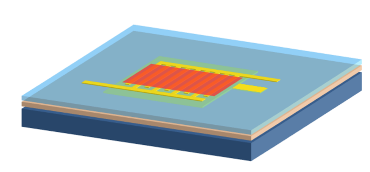


Interdigitated contacts

MoS$_2$

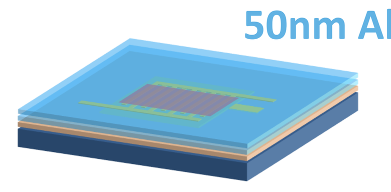


MoS$_2$ 4cm^2 transfer
- PMMA assisted:
  - Wet transfer
  - Semi-dry transfer

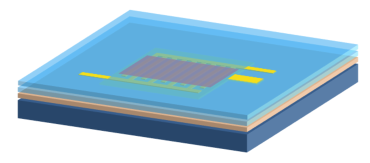


MoS$_2$ definition (2)

50nm Al$_2$O$_3$



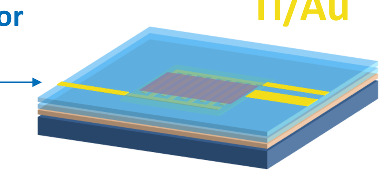
MoS$_2$ encapsulation (3)
- AlO$_x$ seed layer + Al$_2$O$_3$



Via-hole etching

Ti/Au

Graphene sensor

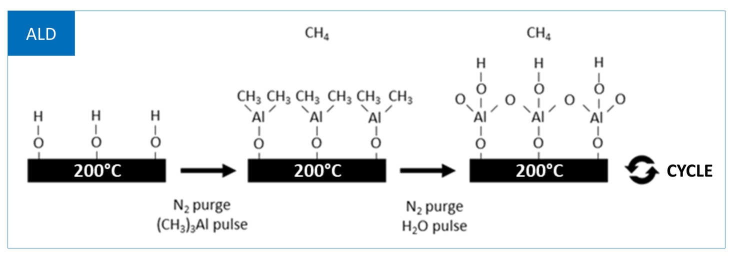


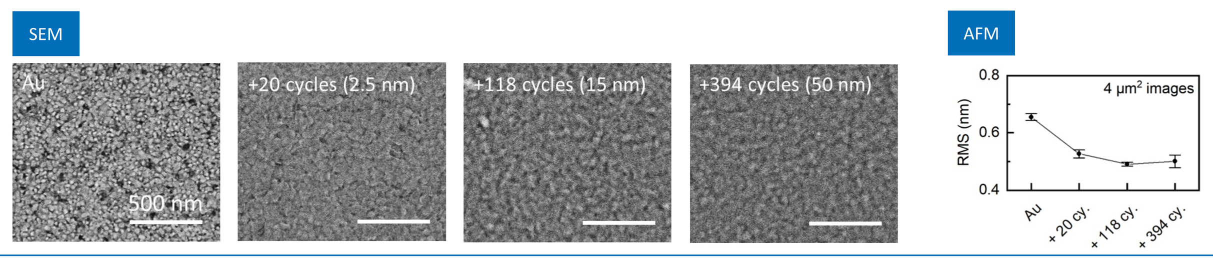
Metal path

Dielectric growth by Atomic Layer Deposition (ALD)

1

- Need for compatible high-K dielectrics such as Al$_2$O$_3$
- ALD allows conformal growth with atomic thickness control
- Trimethyl aluminium and water as gas phase precursors



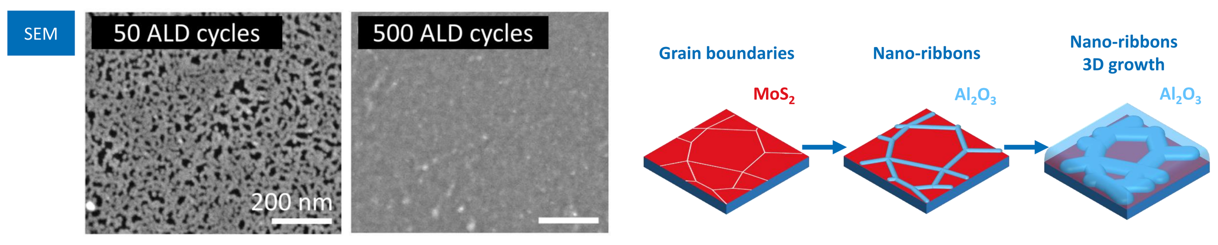


MoS$_2$ encapsulation with AlO$_x$ seed layers

3

x ALD on polycrystalline MoS$_2$ yields a no-uniform dielectric with pinholes

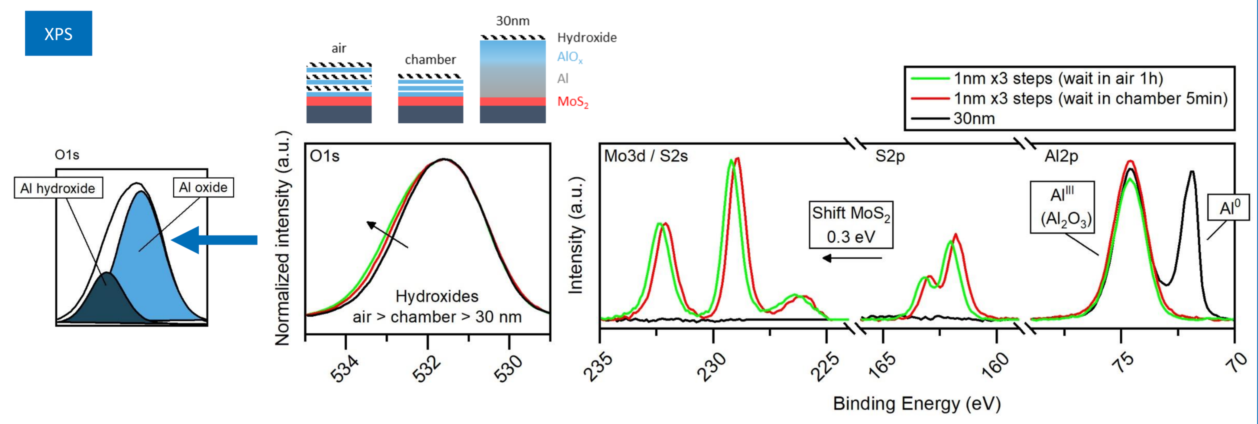
x Chemisorption on the dangling-bonds-free basal plane is inhibited



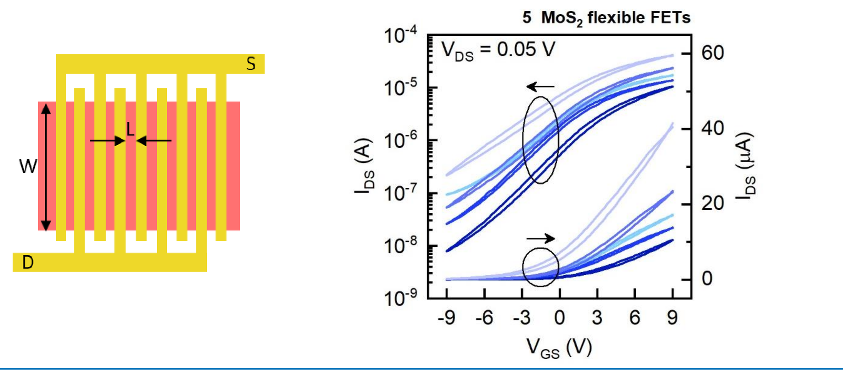
✓ Solution: prior to ALD, E-beam evaporation of Al seed layers (1nm x3), which oxidise upon air exposure or by the residual O$_2$ inside the evaporator chamber (10^{-7} Torr).

! AlO$_x$ causes charge transfer doping (O-vacancies) and introduces charge traps

! Al hydroxides formed upon H_2O-air exposure may also contribute



MoS$_2$ FETs performance prior to optimizations



W/L = 6000

ON/OFF ~ 10^3

Window ±9 V

Low subthreshold slope

Negative V threshold

Hysteresis

Future plan

- **MoS$_2$ growth:**
  Increase grain size – sodalime substrate and seed promoters
  Aixtron Black Magic Box– low temperature 4-inch growth compatible with polyimide and BEOL
- **Dielectric growth:**
  Reduce dielectric thickness and implement HfO$_2$ – thermal or plasma enhanced ALD
  Conductive AFM to address the dielectric quality
  Valence band analysis of the AlO$_x$ seed layers
- **Metal electrodes and contacts:**
  Determine the current contact resistance limitation – explore other metals and finger width effect
  Implement metal diffusion barriers (such as Pt or Ni) between Ti and Au

References

[1] Lemme M.C. et al., "*2D materials for future heterogeneous electronics*", Nature Communications 13, 1392 (2022).

[2] Masvidal-Codina E. et al., "*High-resolution mapping of infraslow cortical brain activity enabled by graphene microtransistors*", Nature Materials 18, 280–288 (2019)

[3] Schaefer N. et al, "*Multiplexed neural sensor array of graphene solution-gated field-effect transistors*", 2D Materials 7, 2, 025046 (2020).

[4] Schaefer C.M. et al., "*Carbon incorporation in MOCVD of MoS$_2$ thin films grown from an organosulfide precursor*", Chem. Mater 33, 12, 4474-4487 (2021).

[5] McClellan C. J. et al., "*High current density in monolayer MoS$_2$ doped by AlO$_x$*", ACS Nano 15, 1, 1587-1586 (2021).

Acknowledgements

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