NOVA SCHOOL OF SCIENCE & TECHNOLOGY



FLEXIBLE E-SKIN SENSORS, ENERGY HARVESTING AND MICROFLUIDIC DEVICES



ALLSENSORS 2022 June 26, 2022 to June 30, 2022 Porto, Portugal i3N CENIMAT, Materials Science Department,
NOVA School of Science and Technology,
NOVA University of Lisbon and CEMOP/UNINOVA,
Campus de Caparica, 2829-516 Caparica, Portugal

Rui Igreja, rni@fct.unl.pt

Presenter Short Resume:

Researcher areas and interests:

- Impedance Spectroscopy
- Planar electrodes
- Instrumentation
- Sensors
- Biosensors
- e-skin devices
- Digital microfluidic systems

Participated in a total of 18 scientific research projects, national and European, 4 as local coordinator. Professor @ DCM-FCT/UNL Coordinator of BSc in Micro end Nanotechnology Researcher @ CENIMAT|i3N and /CEMOP Former sub-director of CENIMAT|i3N



Prof. Rui Igreja <u>rni@fct.unl.pt</u> **Researcher ID** J-3670-2013 **Scopus ID** 6602536589



Outline

• CENIMAT | I3N and UNINOVA-CEMOP

• e-skin devices

- Piezoresistive pressure sensors (health, robotics)
- Piezoresistive using Temperature Shrinking Polymer Molds
- Piezoresistive temperature/pressure sensing
- Flexible Piezo/Tribo devices

• Fiber based devices

- Energy harvesting devices
- TFT for smart textiles
- Digital µFluidics



Outline

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Where are we?

FCT-NOVA campus, ~15min south Lisbon, Portugal





CENIMAT | I3N and UNINOVA-CEMOP

CENIMAT

- Center for materials research, directly linked to the Materials Science Department
 @ FCT-NOVA
- Evaluated as *Excellent* by panel of international experts in Materials Science and Engineering since 1996.
- Since 2006 integrates the Associated Laboratory <u>i3N Institute for Nanostructures</u>, <u>Nanomodelling and Nanofabrication</u>
- 3 research groups:
 - Materials for Electronics, Optoelectronics and Nanotechnologies (MEON)
 - Soft and biofunctional materials group (SBMG)
 - Structural Materials (SM)

<u>UNINOVA</u>

- Private non-profit research institute acting in the fields of robotics, electronics, micro/nanoelectronics, optoelectronics, telecommunications, artificial intelligence, environment and vacuum/production technologies.
- Organized in centers of excellence (>150 scientists and technologists)
 - Centre of Excellence in Microelectronics, Optoelectronics and Processes (CEMOP)
 - Centre of Technology and Systems (CTS)



Prof. Elvira Fortunato Ex-Director of CENIMAT|i3N Since 30th March 2022: Minister for Science, Technology and Higher Education



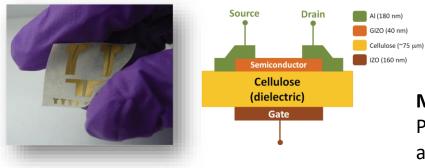
Prof. Rodrigo Martins Director of UNINOVA-CEMOP



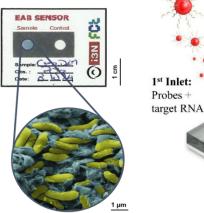


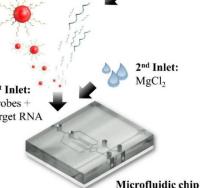
MEON-i3N | CENIMAT Research lines

Paper Electronics: Tunning paper properties towards different applications



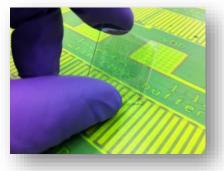
Sensors/Biosensors/Microfluidics: e-skin sensors; DNA sensors; Glucose sensors; PDMS based sensors, paper and digital Microfluidics, Electroactive bacteria detection, SERS





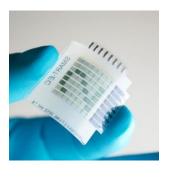
Micro and Nanoelectronics Processing: Physical and chemical processing, nanoscale

and maskless patterning



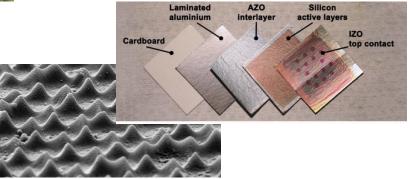


Chromogenic materials: Electrochromic displays; Thermochromic materials; pH sensors; Printed displays on paper





Energy Materials: Solar cells on paper; Thin film nanostructured silicon; Perovskites; BIPV; Thermoelectric devices; Supercapacitors; Photonics; Batteries











PROCESSING

Ø cemop







Microwave reactors



1



High-T furnace

Further info @ https://www.cenimat.fct.unl.pt/lab-facilities

Laser Cutting and Engraving





*CO*₂ and fiber laser

Paper Lab



Inkjet printer

Screen printer





Flexo Printer

Wax printers

8

Chemical synthesis

ALD



PECVD





Evaporator



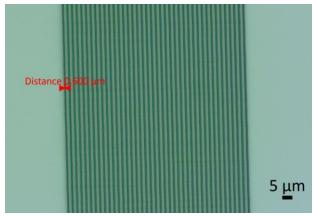
Lithographic processes for sub-micron scale devices

Direct laser writer (DLW)

600 nm

Linewidth

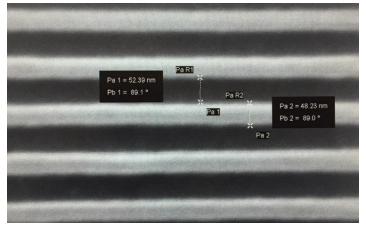




Nanoimprint lithography (NIL)

50 nm

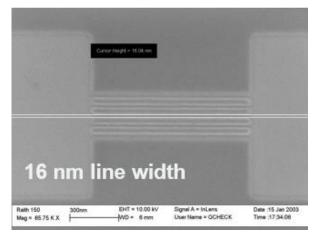




Electron-beam lithography (EBL)

10 nm









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Electrical and electrochemical



Cryogenic probe station



Potentiostats

CV, IV, pulsed IV analyzers



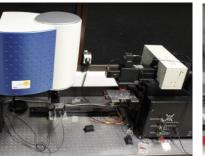
Spectroscopy



XRD



Spectroscopic ellipsometer



Spectral Response



FTIR

Micro-Raman





XPS

UV-Vis-NIR







ITUTO DE OESTRUTURAS, OMODELAÇÃO E OFABRICAÇÃO ITIGAÇÃO, INOVAÇÃO E ACOES DE ENDEDINA EIA



STEM (2021) Cold-FE SEM

SEM-FIB-EBL





Tabletop SEM

AFM

Outline

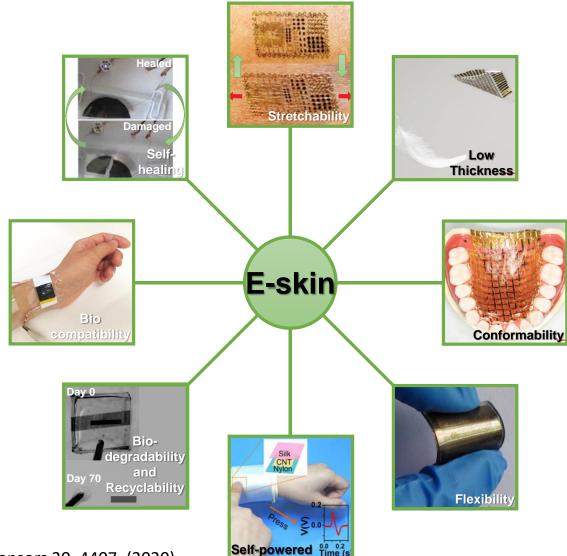
• CENIMAT | I3N and UNINOVA-CEMOP

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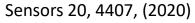
e-skin devices



Skin: detection of pressure, touch, vibration, tickle, heat, cold, pain.

E-skin devices key features:

- self-healing
- Stretchability
- low thickness and conformability
- Flexibility
- self-powered
- biodegradability and recyclability
- biocompatibility









e-skin devices



E-skin devices applications:

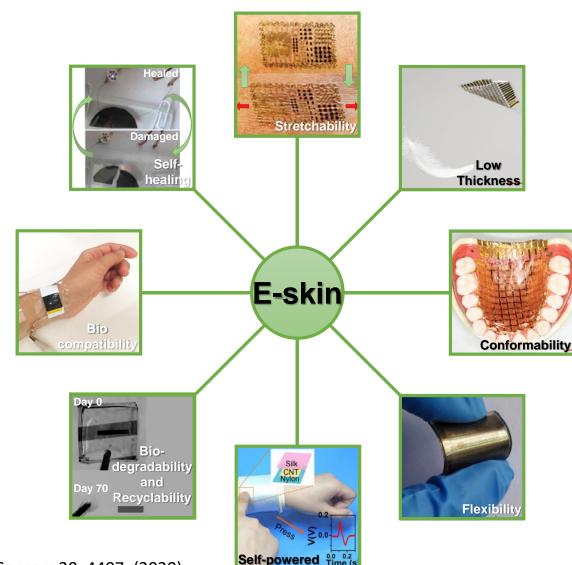
- health monitoring
- functional prosthesis
- robotics
- Human-machine interfaces
- Power harvesting







e-skin devices



Sensors 20, 4407, (2020)

E-skin devices transducer mechanisms:

- Capacitance
 - Simple design and analysis;
 - Limited miniaturization; Prone to hysteresis and high × response times; More complex readout electronic.
- Piezoelectricity
 - Self-powered; Fast response time; High sensitivity
 - Unable to detect static pressure; Prone to noise from x vibrations or high frequency stimuli; Drift in sensor's response over time; Temperature interference.
- Piezoresistivity ٠
 - Simple structure; Simple readout mechanism
 - Power supply required; Requires micro-structuration × for performance improvement;
- Triboelectricity ٠
 - Simple design and analysis; ~
 - Unable to detect static pressure; Output affected by × frequency of stimulus.

sensors

Review

i3

INSTITUTC NANOESTI NANOMOD NANOFABE

CENIMAT

CENTRO DE INVESTIGAÇÃO DE MATERIAIS

NOVA SCHOOL OF

DEPARTMENT OF MATERIALS SCIENCI

SCIENCE & TECHNOLOGY

Transduction Mechanisms, Micro-Structuring Techniques, and Applications of Electronic Skin Pressure Sensors: A Review of Recent Advances

Andreia dos Santos[®], Elvira Fortunato, Rodrigo Martins, Hugo Águas *[®] and Rui Igreja *[®] CENIMATIi3N, Departamento de Ciência dos Materiais, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa, Campus de Caparica, 2829-516 Caparica, Portugal; ass.santos@campus.fct.unl.pt (A.d.S.); emf@fct.unl.pt (E.F.); rm@uninova.pt (R.M.)

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MDPI



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e-skin devices: main goals @ cenimat

• Develop of multifunctional e-skin sensors

- Pressure Sensing
- Temperature Sensing
- Energy Harvesting (self-power)

• Implementation of a Novel Micro-structuring Strategy for Pressure Sensors

- Low cost and high tailoring
- Study of several parameters that affect the micro-structuring outcome

• Proof-of-concept – Tuning the E-skin Sensors for Different Applications

- pressure wave detection at the wrist
- General health monitoring
- Functional prosthesis and robotics

Development Multifunctional Sensors

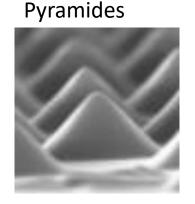
- Temperature and Pressure out of the same functional material/device

• Development of Energy Harvesters

- Novel micro-structured composites

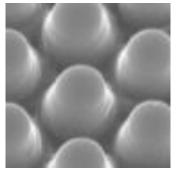


Photolitography





Domes



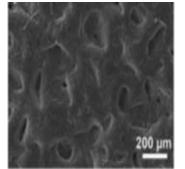
J. Park; et al. BioNanoScience 2014, 4 C. Pang; et al. Nature Materials 2012, 11 J. Park; et al. ACS Nano 2014, 8, 5

Fibres

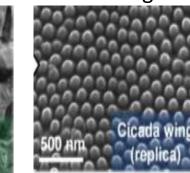


Natural molds

Sandpaper



Insect wings



X. Wang; et al. Adv. Mater. 2014, 26, 1336 Q.-J. Sun et al. ACS Appl. Mater 2018, 10, 4086 M.-L. Seol; et al. Small 2014, 10, 3887 M.-L. Seol; et al. Small 2014, 10, 3887

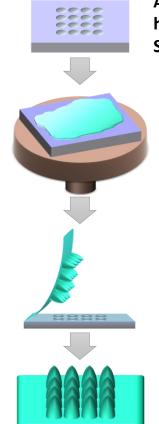


Leafs



cemop

Our work (laser engraved molds)

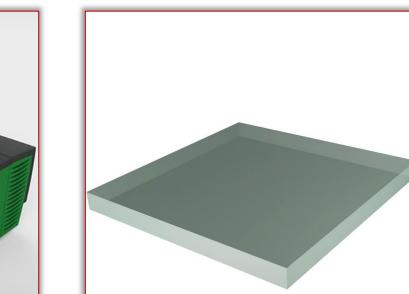


Acrylic h-PDMS Shrinking polymer



Adv. Electron. Mater. 4 (9), 1870041, (2018)

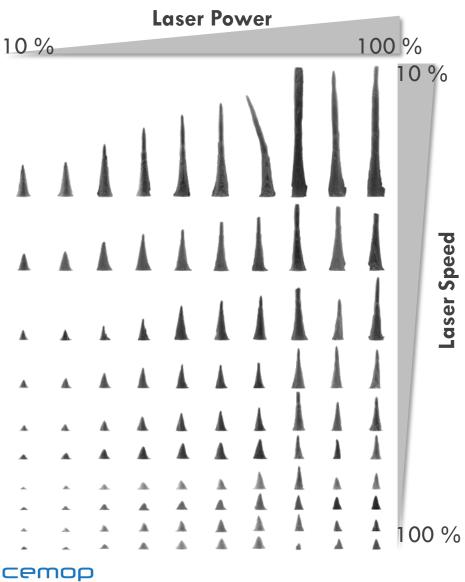
CO₂ Laser engraving technique



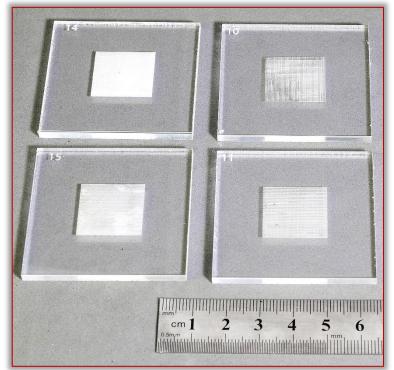
Parameters that impact the micro-structuring:

- Shape, size, spacing, and line thickness of figures being engraved
- Material (substrate) being engraved
- Distance between laser beam and substrate
- Laser power
- Laser speed
- Laser mode (vectorial/rast)

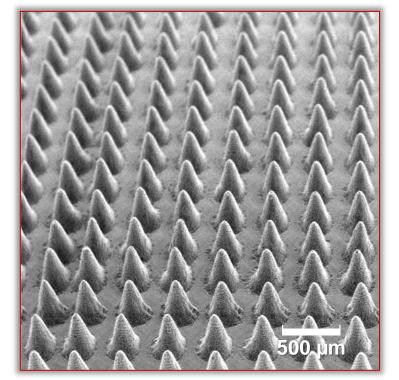




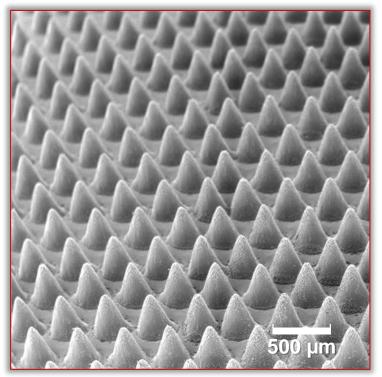
Acrylic Molds



PDMS Membrane (aligned pyramids)



PDMS Membrane (misaligned pyramids)





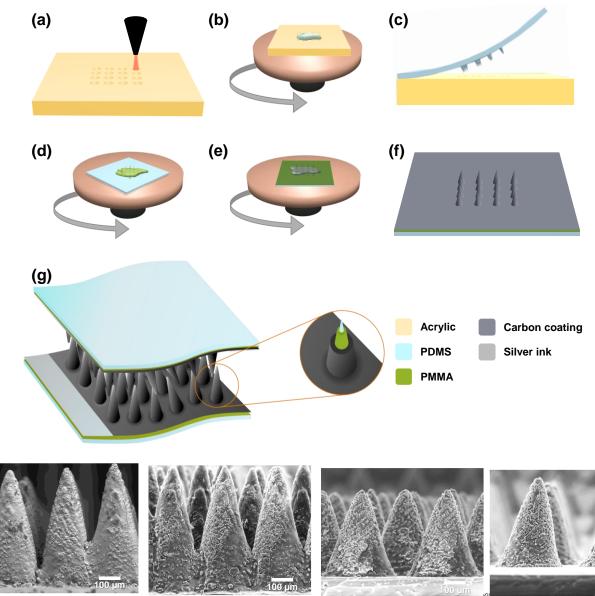




Adv. Electron. Mater. 4 (9), 1870041, (2018)

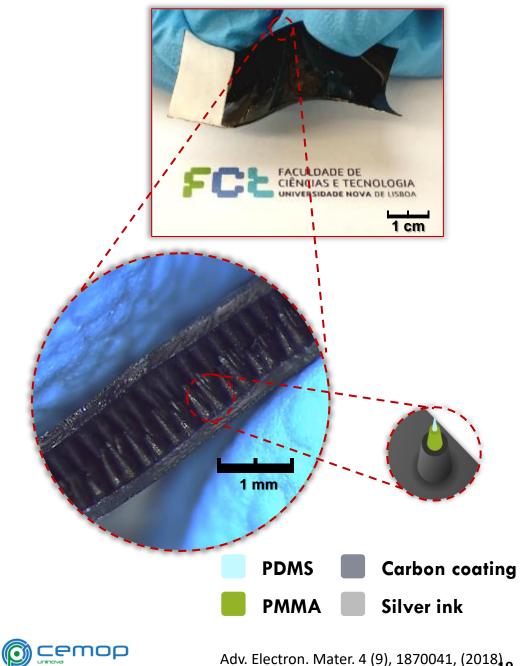
Engraved Patterns:

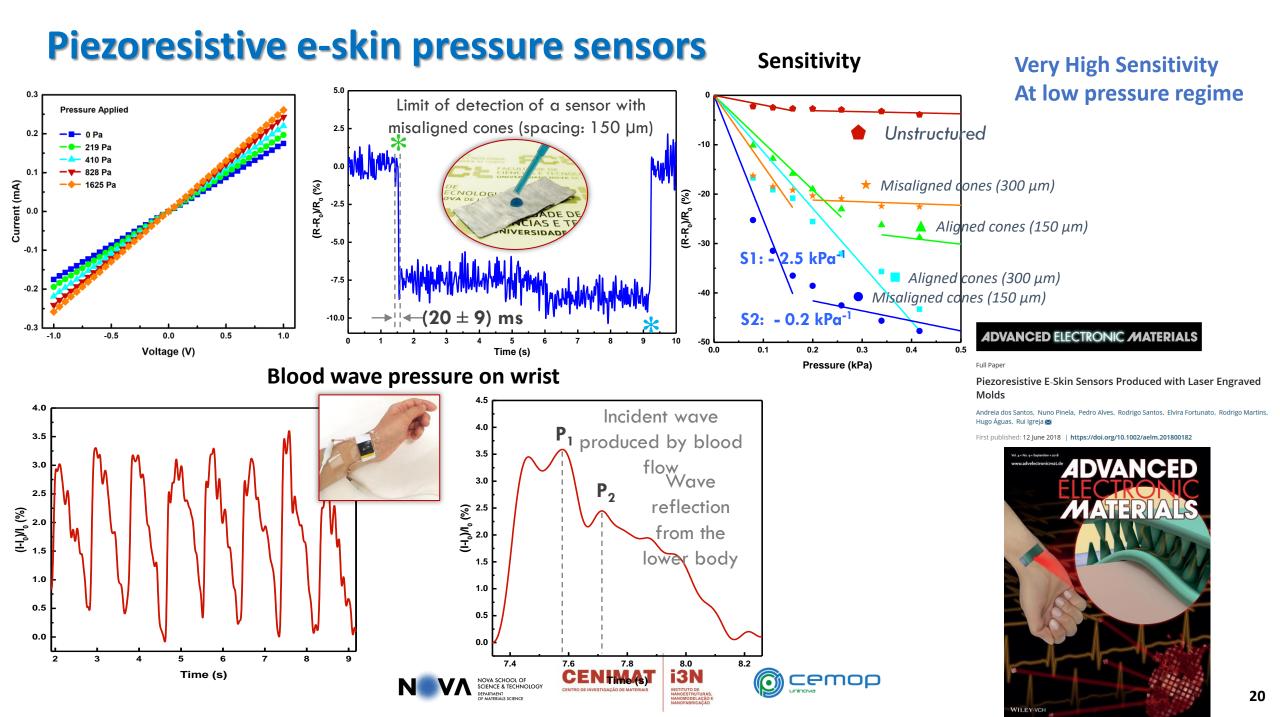
Spacing = 150 μm to 300 μm



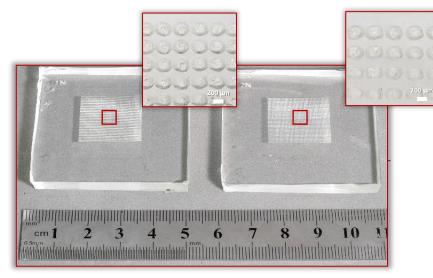
DEPARTMENT OF MATERIALS SCIENCE





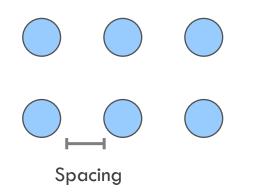


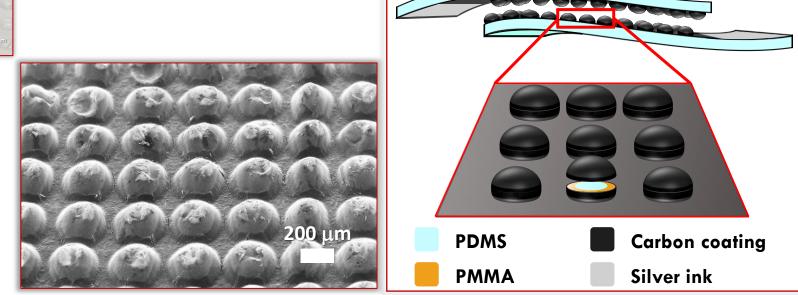
hard-PDMS Molds



Engraved Patterns:

Spacing = 150 μ m to 200 μ m



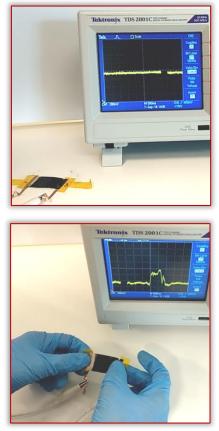


- To produce an e-skin to target robotics/functional prosthesis applications
- Constant sensitivity between < 10 kPa and 100 kPa
- Semi-spheres are less compressible: can withstand higher pressures before saturating

cemop

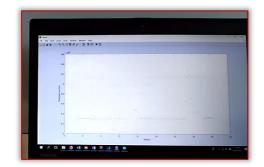






Sensitivity Without PMMA Spacing = 150 μm A Spacing = 200 μm With PMMA -20 Spacing = 150 μm ★ Spacing = 200 μm $S = -2.3 \times 10^{-3} kPa^{-1}$ ΔR/R₀ (%) $= -6.4 \times 10^{-3} kPa^{-1}$ $S = -5.8 \times 10^{-3} kPa^{-1}$ -60 $S = -5.8 \times 10^{-3} kPa^{-1}$ -80 -100 0 50 100 Pressure (kPa)

Pressure detection for robtics and prosthesis





Stable performance over 27 500th



Open Access Proceeding

Nova de Lisboa, Campus de Caparica, 2829-516 Caparica, Portugal [†] Presented at the Eurosensors 2018 Conference, Graz, Austria, 9-12 September 2018.

Proceedings 2018, 2(13), 1039; https://doi.org/10.3390/proceedings2131039

Author to whom correspondence should be addressed.

Published: 12 November 2018

(This article belongs to the Proceedings of Eurosensors 2018)



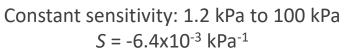
Article

E-Skin Bimodal Sensors for Robotics and Prosthesis Using PDMS Molds Engraved by Laser[†]

Andreia dos Santos ²⁰, Nuno Pinela ²⁰, Pedro Alves ²⁰, Rodrigo Santos ²⁰, Ricardo Farinha [⊠][©], Elvira Fortunato [⊠], Rodrigo Martins [⊠], Hugo Águas 🖾 🔍 and Rui Igreja * 🖾 🔍

CENIMATJi3N, Departamento de Ciência dos Materiais, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa, Campus de Caparica, 2829-516 Caparica, Portugal

Sensors 19 899, (2019)

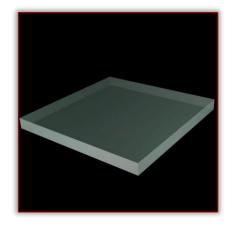




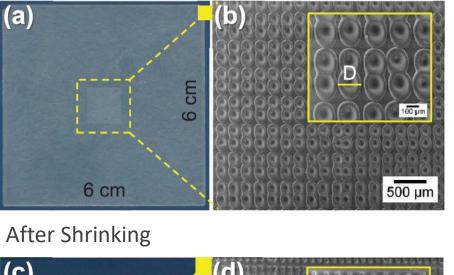




Goals: Thinner and high conformable sensor layers; high microstructure features resolution; high sensitivity al low-pressure regimes and fast recovery times.

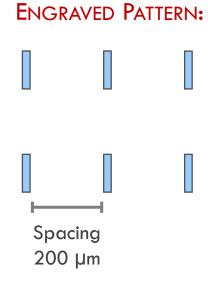


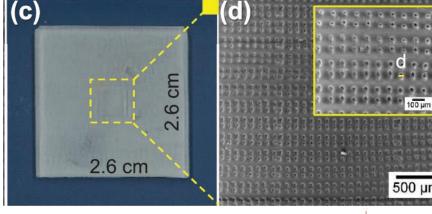
Before Shrinking



- Shrinking polymer films (SPF)
- Ag NWs as conductive layer
- It shrinks with temperature (160 °C)
- If shrunken after engraving, the cavities get smaller: possibility to achieve smaller structures
- Shrunken SPF can withstand temperature during Ag NWs spray coating

Adv. Mater. Interfaces 8 (21), 2100877, (2021)

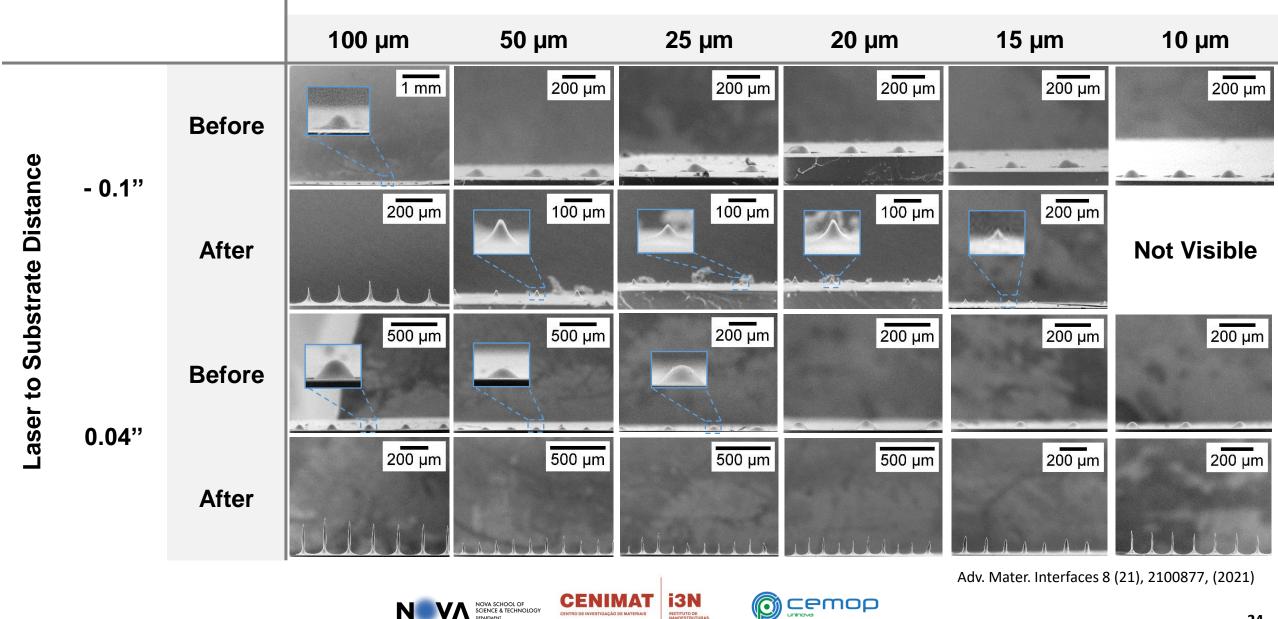


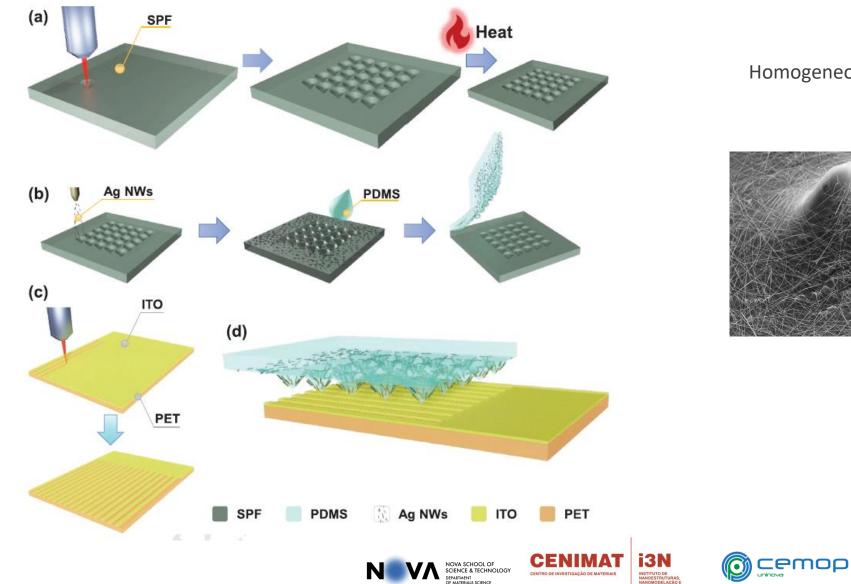


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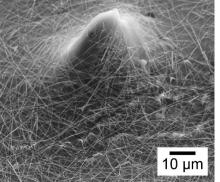


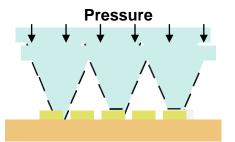
Piezoresistive e-skin pressure sensors Crosses Size





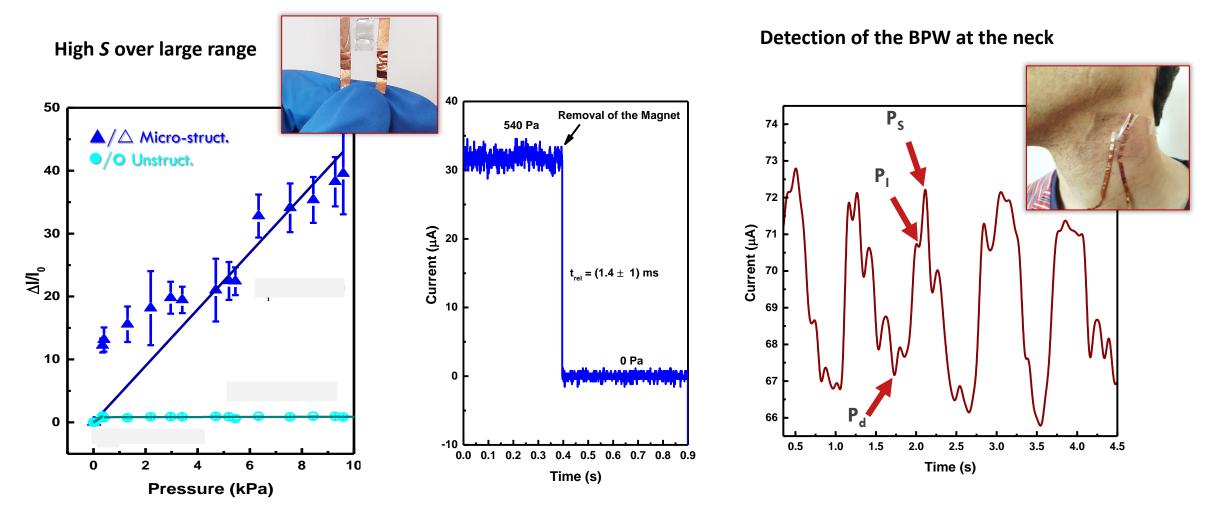
Homogeneous coverage of Ag NWs (except tip)





Pressure sensing mechanism

Adv. Mater. Interfaces 8 (21), 2100877, (2021)



i3N

sensitivity of -1.4 kPa⁻¹ below 10 kPa, a 1.4 ms recovery time

Adv. Mater. Interfaces 8 (21), 2100877, (2021)





Swallowing



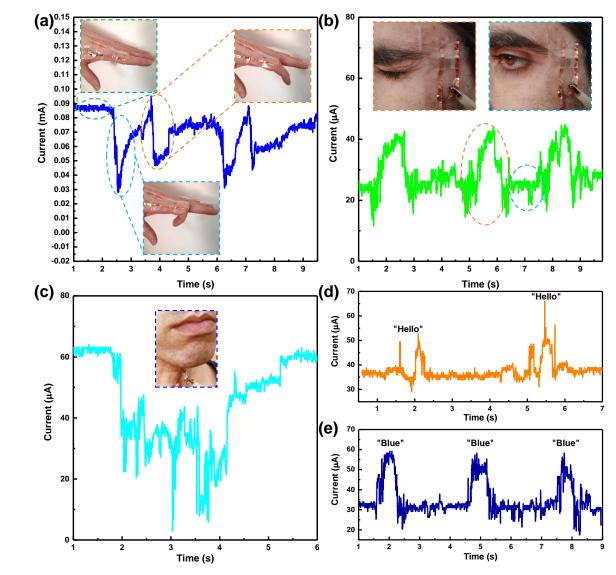
Speaking: "Blue"

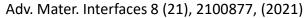


Finger flexing



Eye blinking C





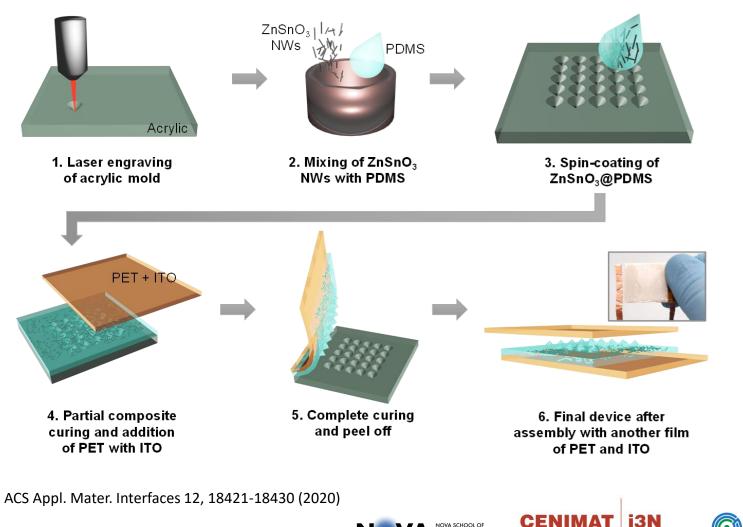






Energy harvesters using ZnO NRs and ZnSnO3 NWs

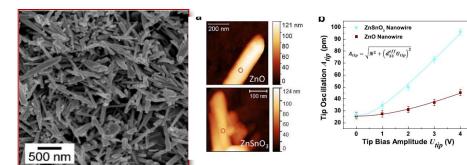
Hybrid ZnSnO₃ NWs @ microstructured PDMS nanogenerators





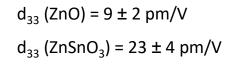
ZnSnO₃ nanowires

One step hydrothermal process @ 200 °C established @ CENIMAT



ACS Applied NanoMat 1(8), 3986 (2018) Nanomaterials, 9(7), 1002 (2019)

cemop

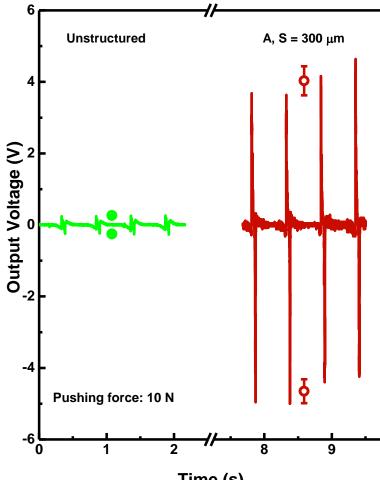


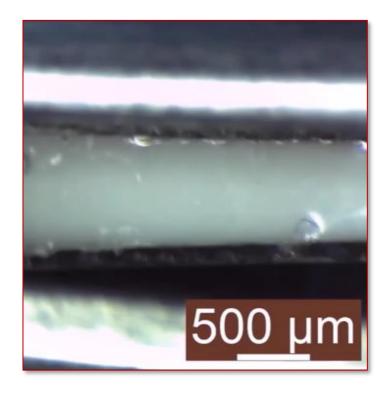
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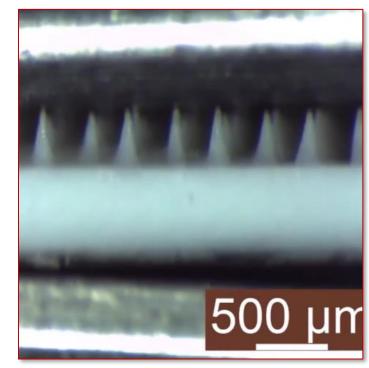


ENTRO DE INVESTIGAÇÃO DE MATERIA

Energy harvesters using ZnO and ZnSnO3 NWs







ACS Appl. Mater. Interfaces 12, 18421-18430 (2020)

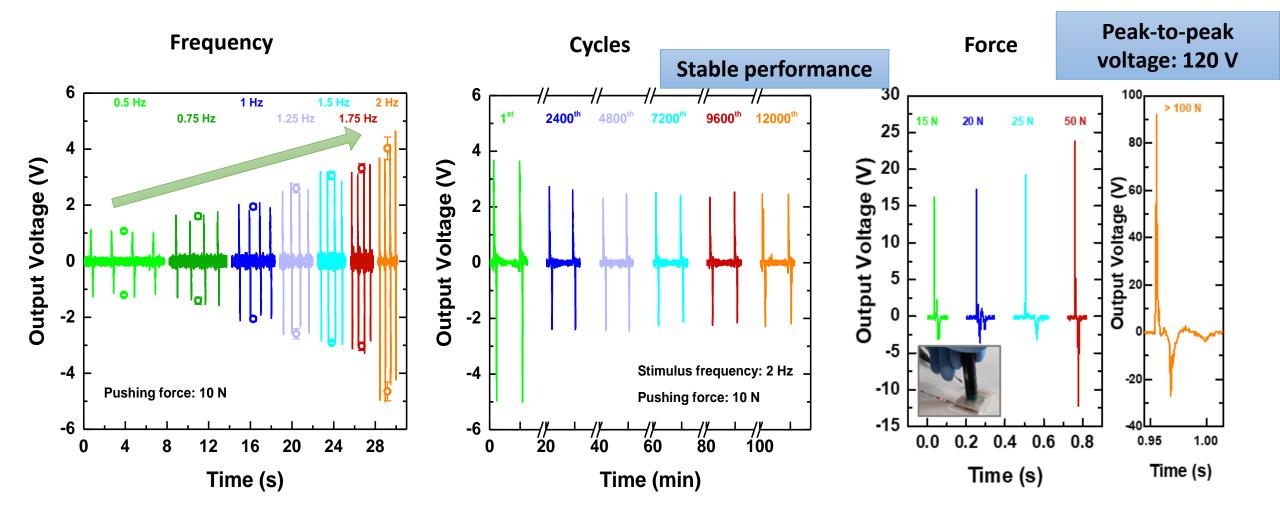








Energy harvesters using ZnO and ZnSnO₃ NWs



i3N

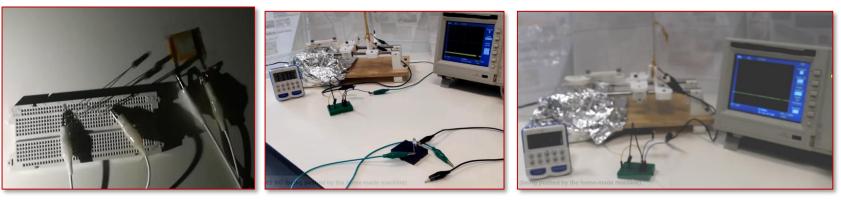
ACS Appl. Mater. Interfaces 12, 18421-18430 (2020)

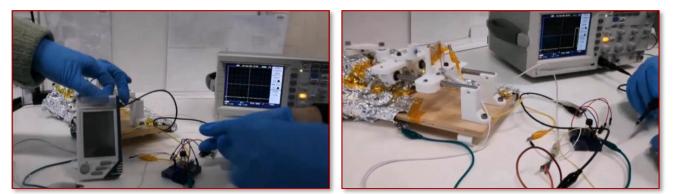
 \bigcirc





Energy harvesters using ZnO and ZnSnO3 NWs





 \ldots or charge a capacitor (15 min, 80 $\mu J)$ to power small electronic devices

Harvester able to directly power LEDs...

RETURN TO ISSUE < PREV RESEARCH ARTICLE NEXT >

Piezoelectricity Enhancement of Nanogenerators Based on PDMS and ZnSnO₃ Nanowires through Microstructuration

Ana Rovisco, Andreia dos Santos, Tobias Cramer, Jorge Martins, Rita Branquinho, Hugo Águas, Beatrice Fraboni, Elvira Fortunato, Rodrigo Martins, Rui Igreja*, and Pedro Barquinha*





Optimization of ZnO Nanorods Concentration in a Micro-Structured Polymeric Composite for Nanogenerators

by 🕐 Andreia dos Santos † 🗵 🔍 (Ų Filipe Sabino † 드, 🌒 Ana Rovisco * 🖾 🔍 (Ų Pedro Barquinha 교생), (Ų Hugo Águas 쯔 🔍 (Ų Elvira Fortunato 쯔, 😮 (Q Rodrigo Martins 쯔 👁 and (Ų Rui Igreja * ⊠ 📀

Institute for Nanostructures, Nanomodelling and Nanofabrication (i3N)/Centro de Investigação em Materiais (CENIMAT), Department of Materiais Science, NOVA School of Science and Technology (FCT-NOVA) and Center of Excellence in Microelectronics Optoelectronics and Processes (CEMOP)/Instituto de Desenvolvimento de Novas Tecnologias (UNINOVA), NOVA University Lisbon, Campus de Caparica, 2829-516 Caparica, Portugal

- Authors to whom correspondence should be addressed
- [†] These authors contributed equally to this work.

Academic Editor: Simas Rackauskas

Chemosensors 2021, 9(2), 27; https://doi.org/10.3390/chemosensors9020027

Received: 28 December 2020 / Revised: 27 January 2021 / Accepted: 28 January 2021 / Published: 31 January 2021

(This article belongs to the Special Issue Nanowire-Based Sensors)







ization of ZnO Nanorods ntration in a Micro-Structured

Polymeric Composite for

Outline

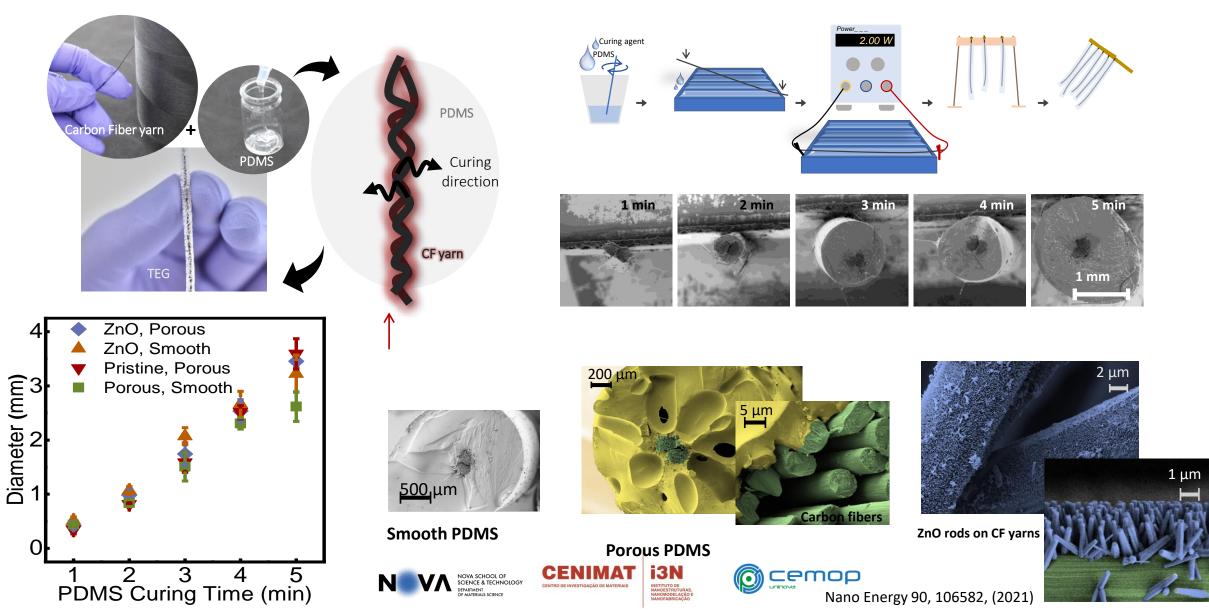
• CENIMAT | I3N and UNINOVA-CEMOP

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 - Piezoresistive temperature/pressure sensing
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- Fiber based devices
 - Energy harvesting devices
 - 1D-NEON projet; MIP2Sensors Projet
- Digital µFluidics

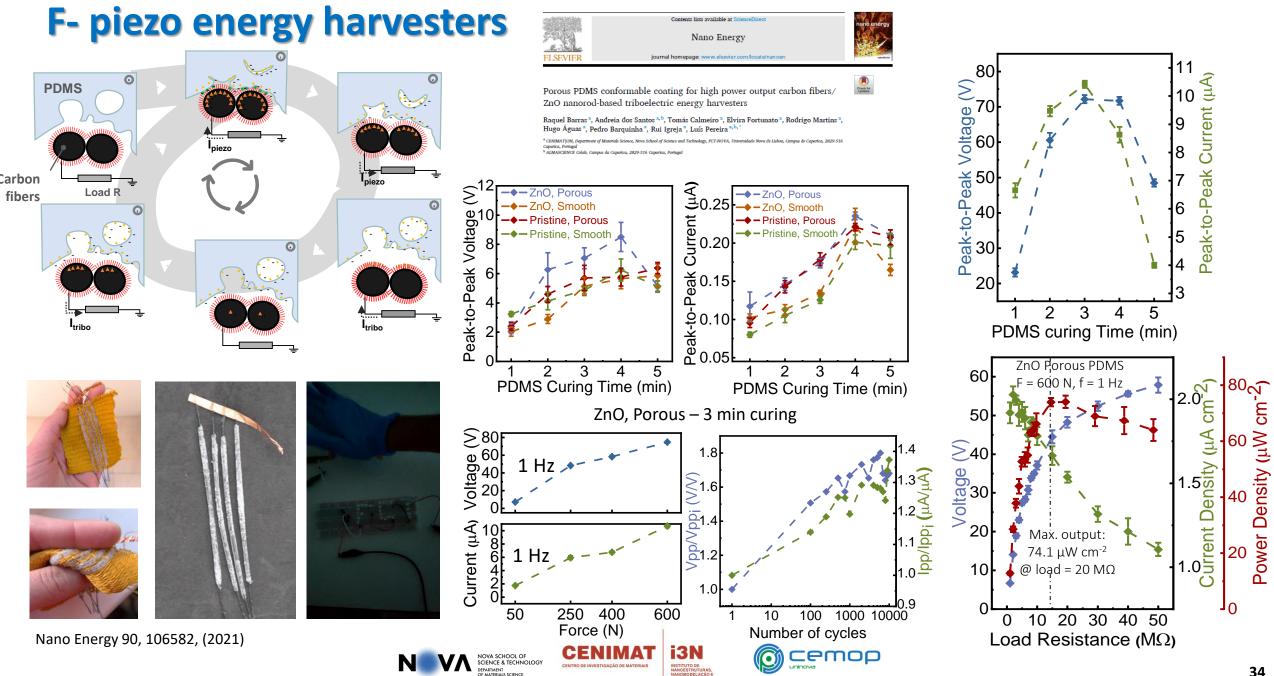


F- piezo energy harvesters

Porous PDMS conformable coating for high power output carbon fibers based triboelectric energy harvesters



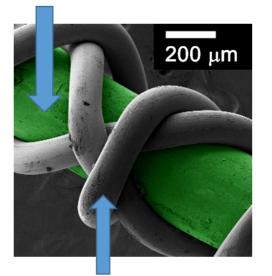
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F- piezo energy harvesters

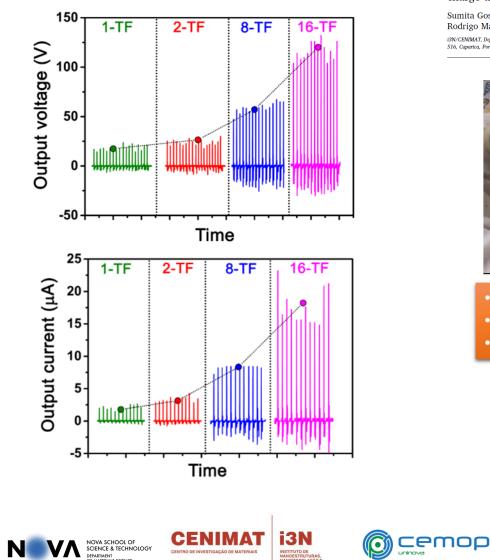
Doped polyaniline (d-PANi) functionalized textile fibre (TF) as energy harvester

Polyaniline functionalized layer



Conducting fibre (150 µm) knitted over functionalized fibre: Acts as stress-deliverer; charge-collector (SDCC) electrode

Nano Energy, 60, 794-801 (2019)





Contents lists available at ScienceDirect

Nano Energy

journal homepage: www.elsevier.com/locate/nanoen

Full paper

Check fo updates

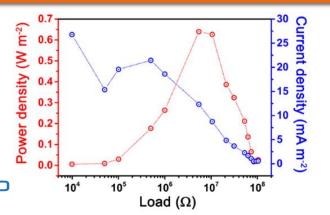
Human-motion interactive energy harvester based on polyaniline functionalized textile fibers following metal/polymer mechano-responsive charge transfer mechanism

Sumita Goswami, Andreia dos Santos, Suman Nandy*, Rui Igreja, Pedro Barquinha, Rodrigo Martins, Elvira Fortunato**

13N/CENIMAT, Department of Materials Science, Faculty of Science and Technology, Universidade NOVA de Lisboa and CEMOP/UNINOVA, Campus de Caparica, 2829-516, Caparica, Portugal



- Peak power-density ~0.6 W m⁻²
- Output current-density ~22 mA m⁻²
- Can power at least 10 white LEDs of 2.5 W



1D-NEON Project

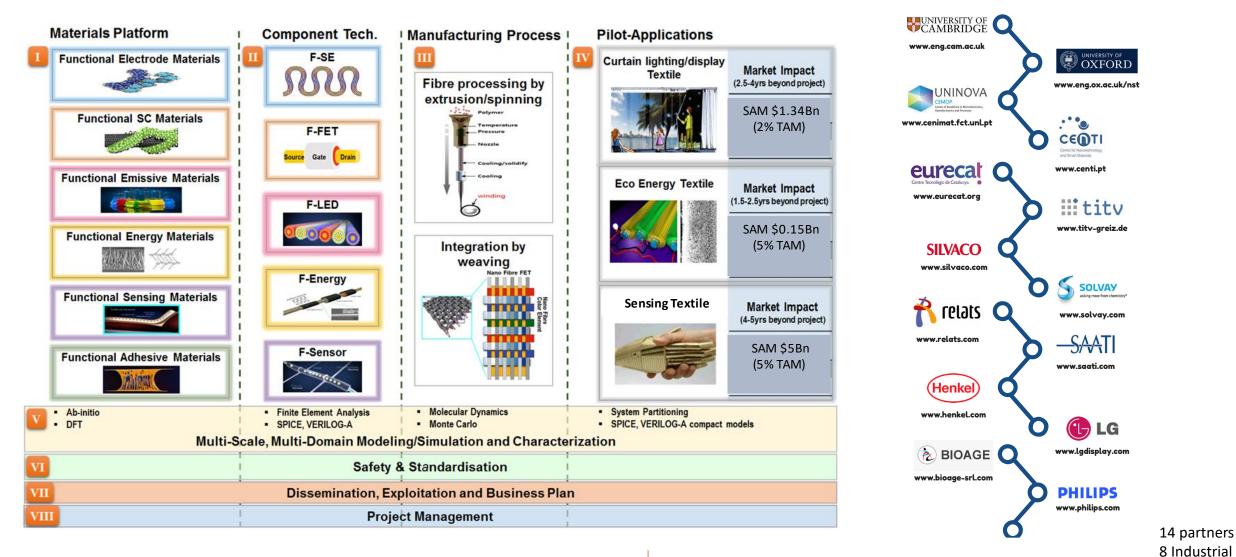
Coordinated by Professor Jong Min Kim, University of Cambridge, Department of Engineering

cemop

3 Universities

3 Res. Inst. 36

- Total project size €9.1M (€8M funding), start Apr. 2016 (48 months)
- Maturity Progression Plan TRL 4 to TRL 6



NOVA SCHOOL OF SCIENCE & TECHNOLOGY DEPARTMENT OF MATERIALS SCIENCE



F- piezo energy harvesters



nature

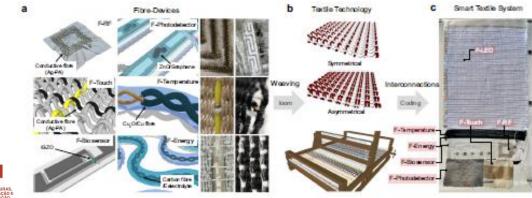
ARTICLE

Check for updates

https://doi.org/10.1038/s41467-022-28459-6 OPEN

Smart textile lighting/display system with multifunctional fibre devices for large scale smart home and IoT applications

Hyung Woo Choi^{1,20}, Dong-Wook Shin^{1,20}, Jiajie Yang^{1,20}, Sanghyo Lee^{1,20}, Cátia Figueiredo ², Stefano Sinopoli³, Kay Ullrich⁴, Petar Jovančić ⁵, Alessio Marrani⁶, Roberto Momentè⁷, João Gomes⁸, Rita Branquinho², Umberto Emanuele³, Hanleem Lee¹, Sang Yun Bang ⁵, Sung-Min Jung ¹, Soo Deok Han¹, Shijie Zhan ¹, William Harden-Chaters¹, Yo-Han Suh¹, Xiang-Bing Fan¹, Tae Hoon Lee¹, Mohamed Chowdhury ¹, Youngjin Choi¹, Salvatore Nicotera³, Andrea Torchia³, Francesc Mañosa Moncunill⁵, Virginia Garcia Candel⁵, Nelson Durães ⁸, Kiseok Chang⁹, Sunghee Cho⁹, Chul-Hong Kim⁹, Marcel Lucassen¹⁰, Ahmed Nejim¹¹, David Jiménez¹², Martijn Springer¹³, Young-Woo Lee^{14,15}, SeungNam Cha ^{14,16}, Jung Inn Sohn ^{14,17}, Rui Igreja², Kyungmin Song¹⁸, Pedro Barquinha ⁵, Rodrigo Martins², Gehan A. J. Amaratunga¹, Luigi G. Occhipinti ¹¹²³, Manish Chhowalla ¹⁹⁶² & Jong Min Kim ¹¹²³

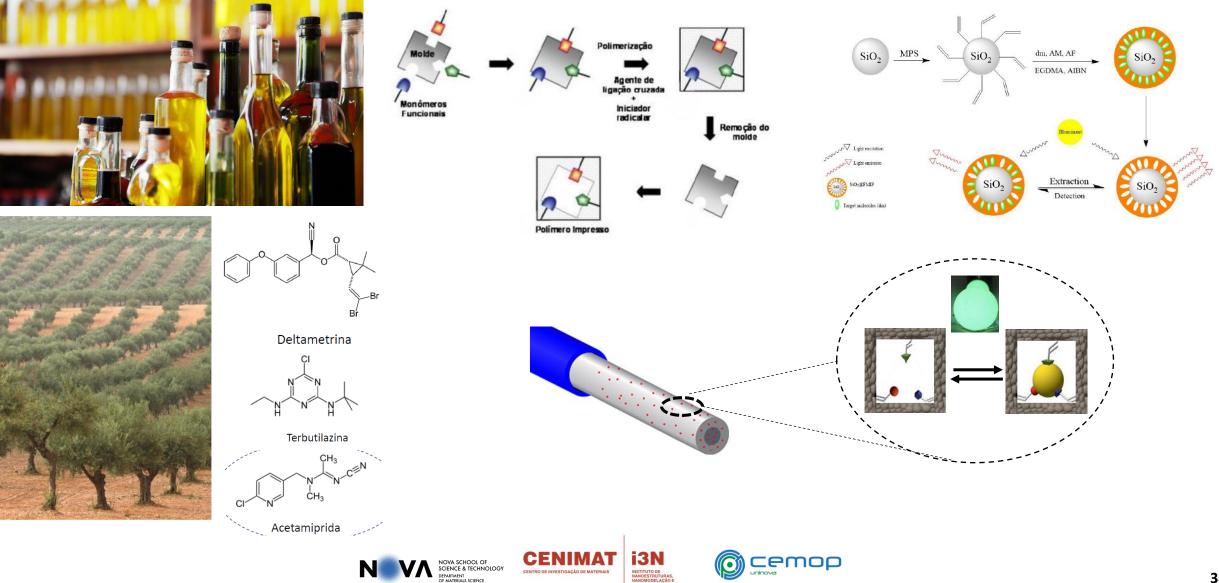


CENIMAT

ENTRO DE INVESTIGAÇÃO DE MATERIAL



MIP2Sensors (NOVA/Uni Évora)

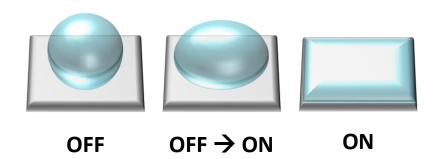


Outline

- CENIMAT | I3N and UNINOVA-CEMOP
- e-skin devices
 - Piezoresistive pressure sensors (health, robotics)
 - Piezoresistive using Temperature Shrinkink Polymer Moulds
 - Piezoresistive temperature/pressure sensing
 - Flexible Piezo/Tribo devices
- Fiber based devices
 - Energy harvesting devices
 - TFT for smart textiles
- Digital µFluidics



Voltage-based technology for automated processing of liquid Biological and Chemical reactions.

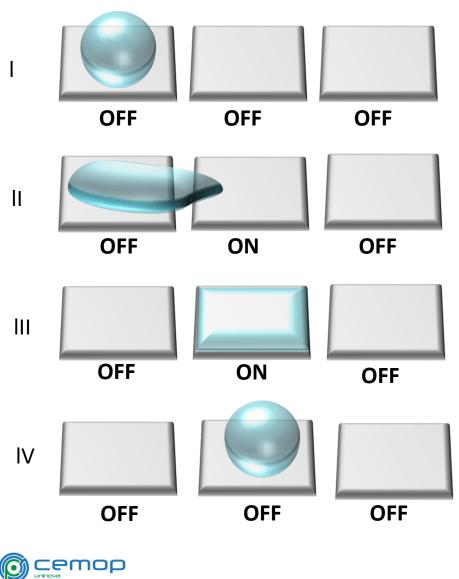


Any pathway is possible

Any electrode sequence is possible

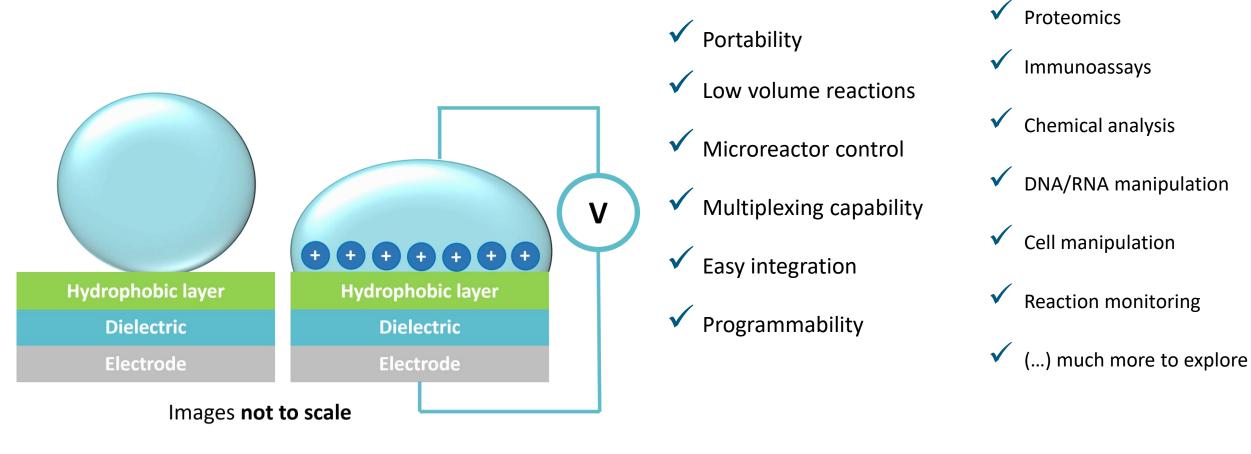
Any reaction/protocol order can be implemented

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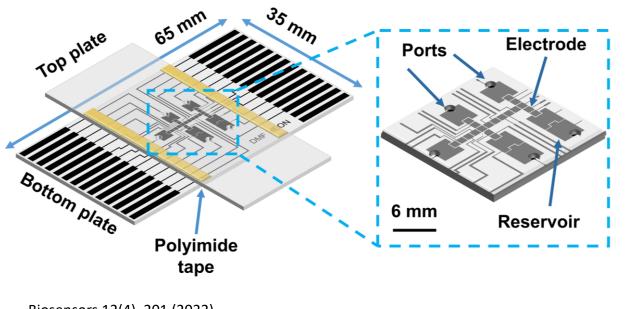


Electrowetting-on-dielectric (EWOD) phenomenon. Change in contact anlge in response to an electric field









Biosensors 12(4), 201 (2022)

• **DNA amplification for diagnostics**

Molecular diagnostics rely heavily on DNA amplification - **opportunity**

DMF for DNA-based diagnostics

Due to its unique combination of automation and low volume droplet handling, such devices are promising candidates for point-of-care testing (POCT) - challenge

Multiplex assays

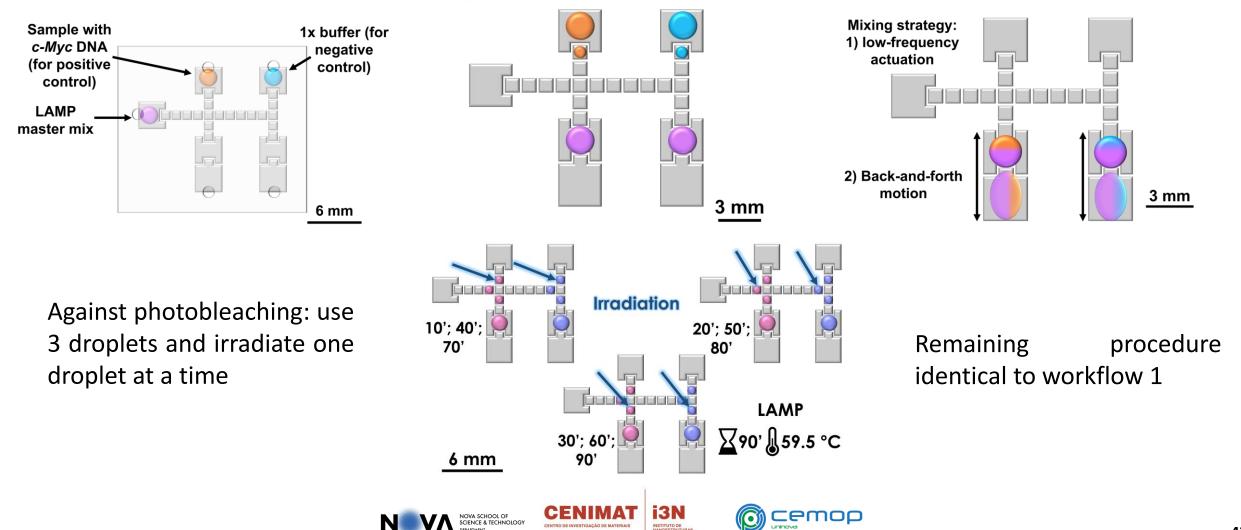
Shifting the electrode architecture allows for several assay configurations, namely multiplex assays, where two or more experiments run simultaneously – **one step further**



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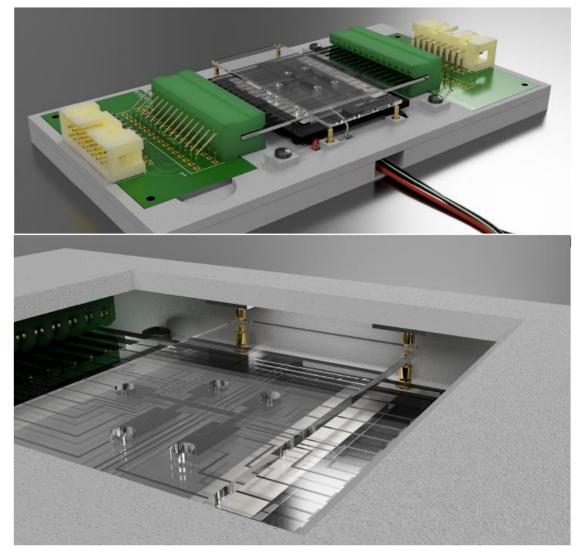
on-chip mixing

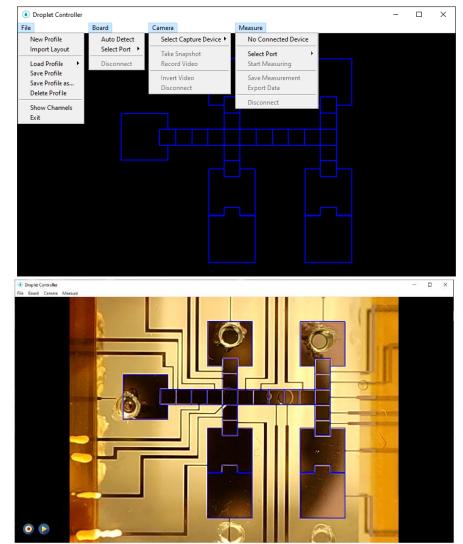


DEPARTMENT OF MATERIALS SCIENCE









Biosensors 12(4), 201 (2022) Sensors 17, 2616 (2017) Sensors 17, 1495 (2017)







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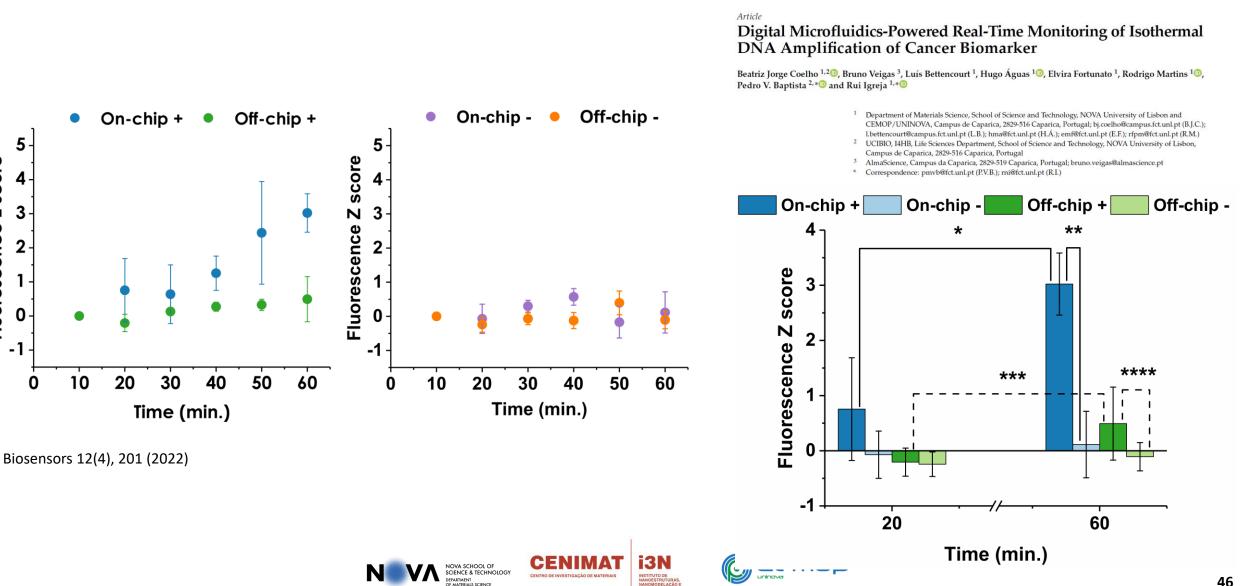
2

0

-1

Z score

Fluorescence



biosensors

MDPI

Supporting publications from MEON group

Nature Communications 13, 814 (2022)

Biosensors 12(4), 201 (2022)

Advanced Materials Interfaces 8 (21), 2100877, (2021)

Nano Energy 90, 106582, (2021)

Nanopores, IntechOpen 1, (2021)

Chemosensors 9 (2), 27, 2, (2021)

ACS applied materials & interfaces 12 (16), 18421-18430, 17, (2020)

Sensors 20, 4407, (2020)

Nano Energy, 60, 794-801 (2019)

Sensors 19, 899, (2019)

Nanomaterials, 9(7), 1002 (2019)

Advanced Electronic Materials 4 (9), 1870041, (2018)

Scientific reports 8 (1), 1-10 (2018)

ACS Applied NanoMat 1(8), 3986 (2018)

Sensors 17, 2616 (2017)

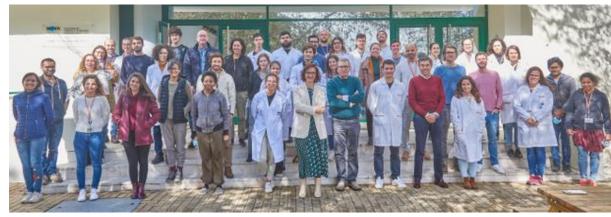
Sensors 17, 1495 (2017)



Rui Igreja, rni@fct.unl.pt

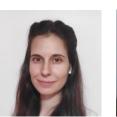
Acknowledgements

MEON group (as March 2022)





People more directly involved in the work presented













Post-doc: Sumita Goswani Suman Nandy Joana Neto

Collaborations:

Prof. Pedro Baptista (UCBIO) – DNA (LAMP) Prof. Raquel Garcia (Unv. Évora) Prof. Pedro Barquinha (i3N|CENIMAT) – Flexible electronics Prof. Luis Pereira (i3N| CENIMAT) – Fiber based devices Prof. Hugo Águas (i3N| CENIMAT) – Microfluidics



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Thank you for your kind attention!

