

Laser-Induced Graphene and MXene on Polymer Substrates for Monitoring Physiological Parameters

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Center for Microelectronic Technologies

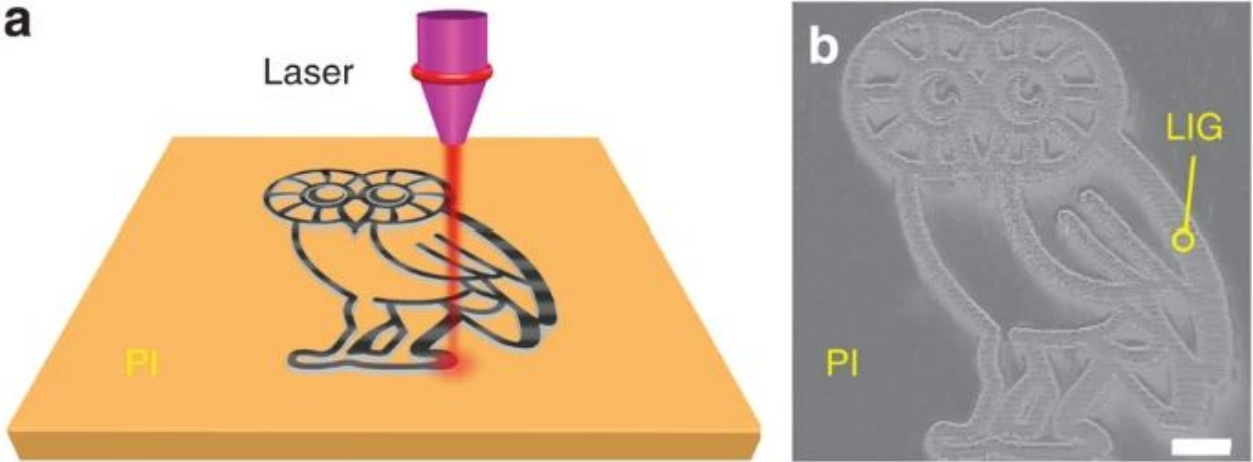
Institute of Chemistry, Technology and Metallurgy - ICTM

Outline

- Laser-induced graphene (LIG) as heartbeat and respiration monitor
- LIG on biocompatible sodium alginate
- LIG on PDMS/Triton composites
- LIG on PDMS/PEG
- LIG on PU networks
- LIG on PI
- MXene



Laser-induced graphene



LIG on Kapton, Lin et al, Nat Comm 5, 5714 (2014)

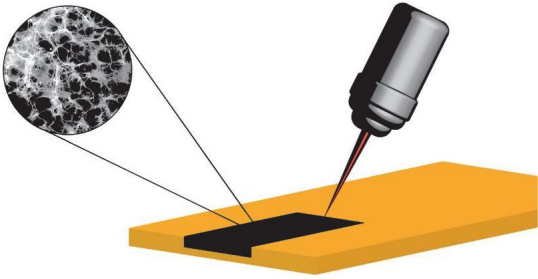


LIG on toast, Chyan et al, ACS Nano 12, 2176 (2018)

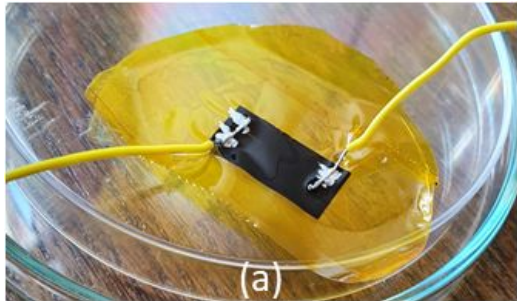


- a) Coconut
- e) Cork
- f) Potato skin

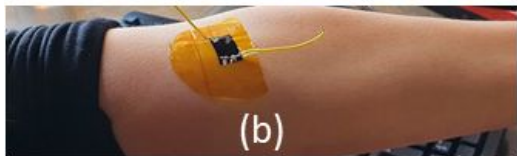
LIG as heartbeat monitor



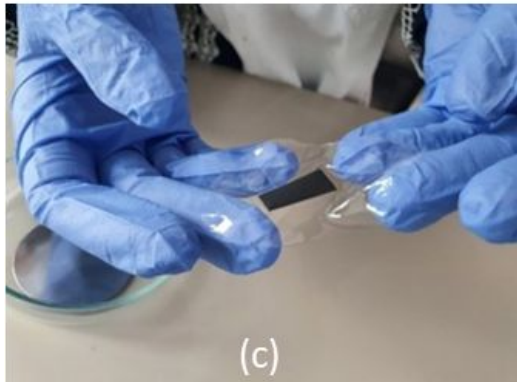
Laser
induction



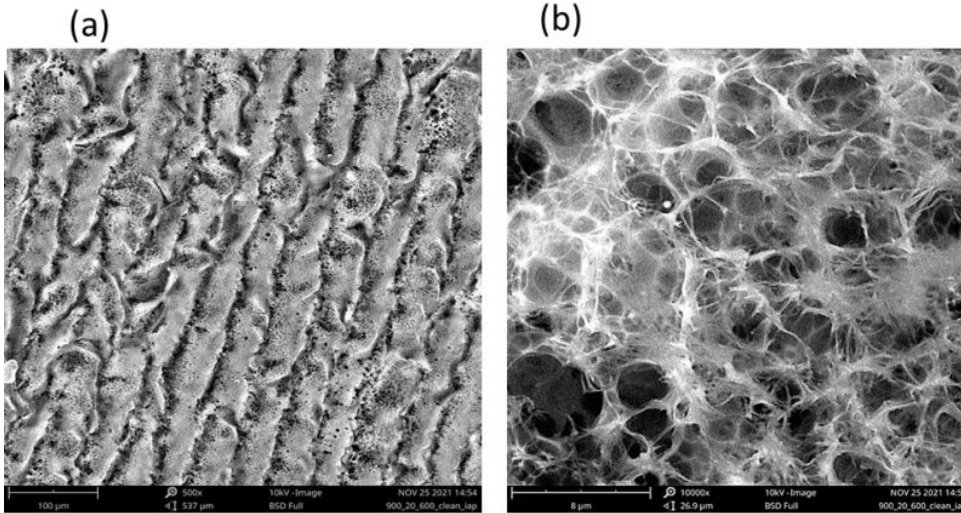
(a)



(b)

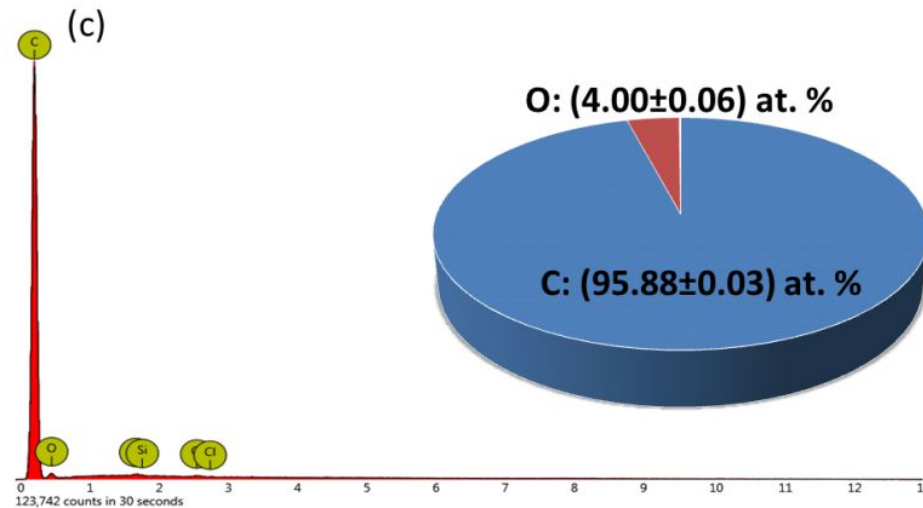


(c)



(a)

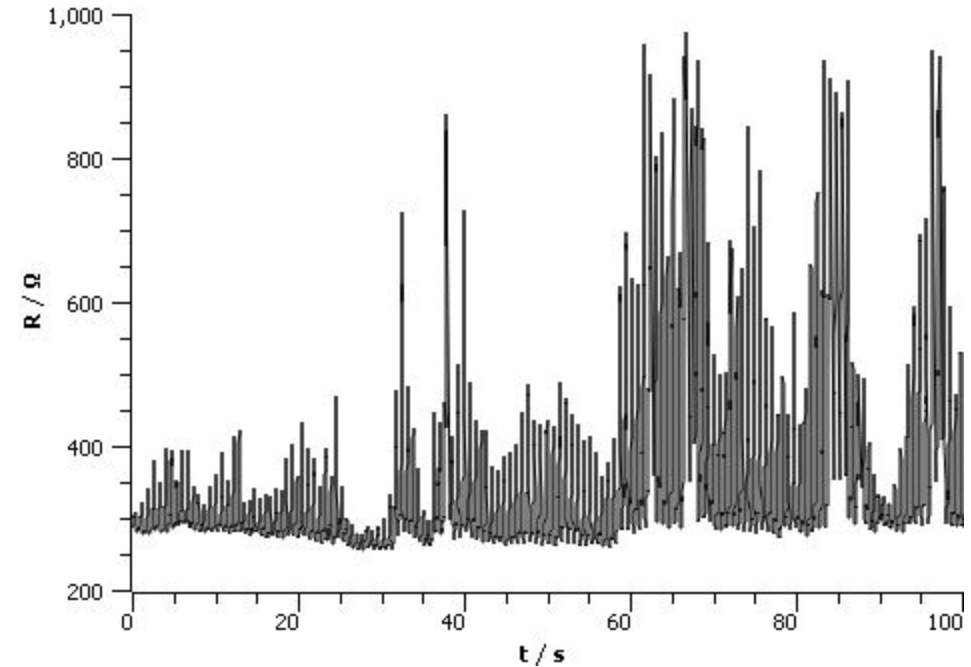
(b)



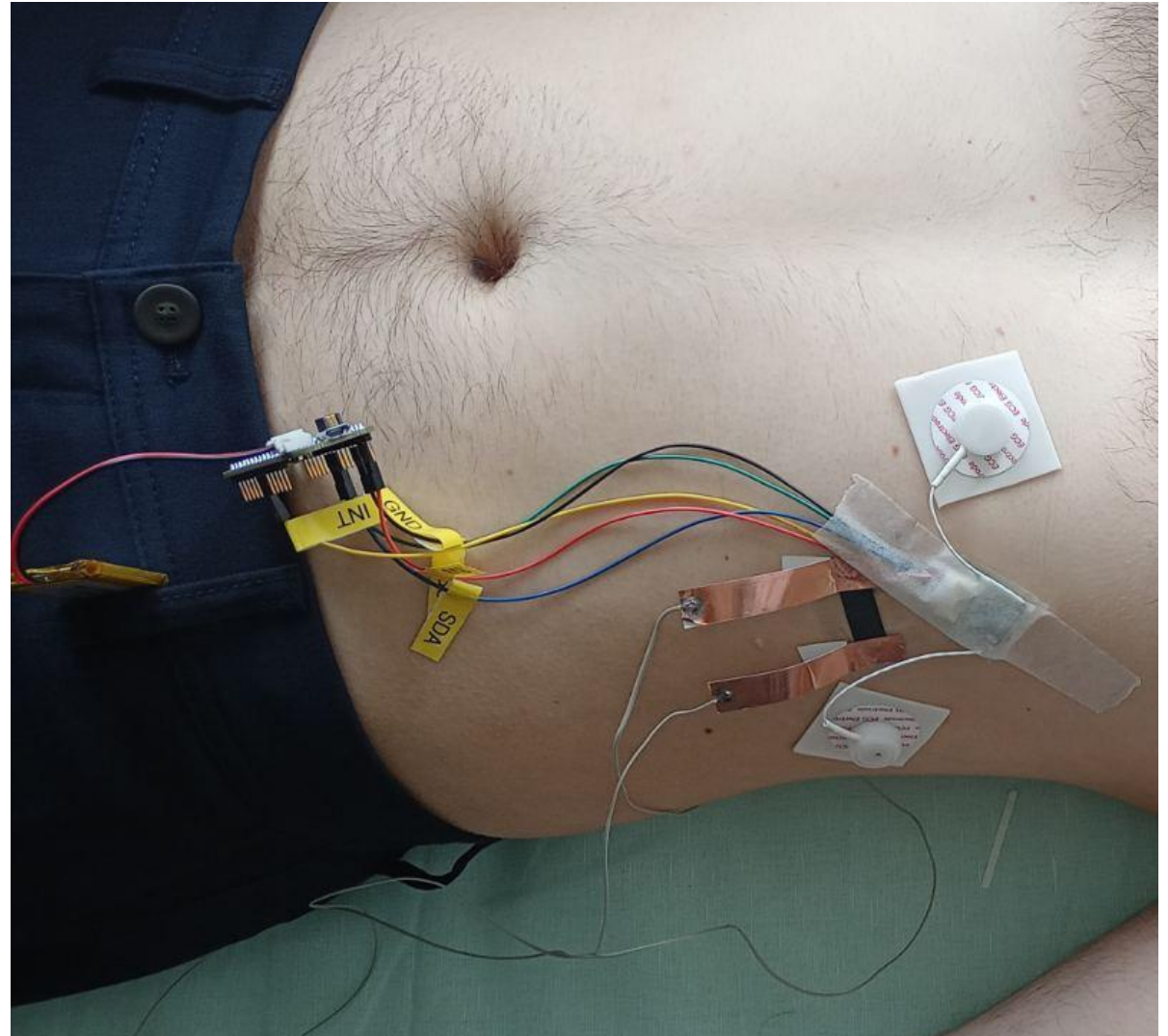
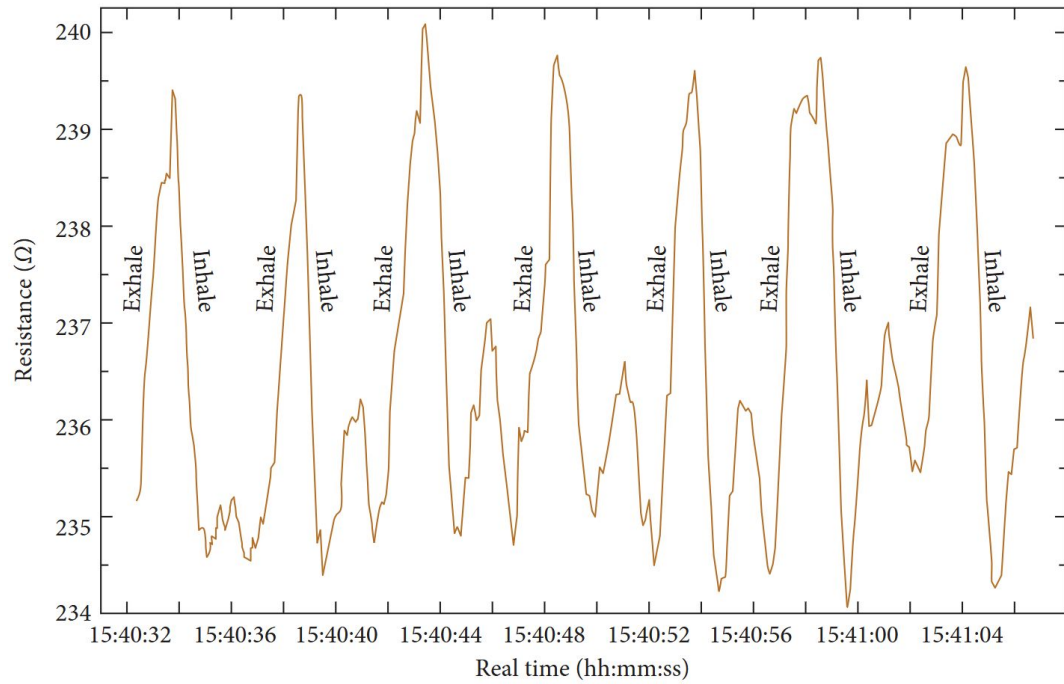
(c)

O: (4.00±0.06) at. %

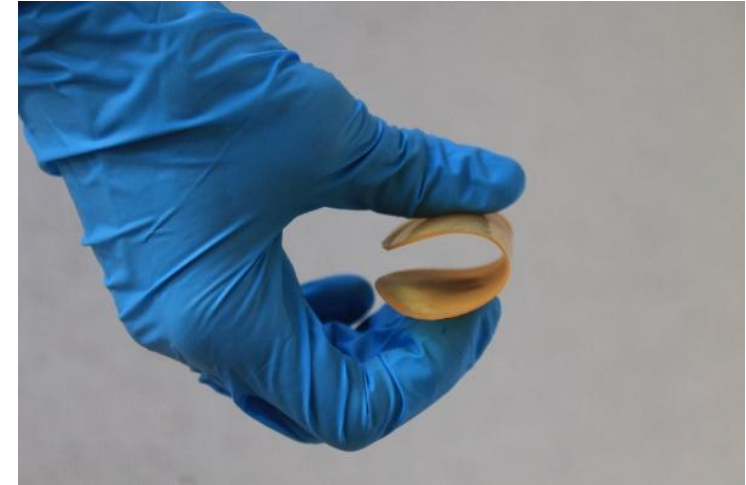
C: (95.88±0.03) at. %



Tracking breathing with graphene



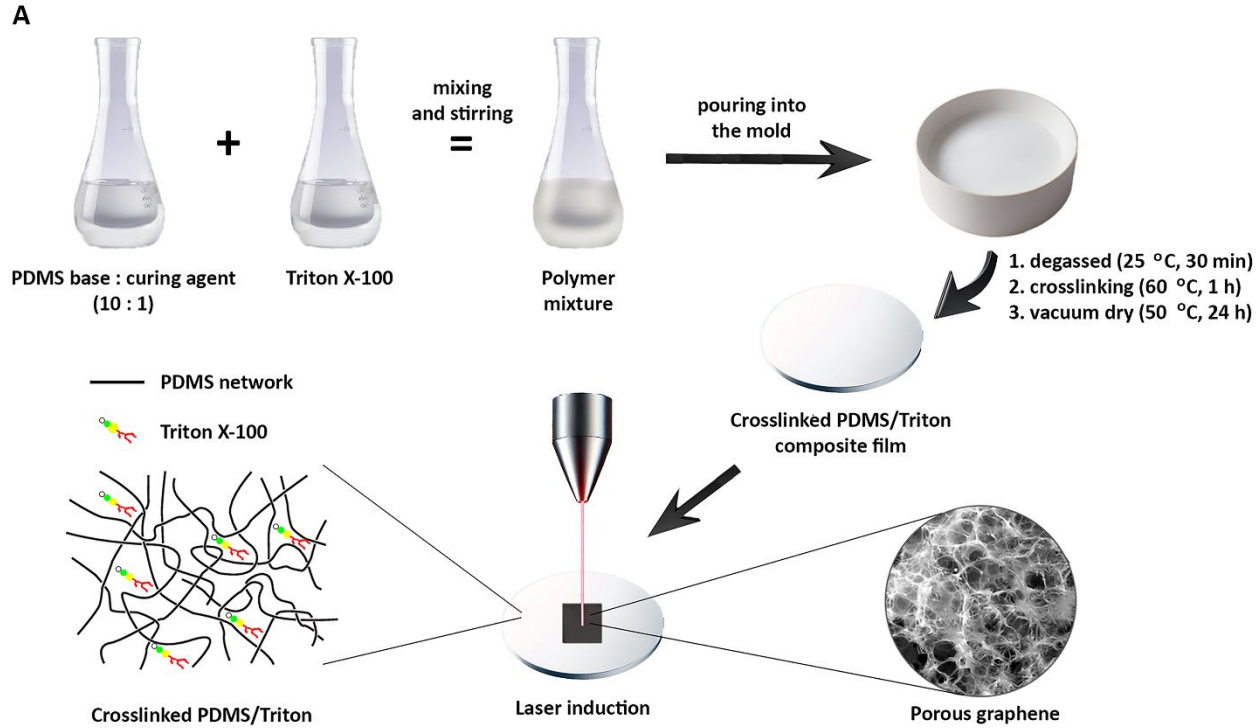
LIG on biocompatible polymers



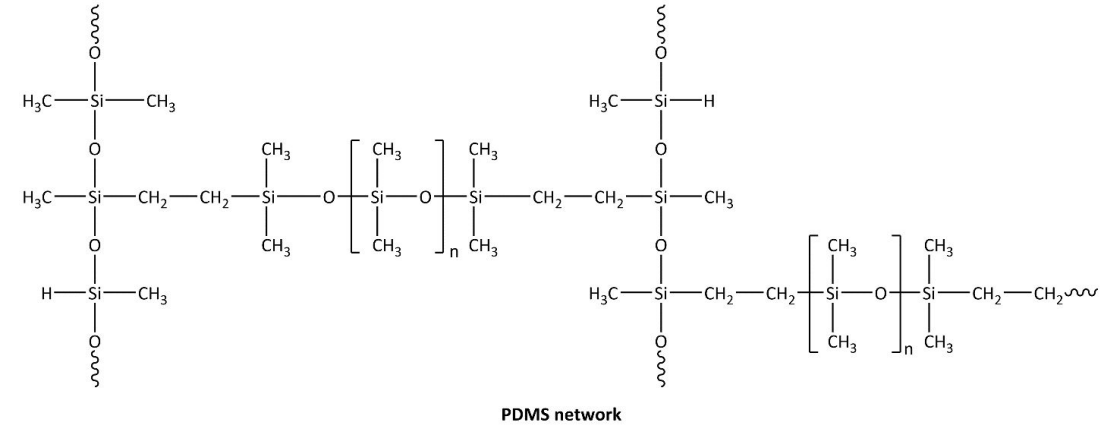
LIG on sodium alginate

T. Vićentić et al, Nanotechnology **35**, 115103 (2023)

PDMS/Triton composites



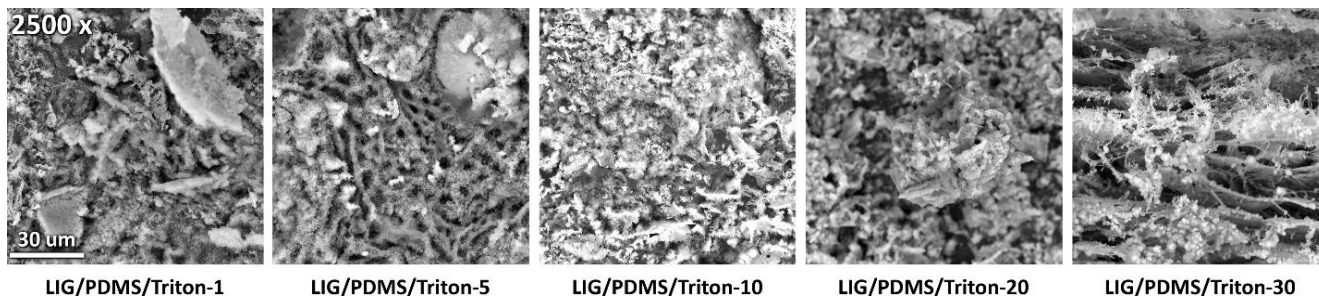
Chemical structures of crosslinked PDMS and Triton X-100:



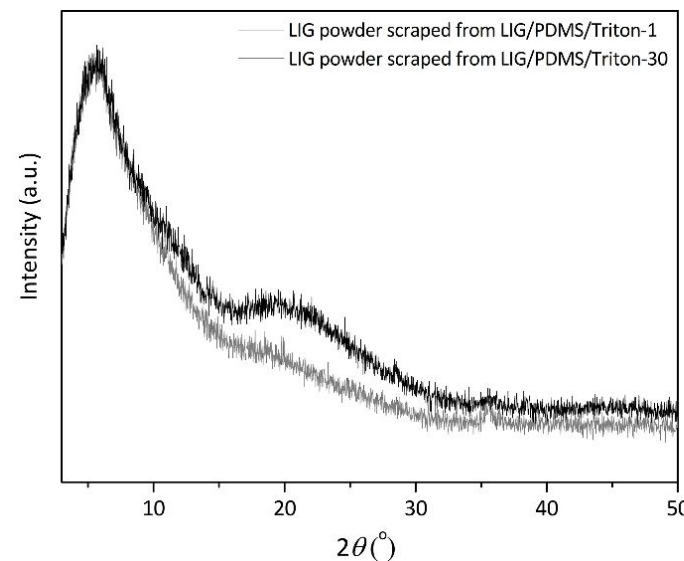
Triton X-100 as a carbon and aromatic ring source to enhance the graphenization of PDMS

PDMS/Triton characterization

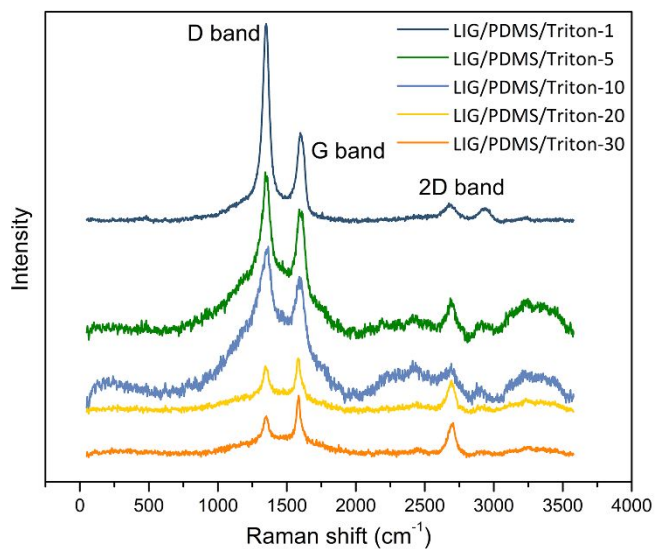
SEM



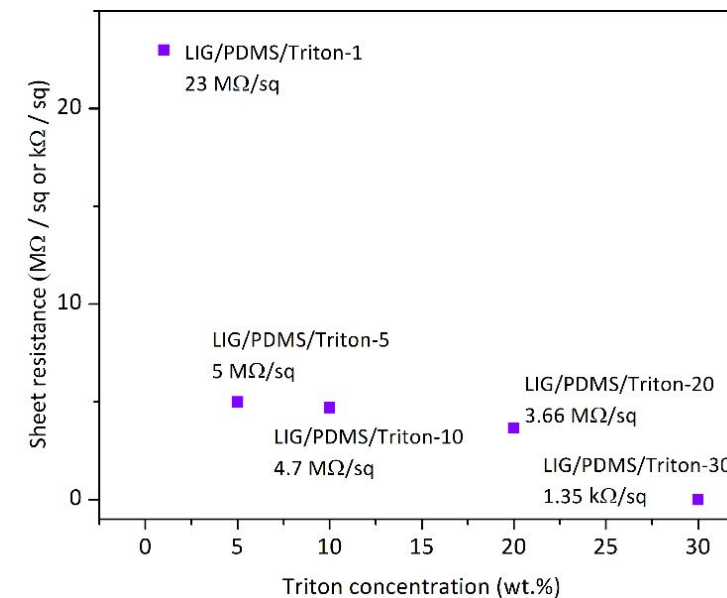
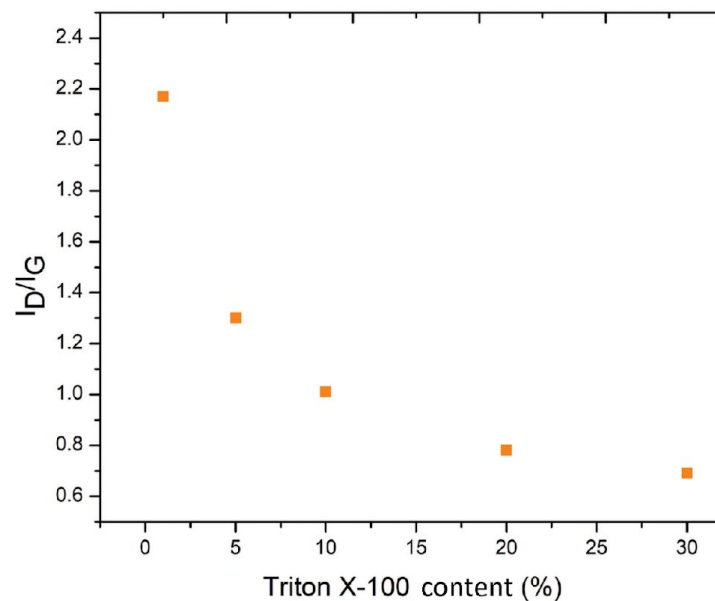
XRD



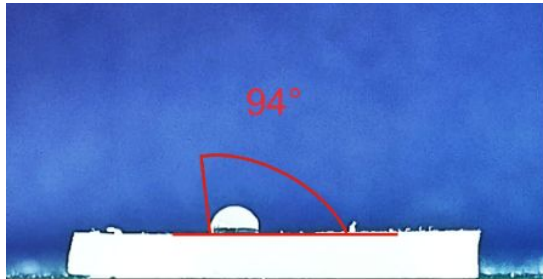
Raman



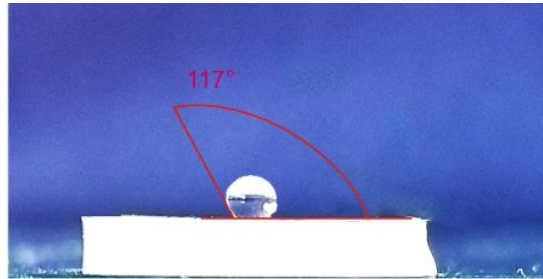
Electrical



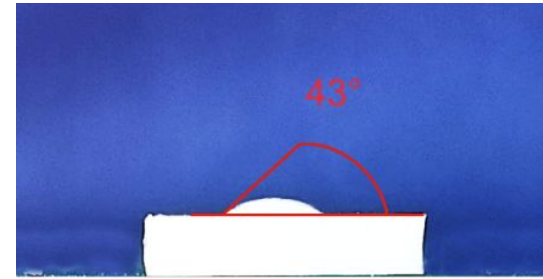
Surface wettability



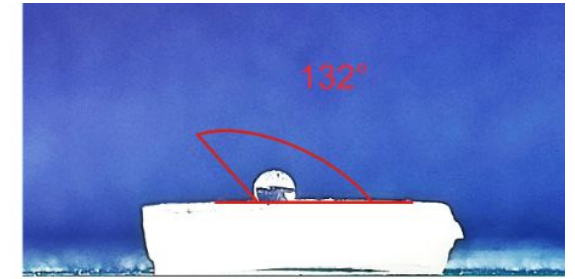
PDMS/Triton-1



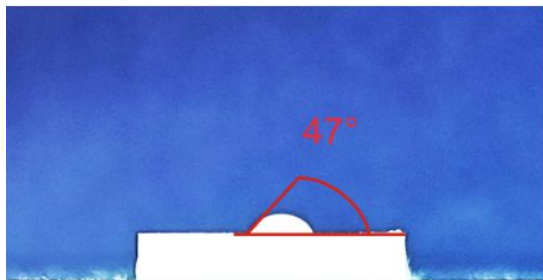
LIG/PDMS/Triton-1



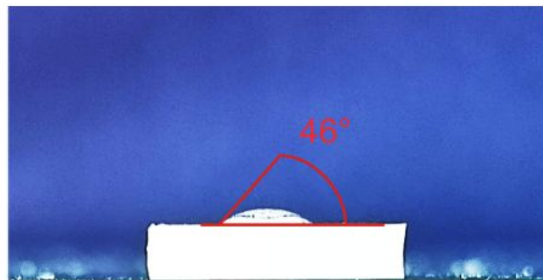
PDMS/Triton-20



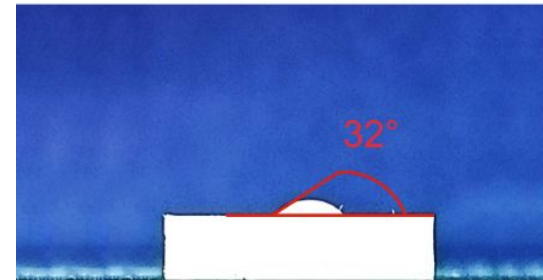
LIG/PDMS/Triton-20



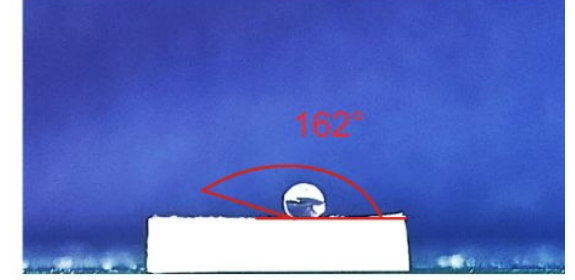
PDMS/Triton-5



LIG/PDMS/Triton-5



PDMS/Triton-30



LIG/PDMS/Triton-30

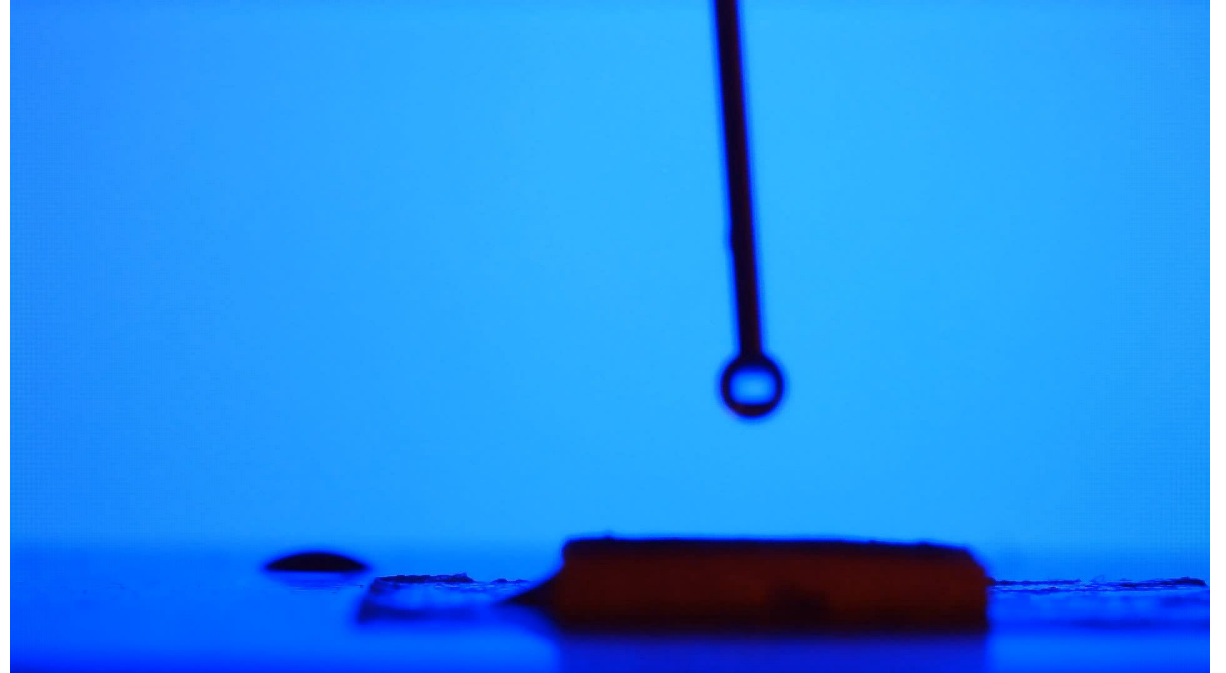
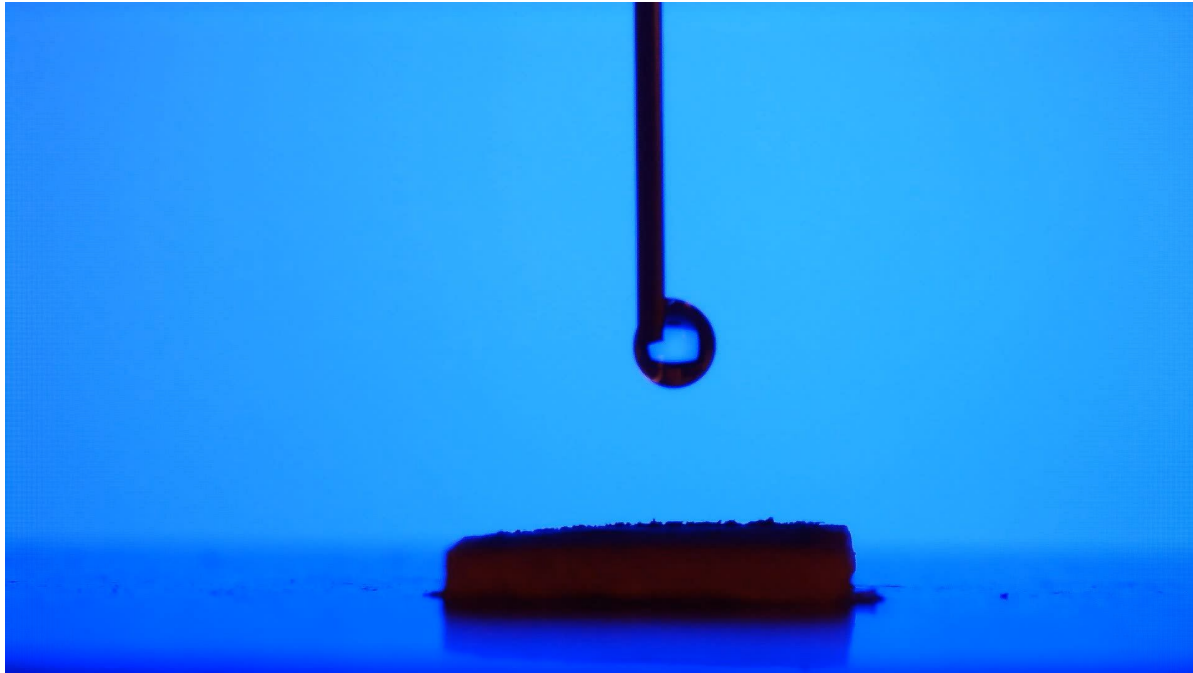
Without
graphene

With
graphene

Without
graphene

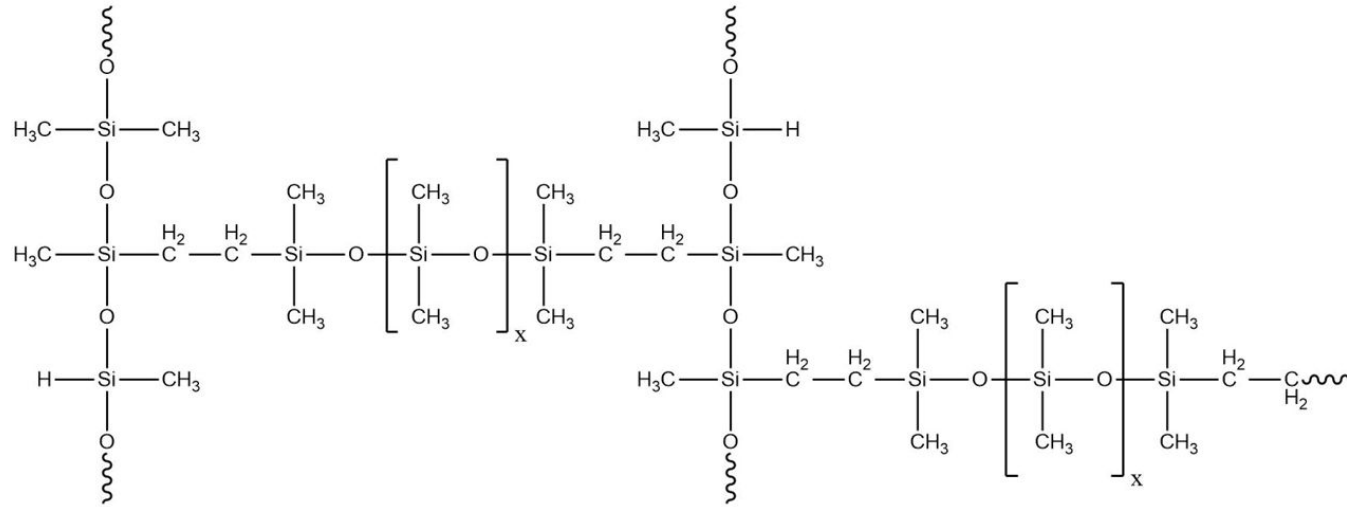
With
graphene

Surface wettability

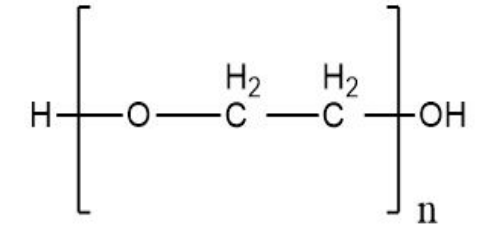


Superhydrophobic surfaces

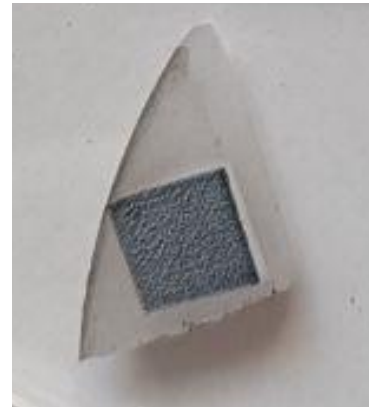
LIG on biocompatible PDMS/PEG



PDMS network



PEG ($M_n = 400 \text{ g/mol}$)



Photographs of pure PDMS, PDMS/PEG composite and LIG/PDMS/PEG

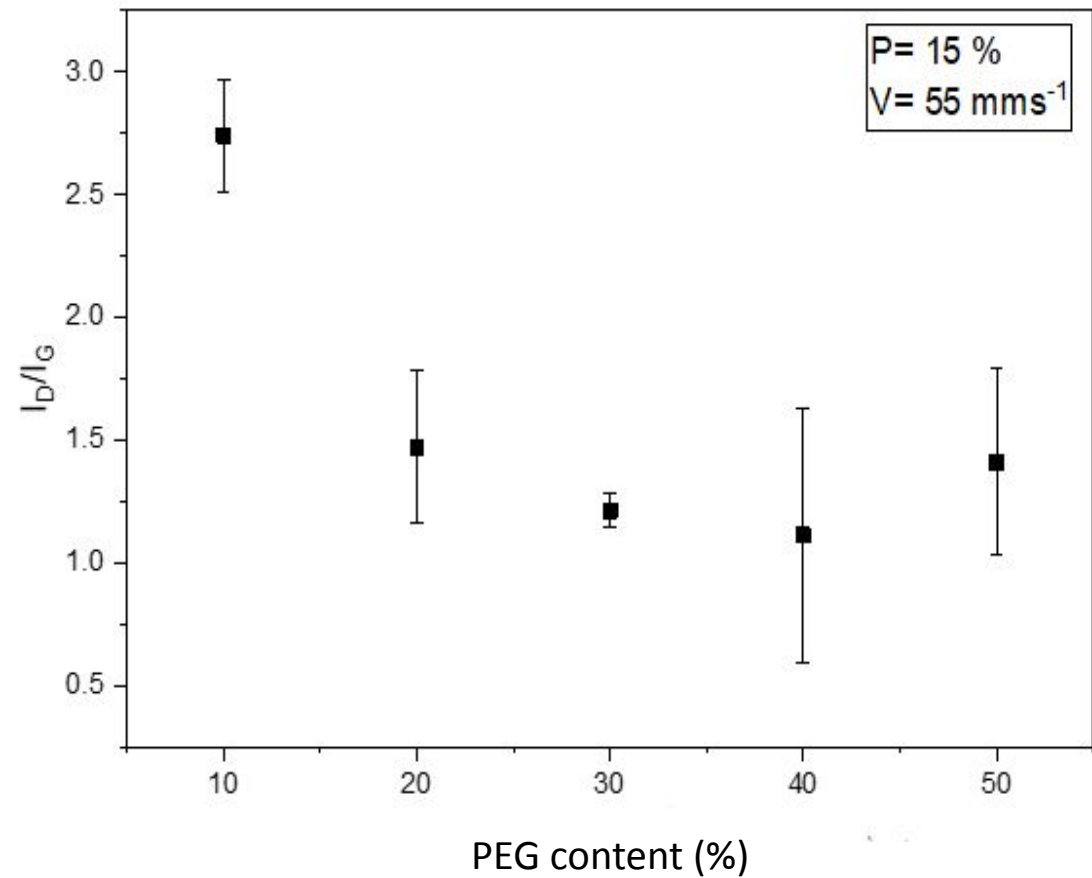
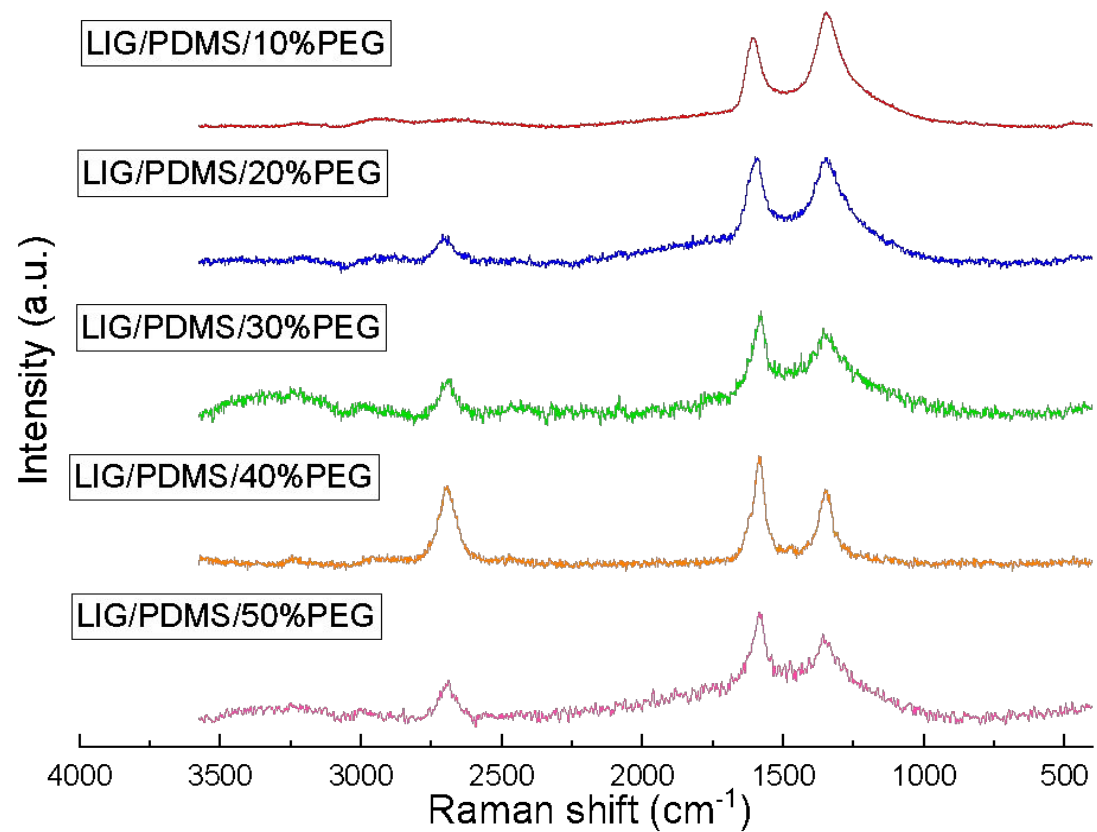
PDMS:

- Biocompatibility
- Flexibility
- Hydrophobicity

PEG:

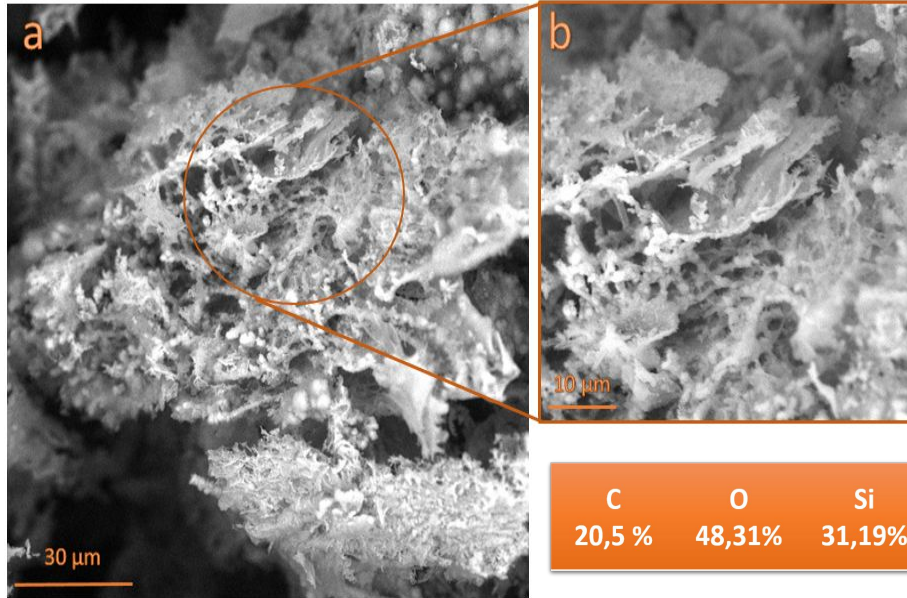
- Hydrophilicity
- Non-toxicity
- Carbon source

Raman of LIG on PDMS/PEG



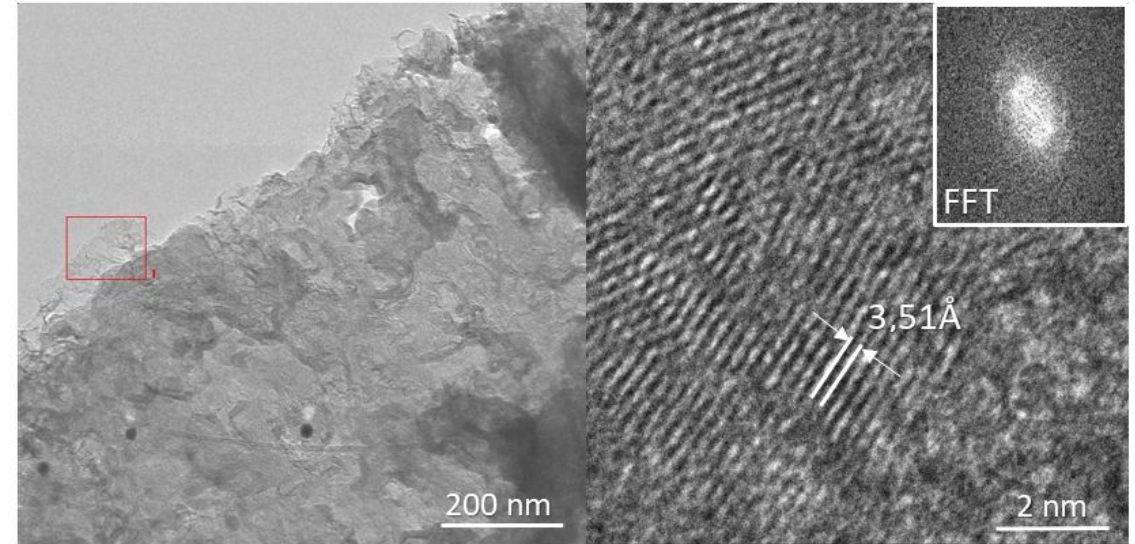
SEM, TEM and XPS of LIG on PDMS/PEG

SEM

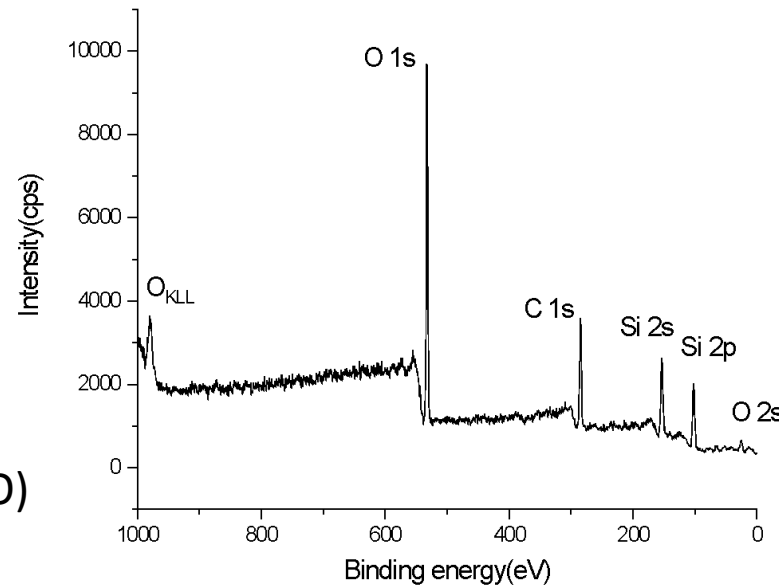


C	O	Si
20,5 %	48,31%	31,19%

TEM



$d = 3.51 \text{ \AA}$ LIG (002)

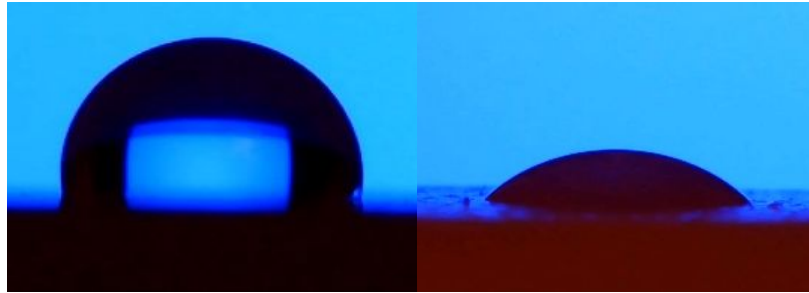


XPS:

- SiO_2 and SiC
- Oxidation of LIG (the presence of C=O)

Application

Without graphene



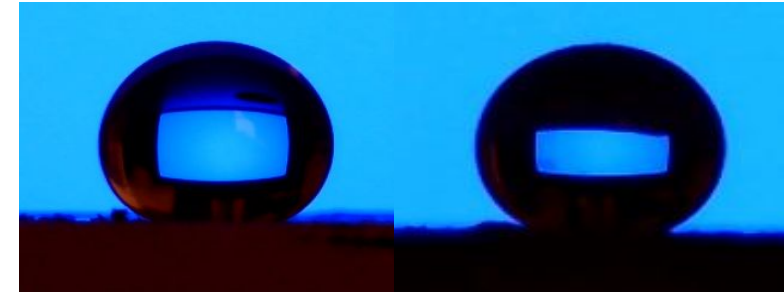
20% PEG

$\theta=99^\circ$

40% PEG

$\theta=38^\circ$

With graphene



20% PEG

$\theta=145^\circ$

40% PEG

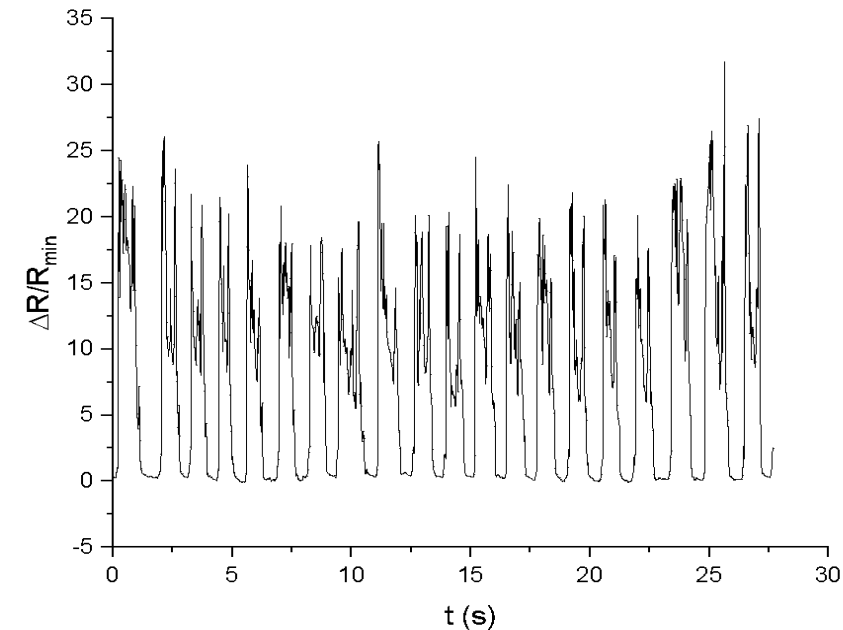
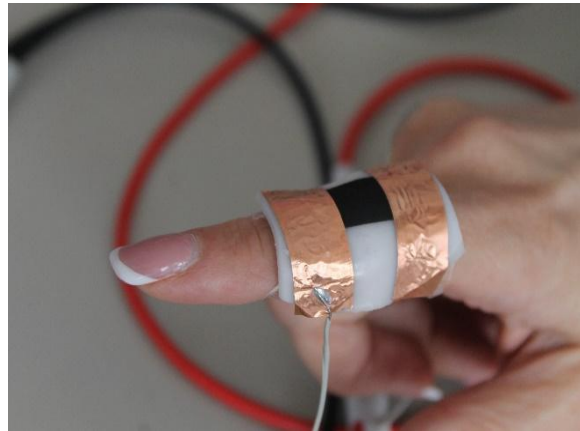
$\theta=154^\circ$

Tensile Test

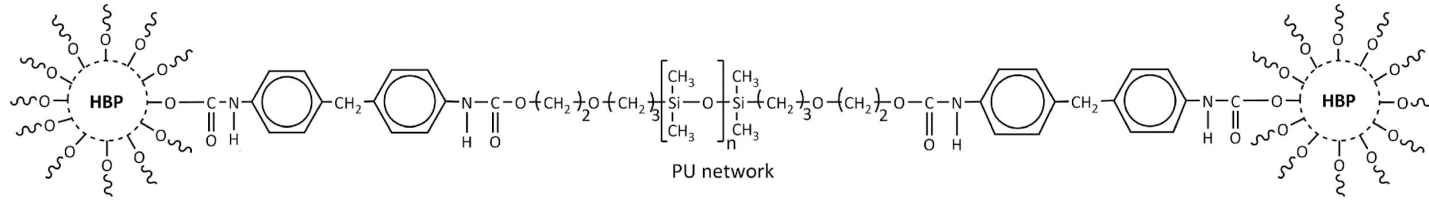


Young modulus = 0.470 MPa
Tensile strength = 1.926 MPa
Elongation at break = 265 %
Gauge factor = 346 (0-10% elongation)

Limb motion sensor



LIG on polyurethane networks

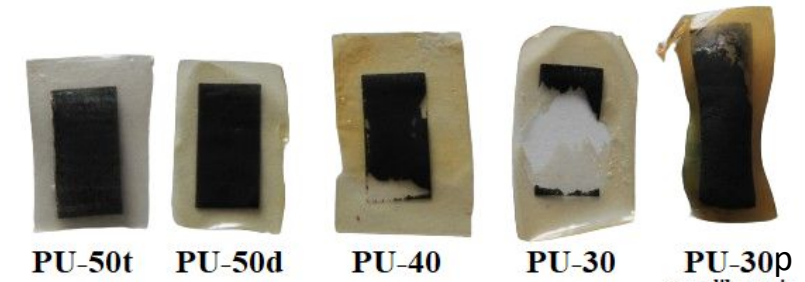
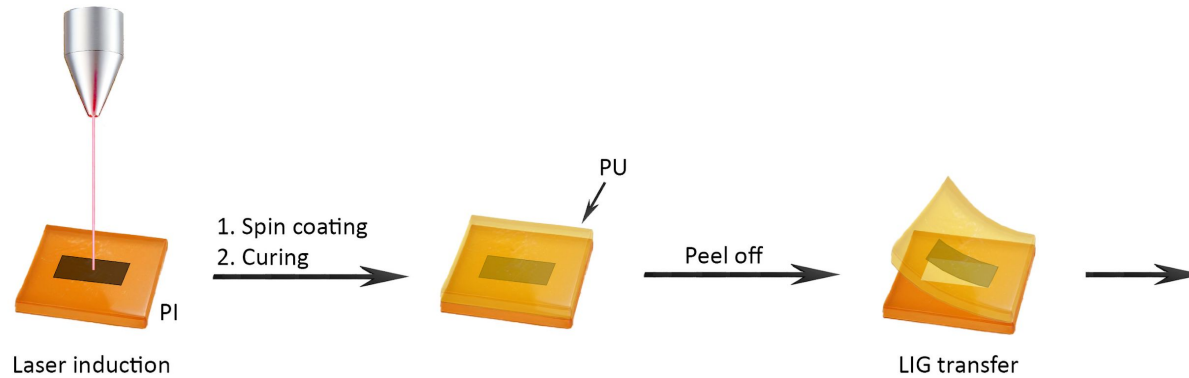


Structure of biocompatible PU-EO-PDMS network

Soft segment: EO-PDMS

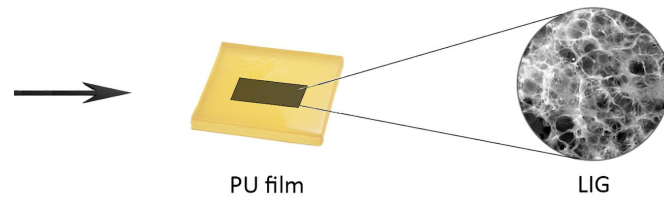
Hard segment: MDI-HBP

SSC = 30-50 wt.%



Transfer LIG on PU film with different SSC

Transfer depends on PU structure and content PU



Schematic illustration of the transfer process of LIG from PI onto PU film

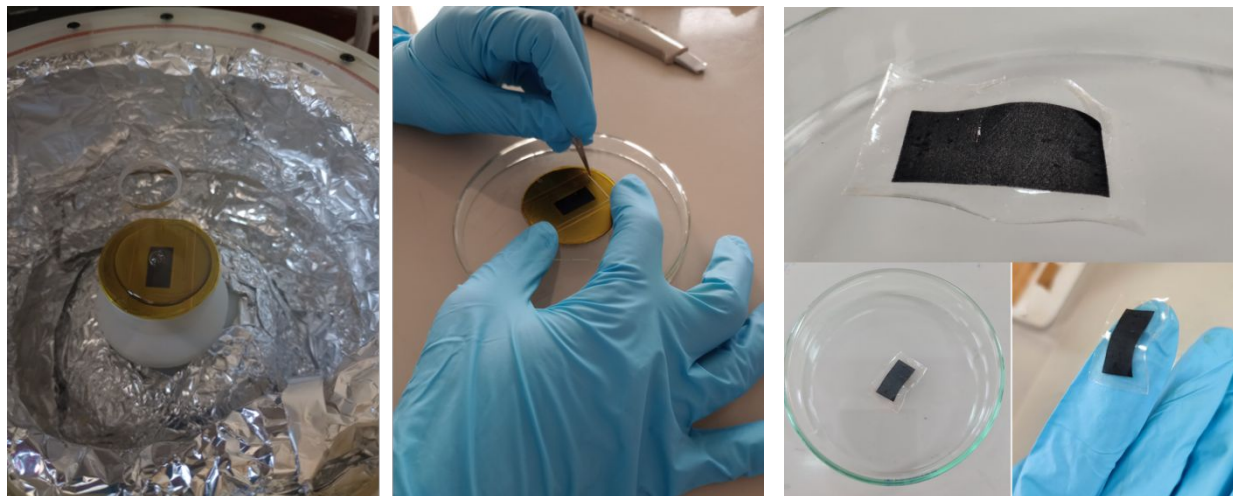
PU properties:

- Good biocompatibility and non-toxicity
- Good flexibility
- Strong adhesion
- Good thermal and oxidative stability

- The sensor is made by transferring LIG from PI to PU materials with varying soft segment content (30, 40, and 50 wt.%). The SSC and thickness of the PU films were adjusted to achieve optimal sensor performance.

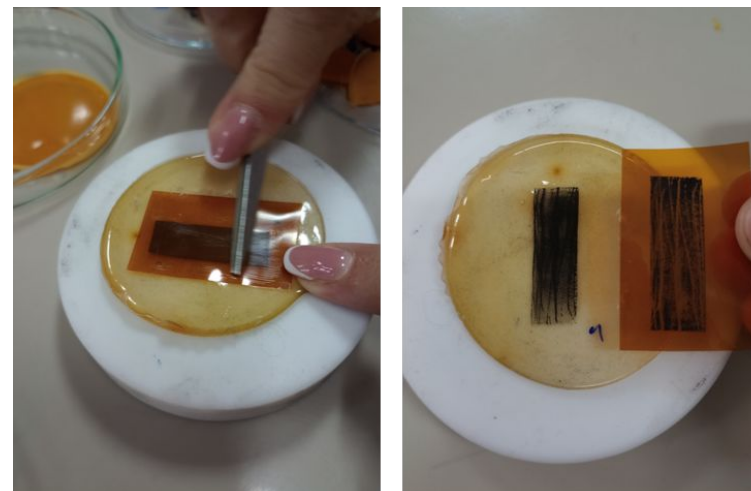
Transfer of LIG to PU

Spin coater



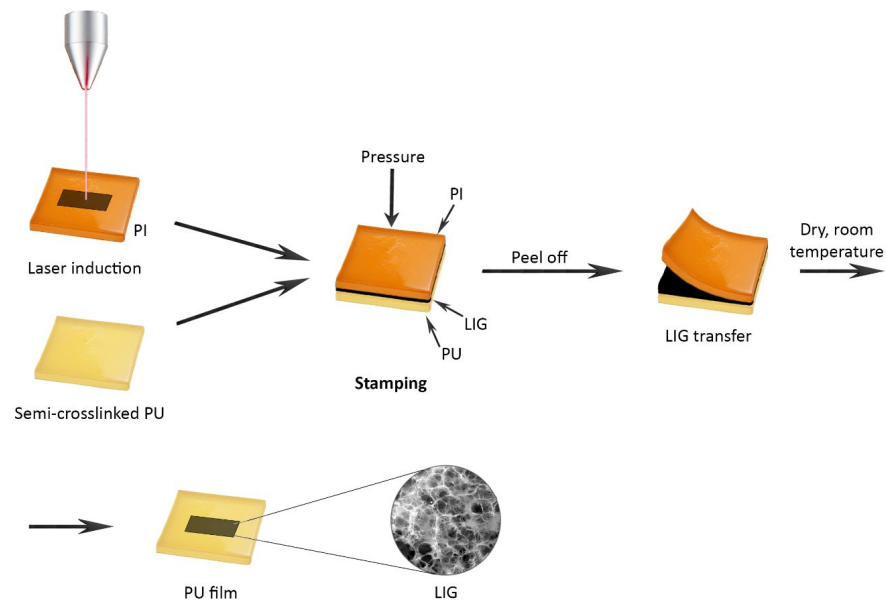
Spin coater – 500 rpm, 30 s; Hot plate – 90 °C, 30 min

Stamping



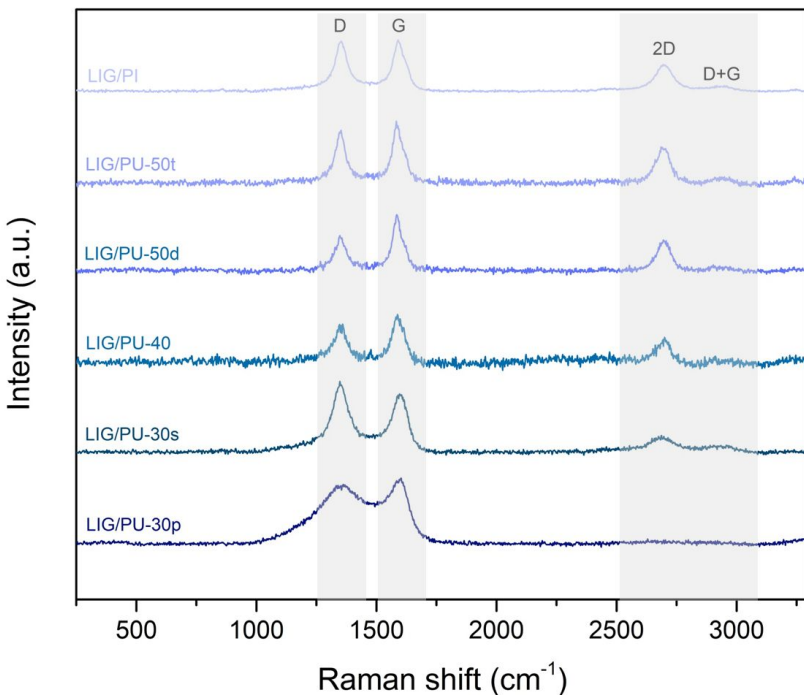
10 h at 80 °C

Polymer	thickness [μm]	resistance [Ω]
PU _t -50	80 \pm 10	
PU _d -50	90 \pm 10	
PU-40	100 \pm 10	
PU-30	100 \pm 10	/
PU-30 _p	500 \pm 10	300

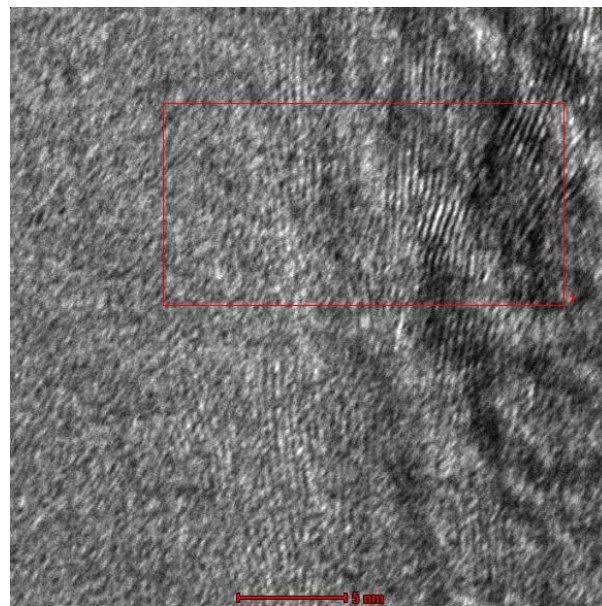


Transferred graphene characterization

Raman

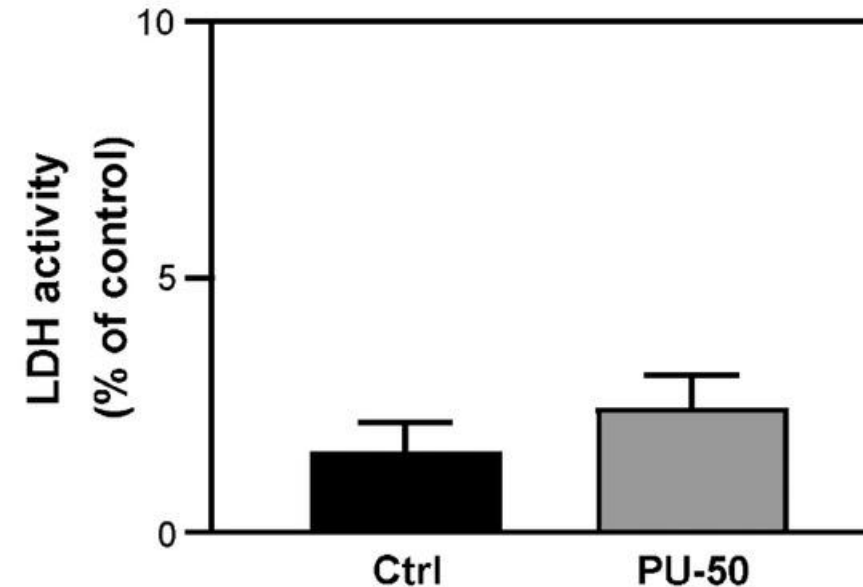


TEM

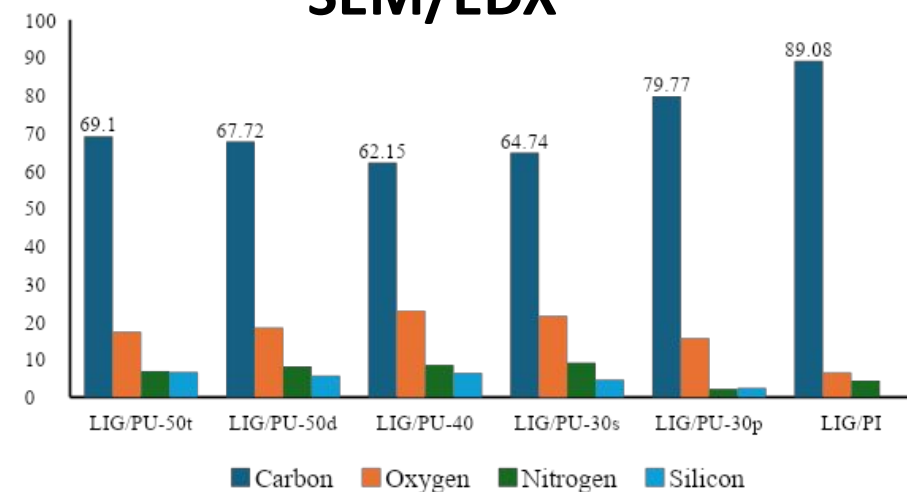


$d = 0.345 \text{ nm}$

Cytotoxicity



SEM/EDX

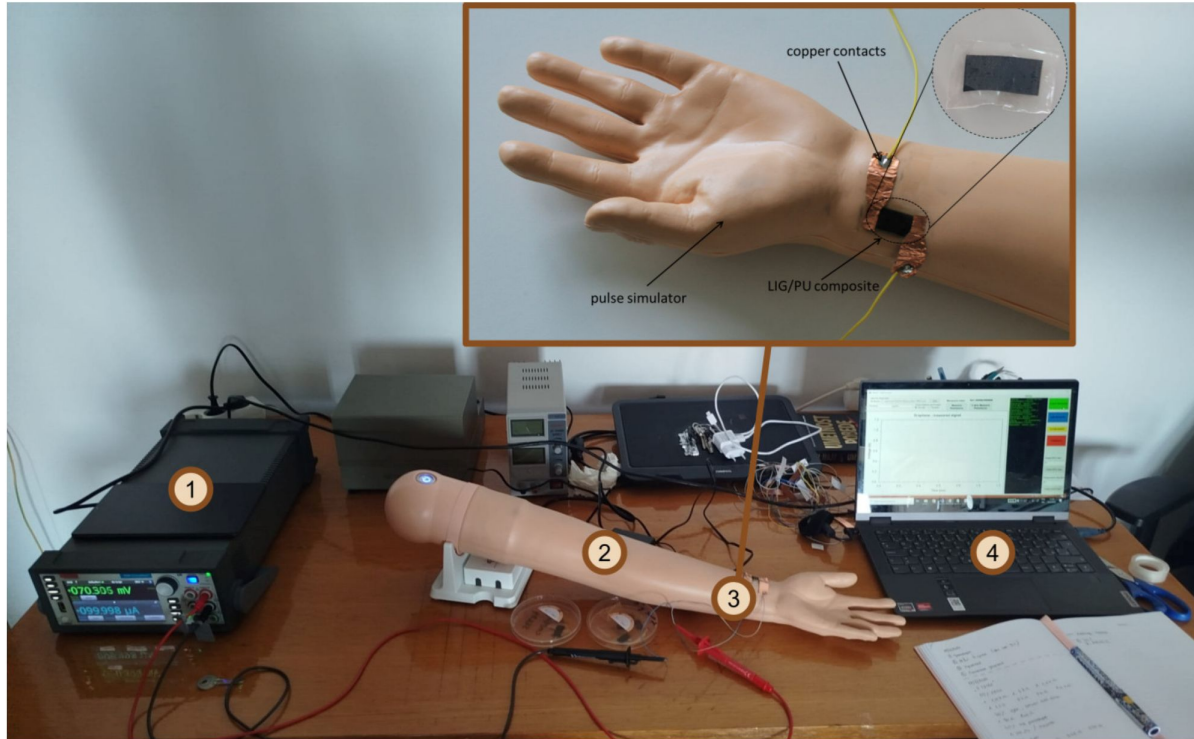


Degree of crystallinity (XRD)

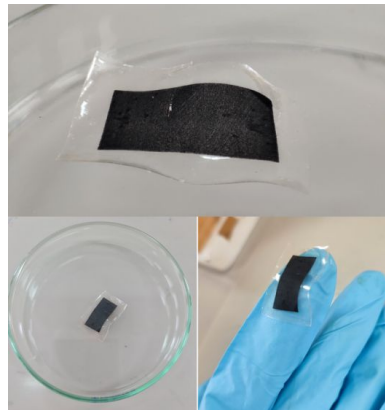
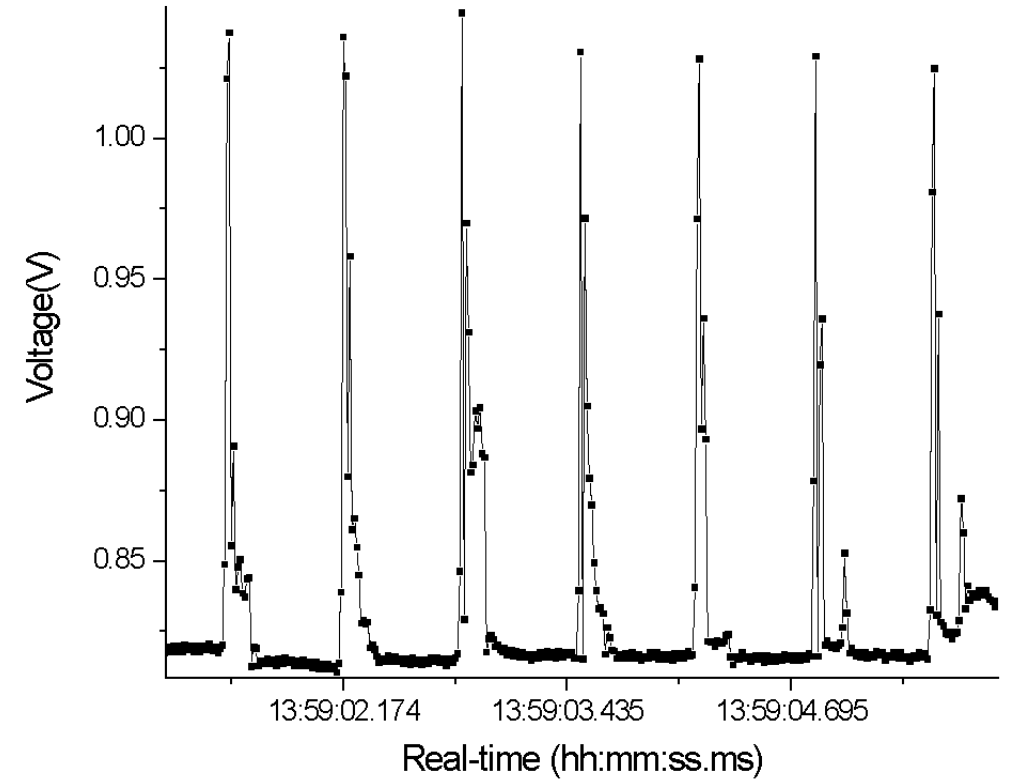
Material	X_c (%)
LIG/PU-50d	42,6
LIG/PU-50t	38,8
LIG/PU-40	Amorphous
LIG/PU-30s	34,8
LIG/PU-30p	26,8

Stamping results in LIG with higher carbon content and fewer impurities. However, adhesion of LIG to the substrate is not as good as with the spin-coating method.

Wearable sensor



1 - Keithley 2450 SMU 2 - Pulse simulator 3 - Pulse sensor 4 - Polygraph software



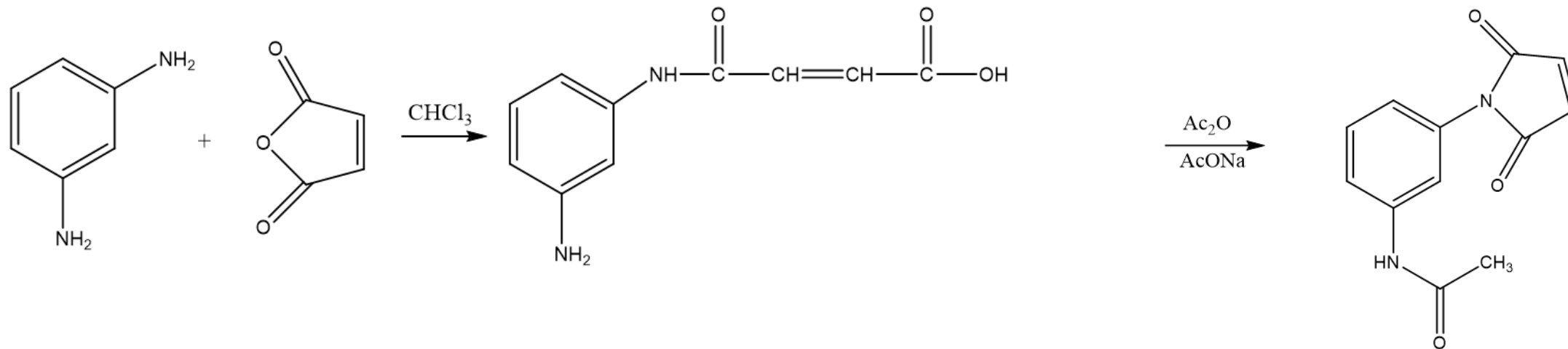
Optimal substrate for LIG transfer and physiological parameters sensing applications with best S/N ratio, near 270:

thin PU film with **50 wt.% of the SSC:**

- ✓ **biocompatible and transparent PU substrate with good mechanical properties,**
- ✓ **strong adherence of LIG to the PU substrate**

V. Vojnović et al, Chemosensors **13**, 122 (2025)

Synthesis of polyimide precursors



I precursor:

(2Z)-4-[(3-aminophenyl)amino]-4-oxobut-2-enoic acid

Yield: 98%, m.p. 206 °C



II precursor: N-[3-(2,5-dioxo-2,5-dihydro-1H-pyrrol-1-yl)phenyl]acetamide

Yield: 65%, m.p. 165 °C



Synthesis involves two precursors

Their structure was confirmed with FTIR, ^1H NMR and ^{13}C NMR

Applications of LIG on polyimide

LIG on Kapton – poor adhesion



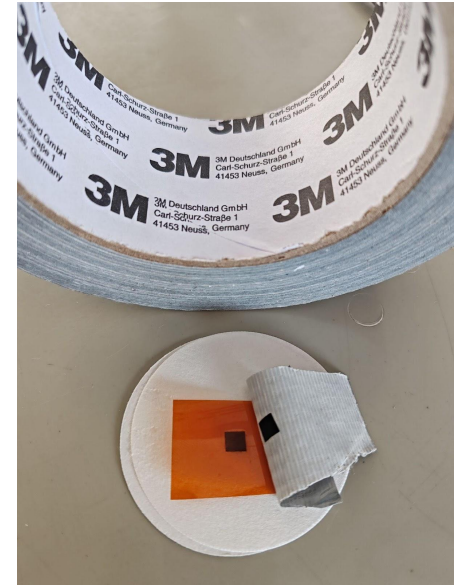
a)

b)

c)

d)

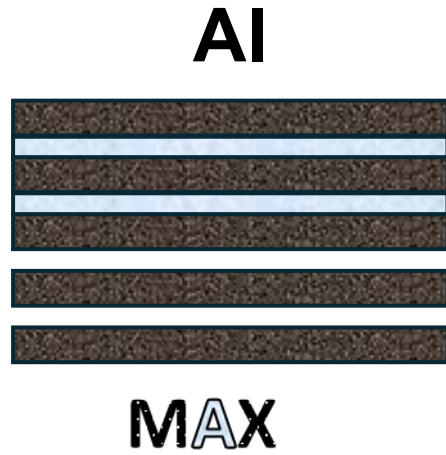
Electrodes for electrochemistry: a) LIG/Kapton, b) LIG/PI-APSA, c) LIG/PI-EDA, d) LIG/PI-UREA



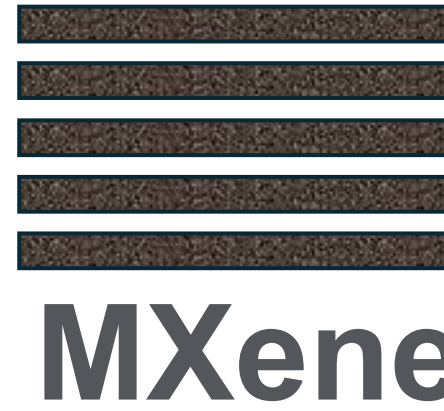
LIG on PI/APSA – excellent adhesion

Polyurethane/MXene composites

MXene ($T_3C_2T_x$)

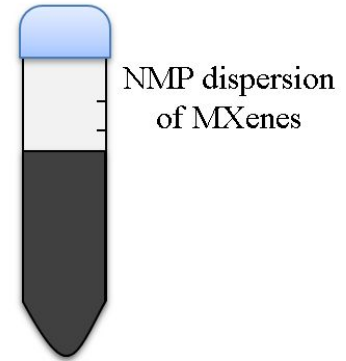
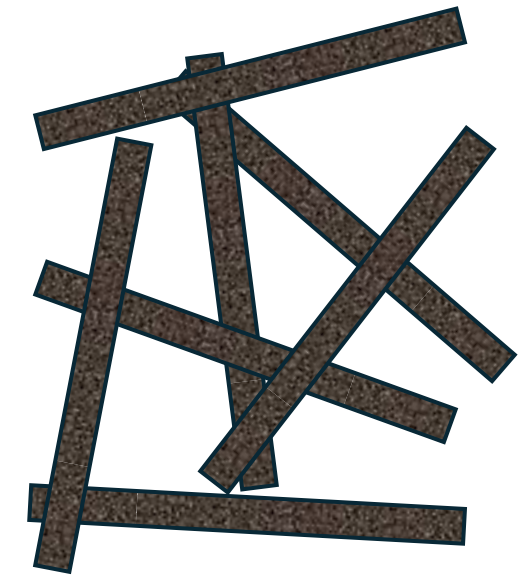


MILD method
Etching
LiF+HCl



$T_x = OH, F, O$

Delamination +
sonication
MXene dispersion in
solvent



PU properties:

- Exceptional chemical corrosion resistance
- Strong adhesion
- Good thermal and oxidative stability
- Good flexibility and durability
- Good biocompatibility and non-toxicity

Properties of MXene - $T_3C_2T_x$:

- Electrical conductivity
- Hydrophilicity
- High specific surface area

Preparation of PU-Mxene composites

I Preparation PU film



PU-50 film, dimension: 1 × 2 cm
thickness: 500 μm

Mechanical properties of PU by tensile test:

Young modulus: 0.975 MPa

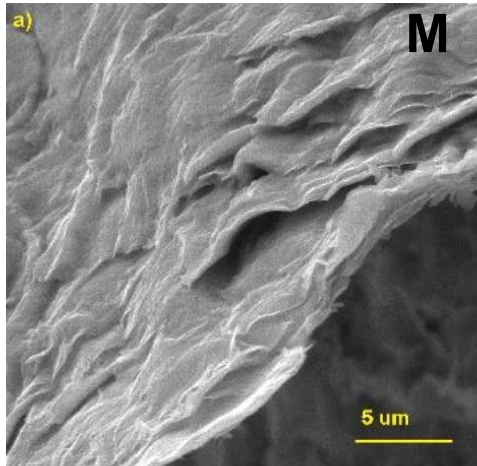
Tensile strength: 0.681 MPa

Elongation at break: 153.75 %

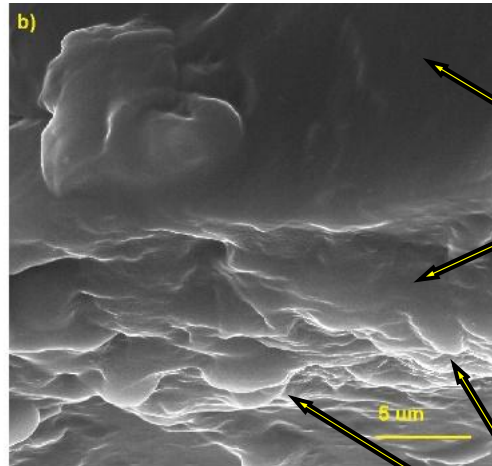
II Preparation of MXene layers



SE
M



MXene



Polyurethane

Soft segments

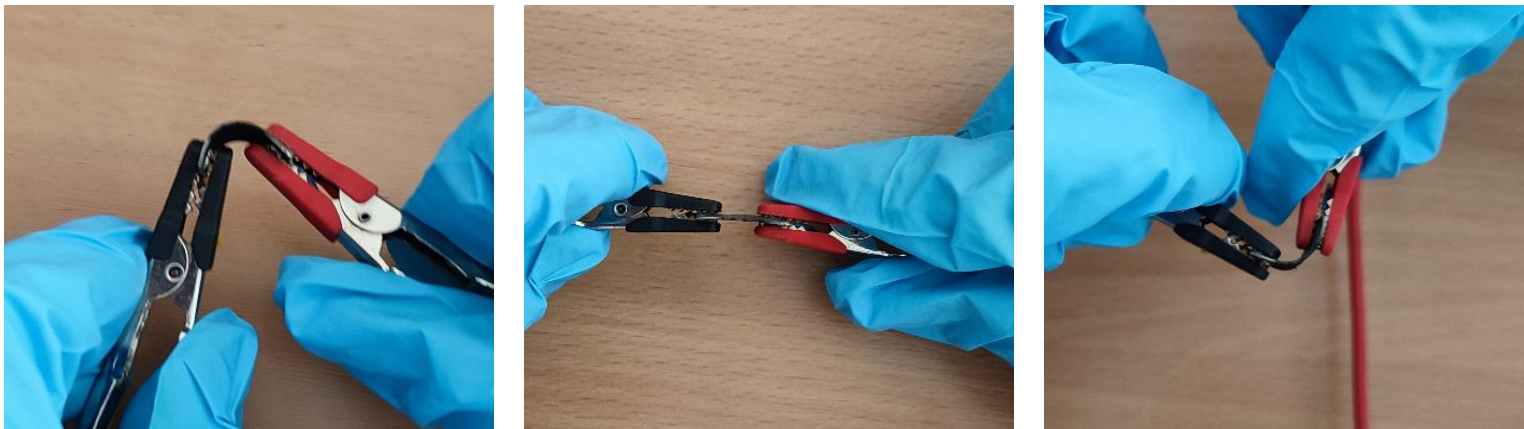
Hard segments

- ✓ Dispersing the MXene in N-Methyl-2-pyrrolidone (NMP);
- ✓ Subsequent coating (500 rpm, 3000 rpm) and
- ✓ Drying of layers (50 °C; 30 min)

* I. Pešić et al, 11th IcETRAN, Niš, 3-6 June 2024

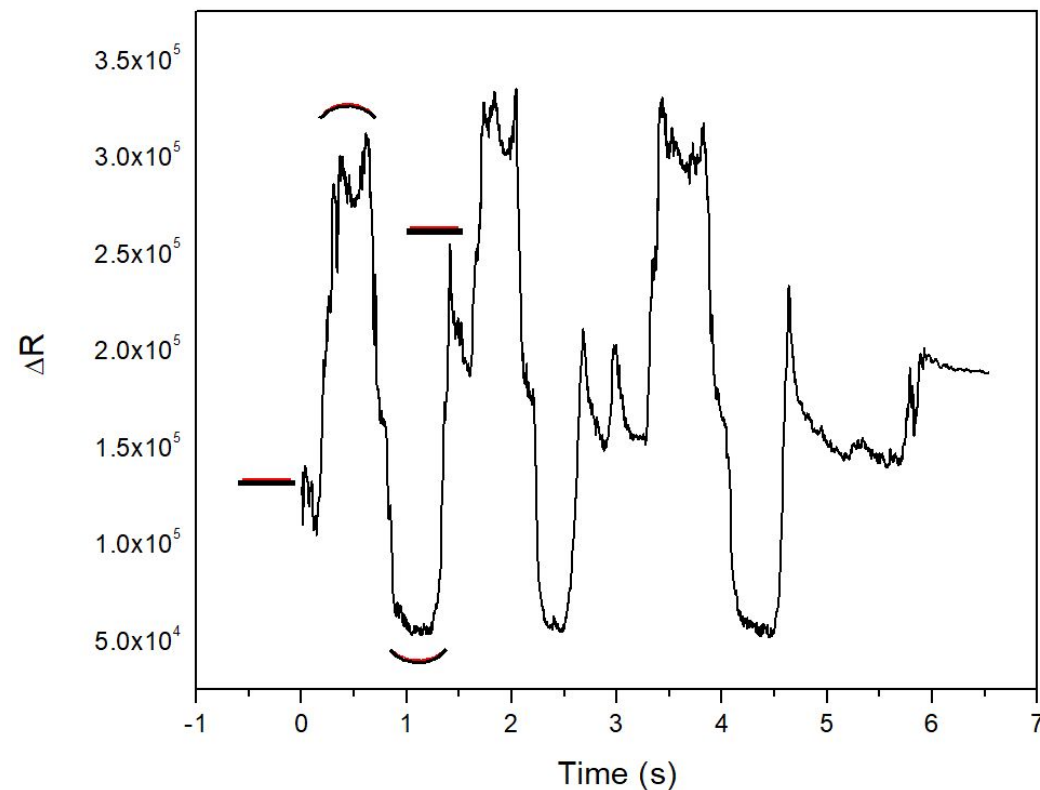
* I. Pešić, et al. Prog. Org. Coat. **203** (2025)

Polyurethane/MXene composites



Relative resistance changes during three cycles of strain at different bending angles

$$\Delta R = 150 \text{ k}\Omega$$

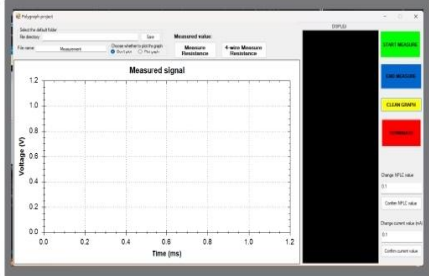


Sensing with PVDF-MXene

PVDF = polyvinylidene fluoride

Electrical measurements of PVDF/MXene sensor

Custom software for data acquisition



GPIB 488 IEEE



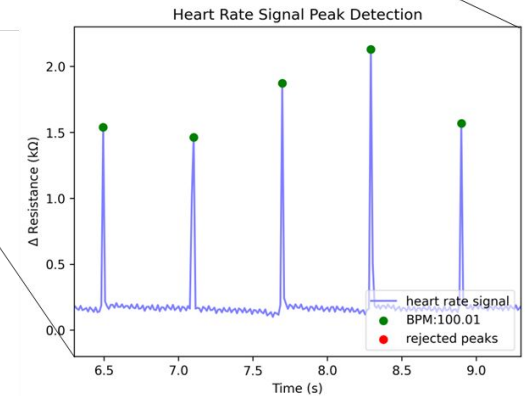
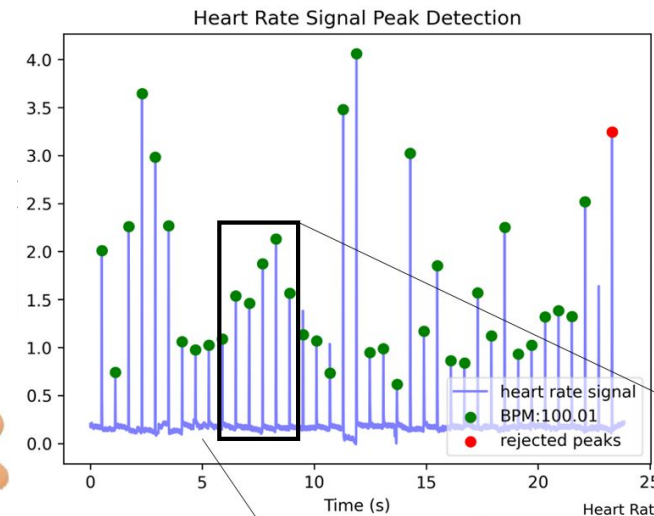
SMU (Keithley 2450)

B.P/Pulse Assessment Simulator (BT-CEAB2)

MXene sensor

Copper plates and wires

Force HI
Force LO



SNR = 26

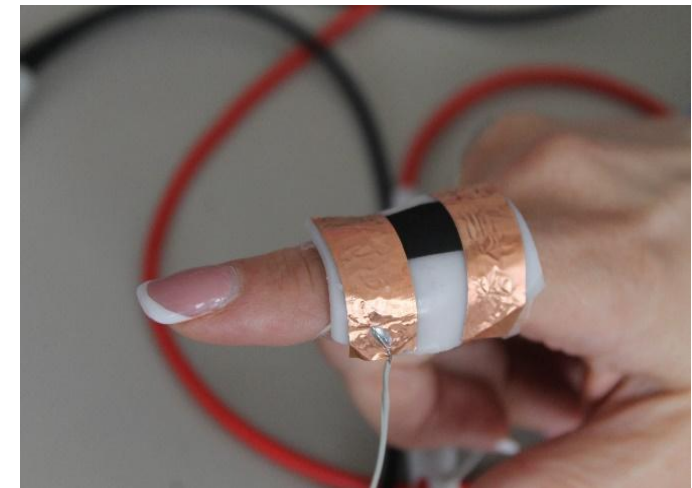
Block diagram of experimental setup for heartbeat monitoring



I. Pešić et al., 12th IcETRAN, Čačak, 9-12 June 2025 - **Award**

Summary

- Laser-induced graphene (LIG) is excellent physiological sensor
- Heartbeat, limb motion and respiration detected
- LIG on PDMS/Triton composites
- LIG on PDMS/PEG
- LIG on PU networks
- LIG on PI
- MXenes
- Easy to make, biocompatible, wearable



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