Graphene R&D and Industries in Thailand

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www.graphenethailand.com

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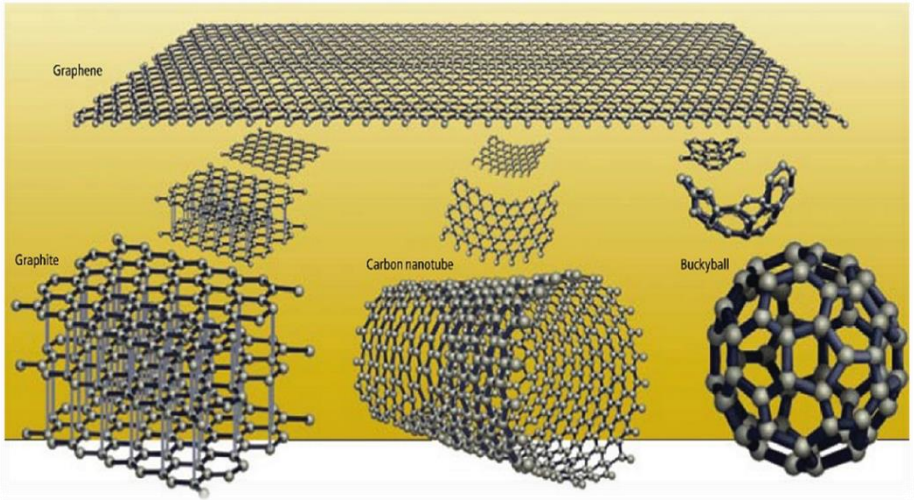
Graphene

Graphene is a flat monolayer of carbon atoms tightly packed into a two-dimensional (2D) honeycomb lattice, and is a basic building block for graphitic materials of all other dimensionalities. It can be wrapped up into 0D fullerenes, rolled into 1D nanotubes or stacked into 3D graphite



Preparation methods

TOP-DOWN

- Mechanical exfoliation
- Chemical oxidation
- Solution-based exfoliation
- Electrochemical exfoliation

BOTTOM UP

- CVD
- Epitaxial growth on SiC
- Organic synthesis

characteristic materials for supercapacitor

- **High specific surface area, the order of 1000 m^2/g**
- **Good intra- and inter-particle conductivity in materials**
- **Good electrolyte accessibility to surface of materials**

Advantage of graphene

- ✓ Large surface area *(about 2630 m^2 g^{-1}, it is double that of SWCNTs)*
- ✓ Large 2D electrical conductivity
- ✓ High electron transfer rate
- ✓ Transparent (97.7%) and Elastic
- ✓ Environmental friendly and Low cost

Graphene Synthesis

Graphite

- **Mechanical cleavage**
- **Direct Sonication**
- **Chemical vapor deposition (CVD)**
- **Graphite Oxide** → **Chemical or Thermal Reduction**
- **Electrolytic exfoliation**

Scotch tape

Novoselov et al, *Science* 2004, 306, 666

Graphite

Scotch Tape

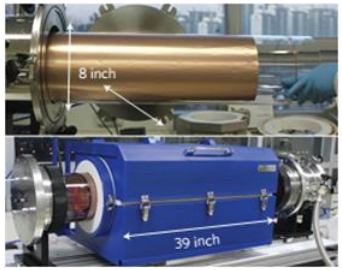
Nobel Prize

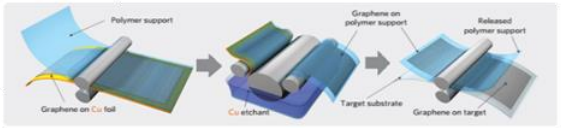
- **Advantages:**
  Pristine graphene
- **Disadvantages:**
  Low Production

surfactant

Lotya et al, *JACS* 2009 131, 3611

Sukang Bae et al, Nature Nanotechnology 2010





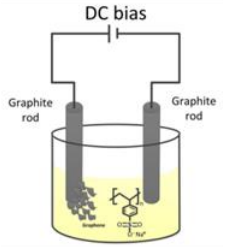
- **flexibility and conductivity of graphene films**
- **large-scale transparent electrodes**
- **replacing ITO**

Li et al, *Nature Nanotechnology* 2008, 3, 101

- **Advantages:**
  High Production
  Higher bulk density
- **Disadvantages:**
  Hazard chemicals
  Slow process

Schniepp et al, *J. Phys. Chem. B* 2006, 110, 8535

- **Advantages:**
  High Production
- **Disadvantages:**
  Slow process
  Low bulk density
  Hard to transport
  Safety issue



- **Stable in aqueous solution**
- **Easily scale up for large scale production**
- **Low cost**

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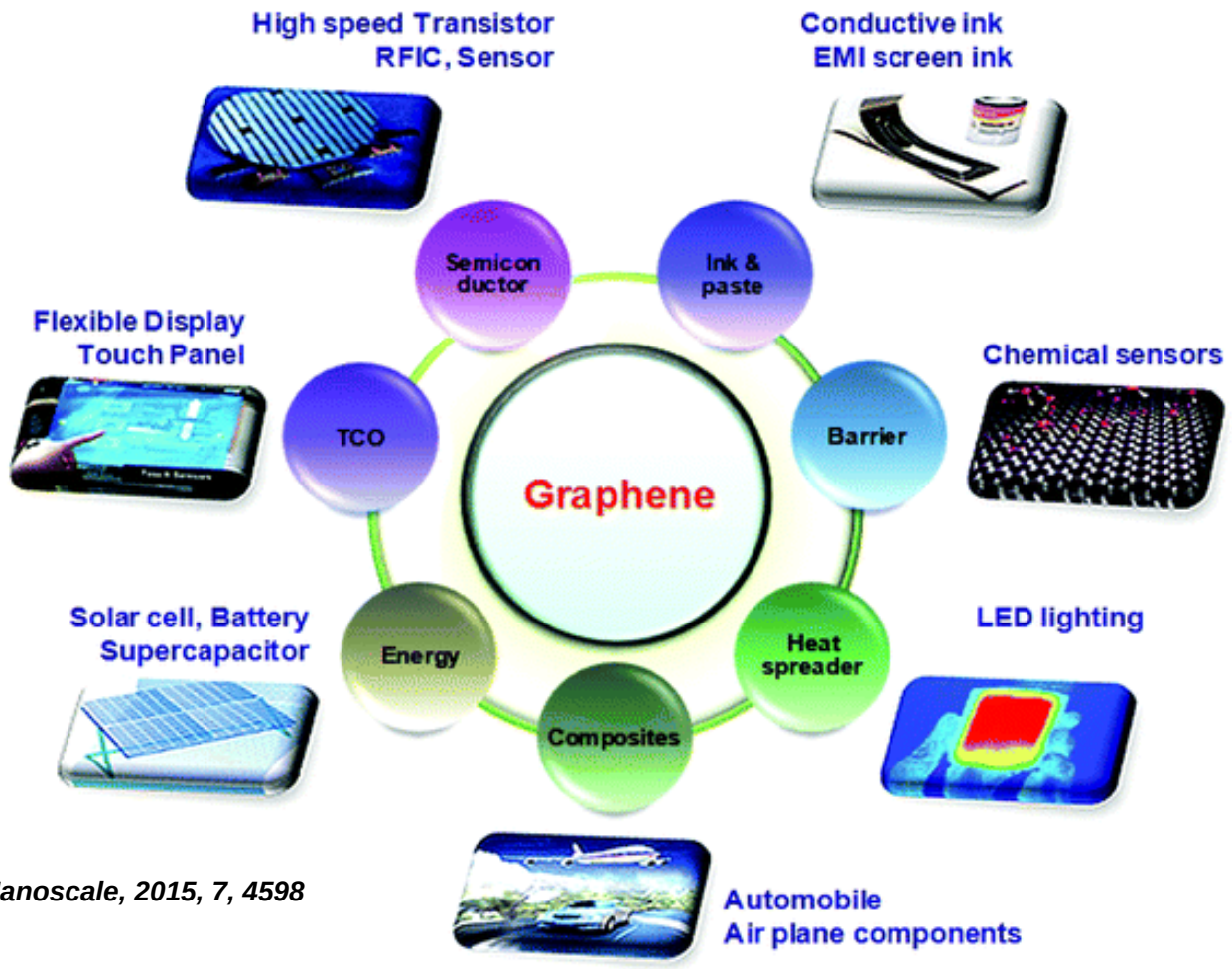
Applications of Graphene



Source: ***Nanoscale, 2015, 7, 4598***

Main Issues in Graphene Research

Practical limitations of graphene

- Agglomeration and aggregation into graphite
- Not easy to control size, density and distribution
- Cost and reproducibility in mass production

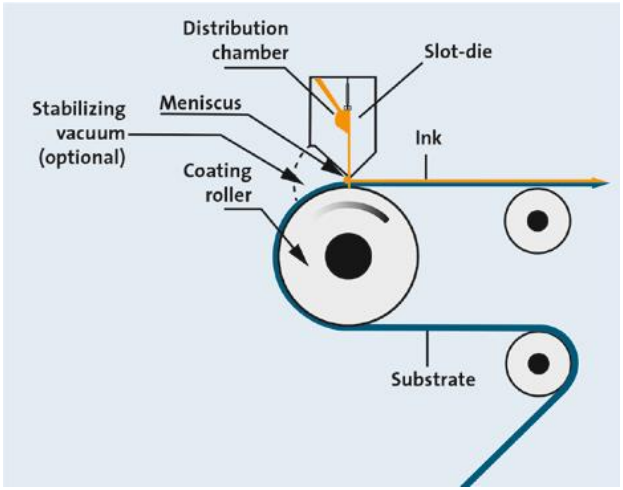
Potential solutions

- Apply spacer materials to prevent graphene agglomeration
- Forming well-defined 3D nanostructures of graphene
  - Self-assembly synthesis
  - Modification/Assembly of 2D graphene structure
  - *Templated growth and etching*

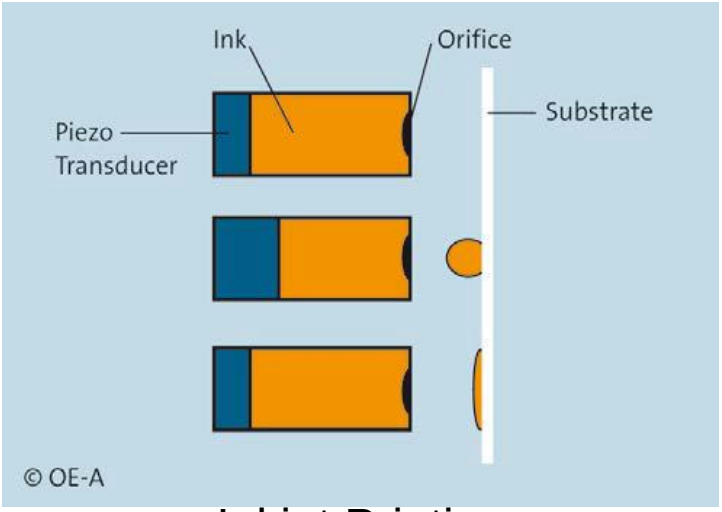
Printed Electronics

- ***Printed Electronics*** **is a revolutionary, new way of electronics: thin, lightweight, and flexible, produced at low cost, enabling single use, ubiquitous electronic devices and new applications**
- Printed electronics = Flexible elec. = Plastic elec.
- Fabrication
  - Printing (Inkjet, Screen, Offset , Flexo, Gravure, Roll-to-roll printing)
  - Nano Resolution Printing (Nano Imprinting)
- Benefits;
  - Cheap and easy
  - No vacuum requirement/ no complicated process
  - Cheap substrate such as paper or plastic can be used
  - Directly printing instead of lithography
  - Flexible and manufacturable for large area device (display, solar cell)
  - Band gap or mobility are chemically tunable

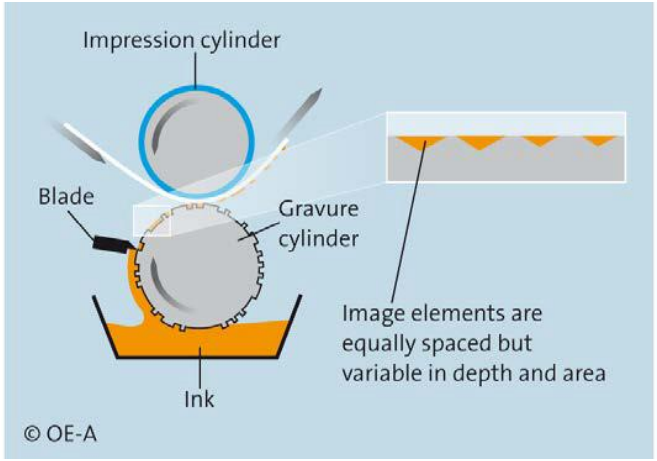
Printing Techniques for OPE



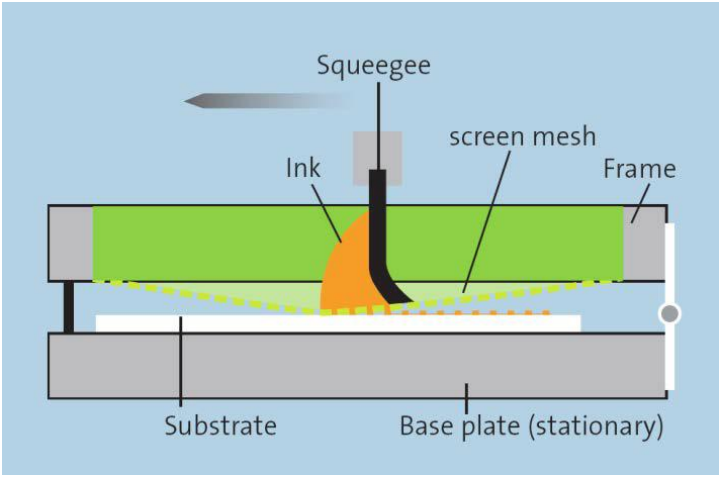
Slot-die coating



Inkjet Printing

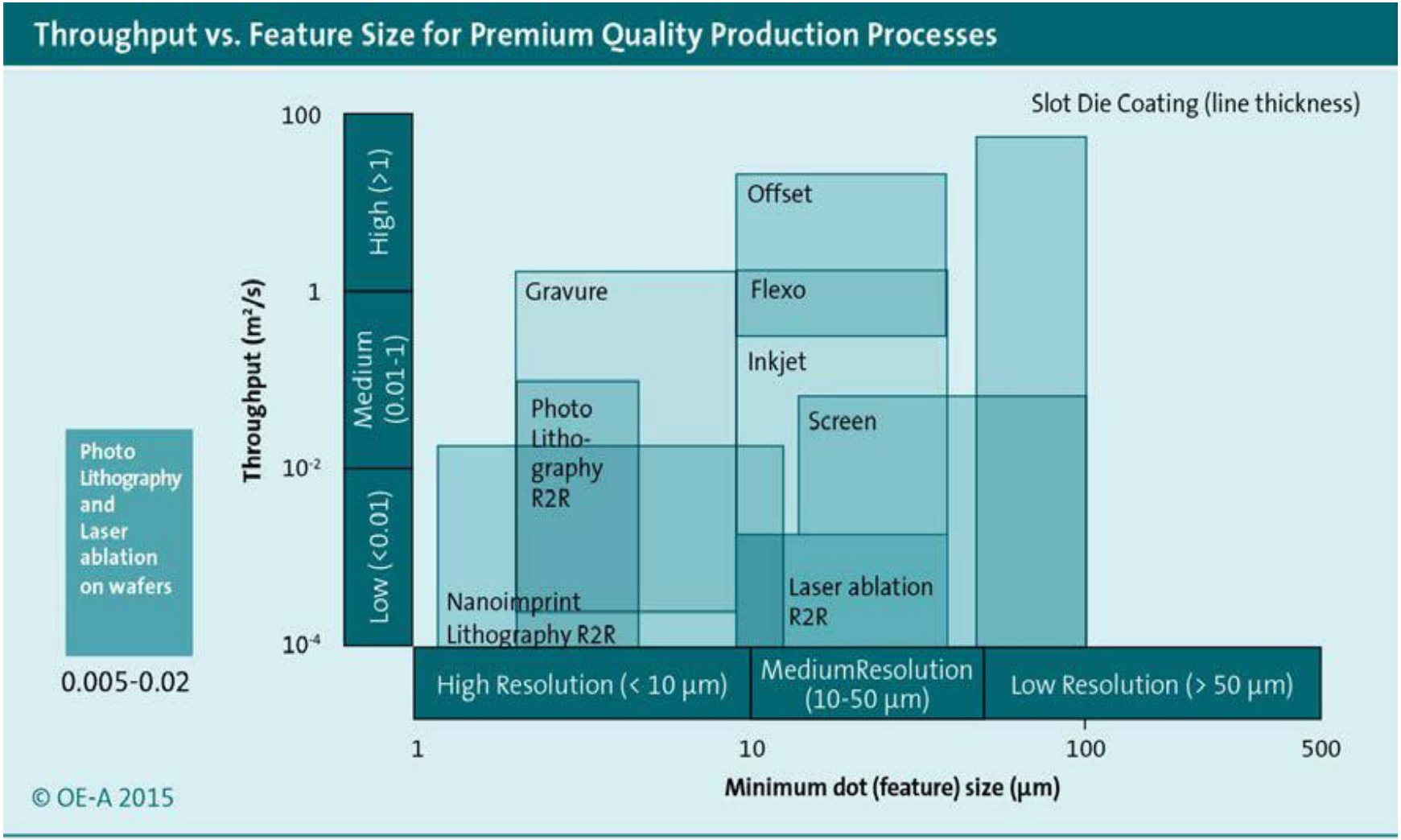


Gravure Printing

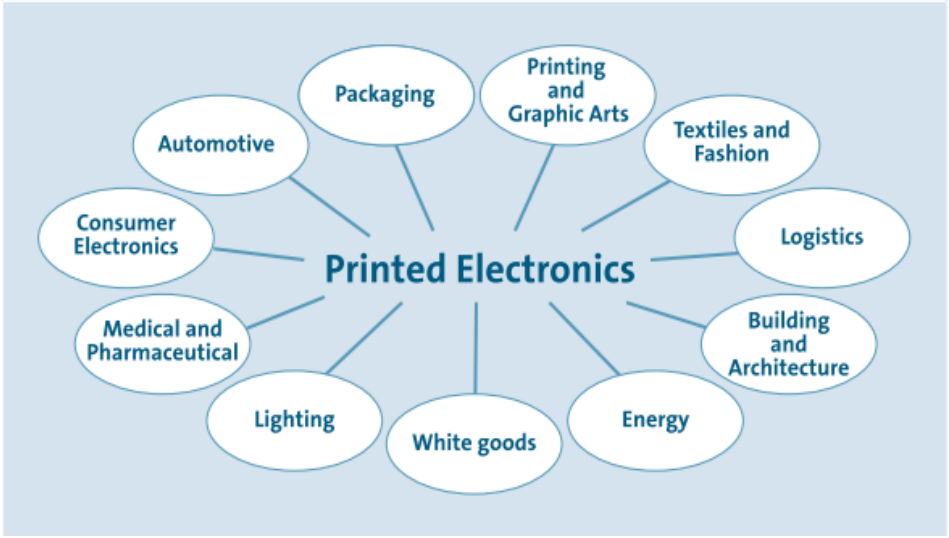


Screen Printing

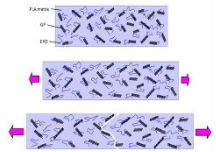
Printing as a production process



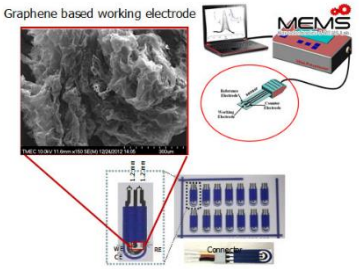
Organic and printed electronics enables new applications for numerous industries



Graphene Thailand R&D



Printed Electronic

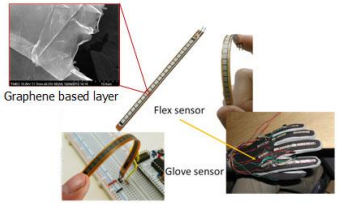


Composite

Sensors

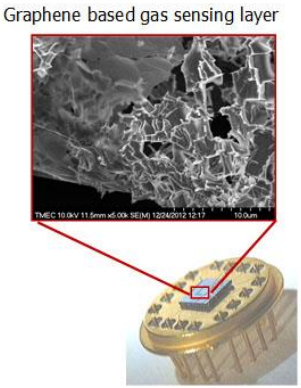
Graphene Synthesis

(2D and 3D)

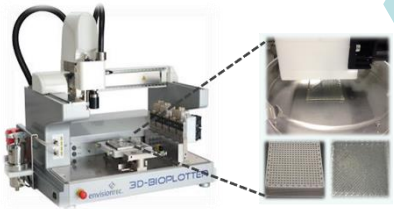


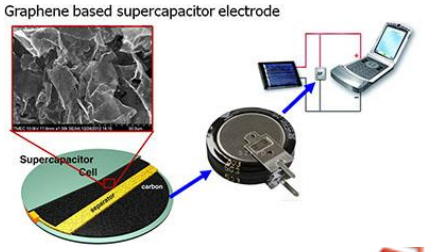
Lighting Signage

Supercapacitor Battery



3D Bio Printing





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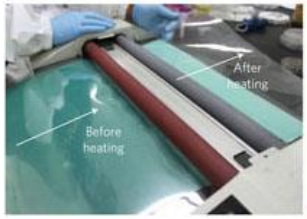
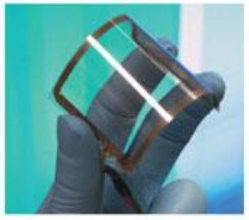
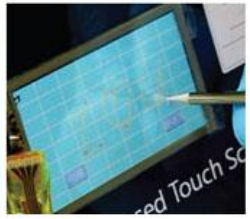
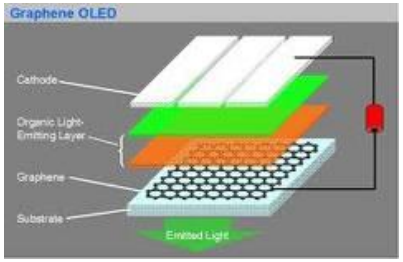
Conductive Ink in OPE

- Need of Conductive Ink
  - High electrical conductivity
  - Thin and robust film
  - High flexibility (stretchable)
  - Standard drying/curing/fusing equipment
  - Ease of processing with existing printing technology eg. Screen print, flexo, gravure printing.
  - Smart Card, RFID, Display, OLED, Solar cell …









Stretchable e-Paper

World's First Transparent Graphene Conductive Ink

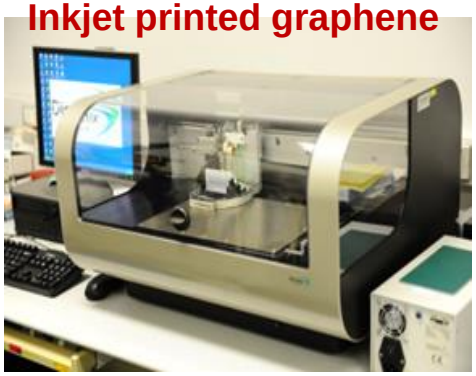
We can prepared transparent graphene conductive ink for printed electronics

- Synthesis graphene by electrolytic exfoliation
- Graphene dispersed solution as an ink for inkjet printing

Synthesis graphene as an ink

fabrication

Inkjet printed graphene



(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
18 May 2012 (18.05.2012)

PCT

(10) International Publication Number
WO 2012/064292 A1

(54) Title: A METHOD FOR PREPARING POLYMER/OXYGEN-FREE GRAPHENE COMPOSITES USING ELECTRO-CHEMICAL PROCESS

(57) Abstract: This invention demonstrates the preparation and use of polymer/oxygen-free graphene composites through an electrolytic exfoliation process. Two graphite rods are placed in an electrolysis cell filled with an electrolyte and a constant potential in the range of 3-10 V is applied between them using a regulated dc power supply for 4-10

Licensed technology to commercial

haydale

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GRAPHENE PRINTED ELECTRONICS

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Thailand's First Commercial Graphene Product

haydale

INNOPHENE
GRAPHENE PRINTED ELECTRONICS

PHENE PLUS

Transparent GRAPHENE CONDUCTIVE

HIGH PERFORMANCE
Transparent Electrodes

Conductivity Performance
With Graphene composite, it is a state of art between transparency and conductivity.

Thermal Performance
With Graphene composite, it is capable to get better and higher thermal performance.

Sensitivity Performance
With Graphene composite, it is enable to enhance more 200 times sensitivity than carbon base printed Bio-sensors.

More information, please contact
info@innophene.com

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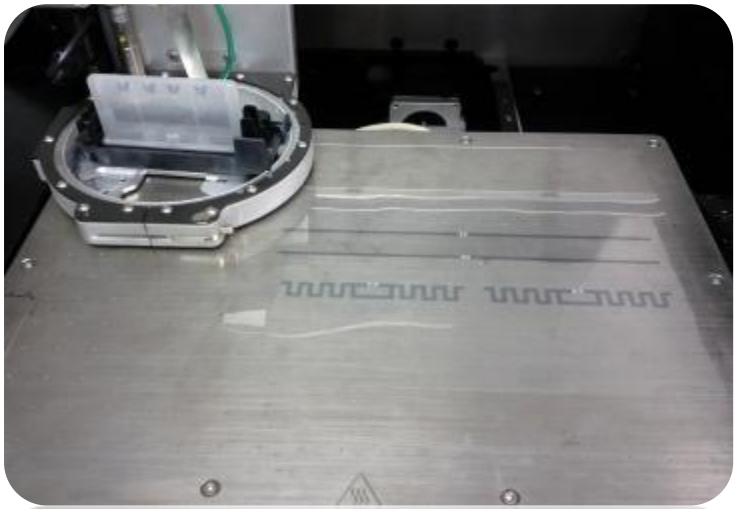
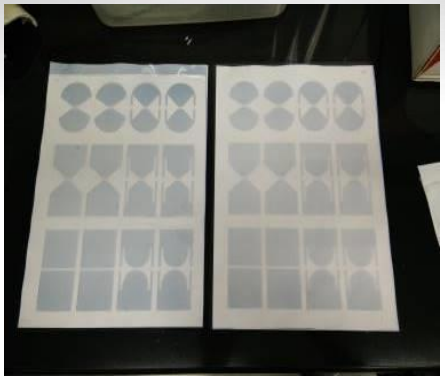
Printed Graphene RFID Antenna

Inkjet printed graphene antenna

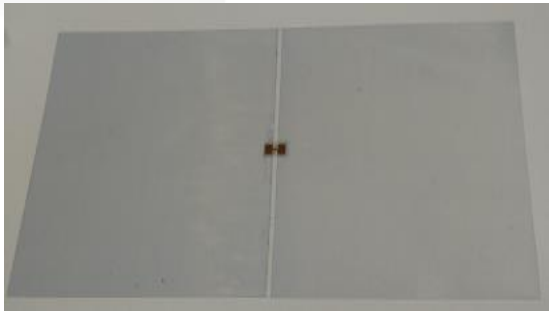


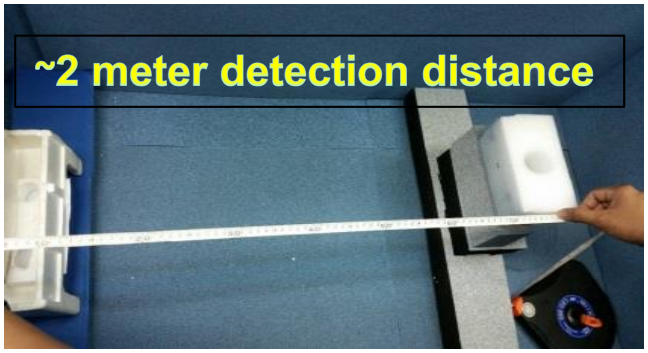
Transparent

Flexible



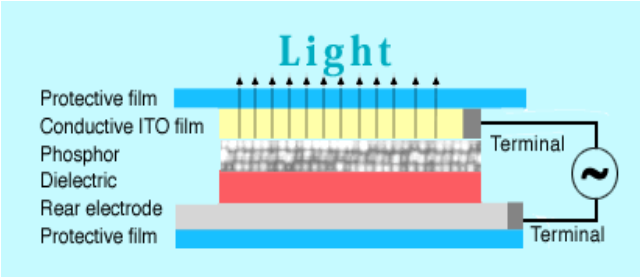
Dipole antenna





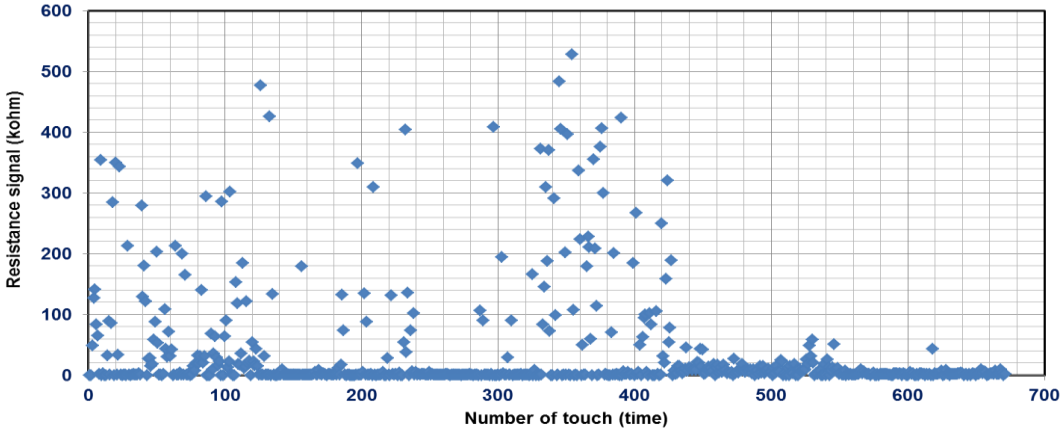
Printed Graphene Transparent electrode for Electroluminescence

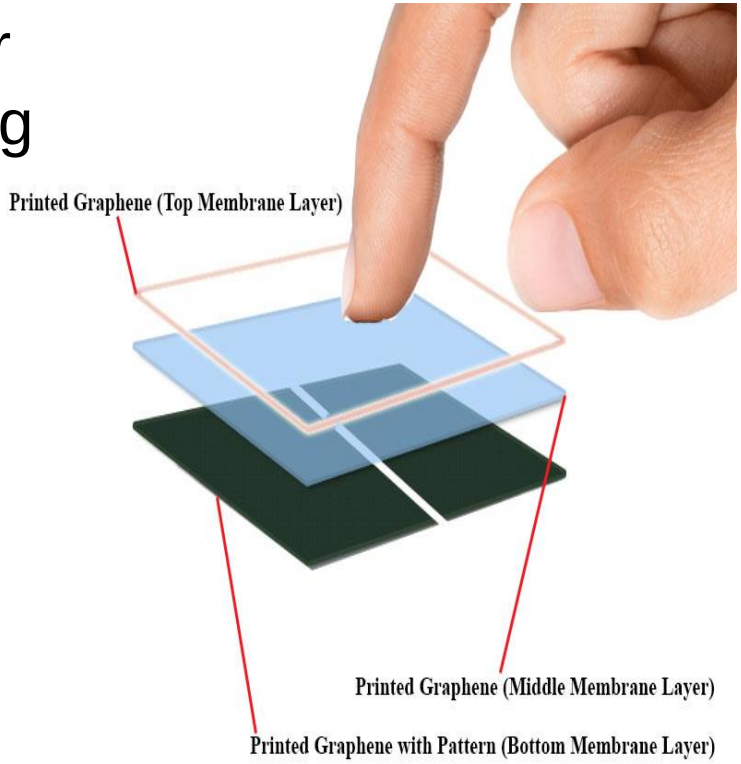
ITO was replaced by graphene



Flexible and Transparent Touch Sensor Based on Inkjet Printed Graphene using Capacitive and Resistive Techniques

Over 600 tested between press and not press of this sensor we can detect difference situation of switch-on and switch-of more than 98% in the humid finger.

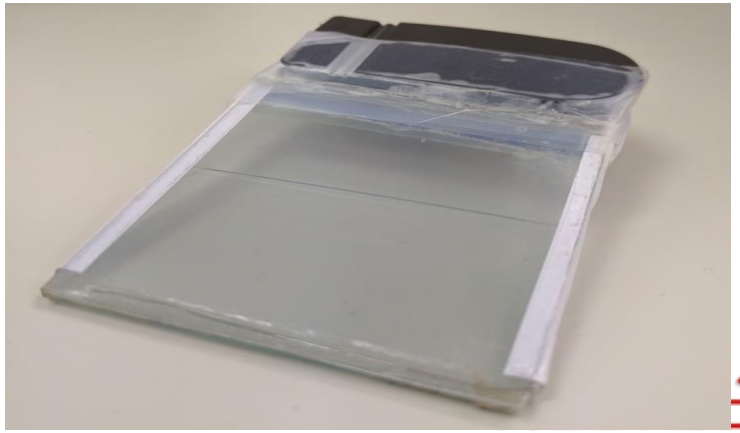




Demonstrate of Transparent Hybrid Touch Switch

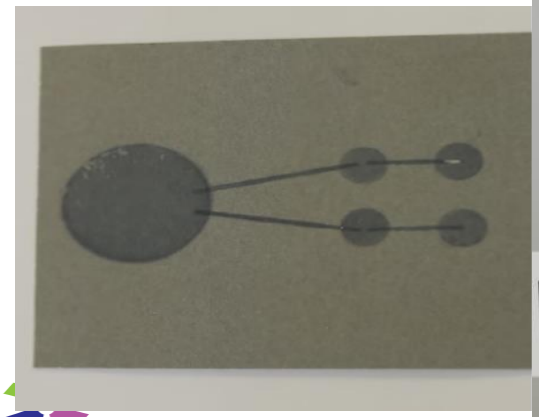
The First Prototype

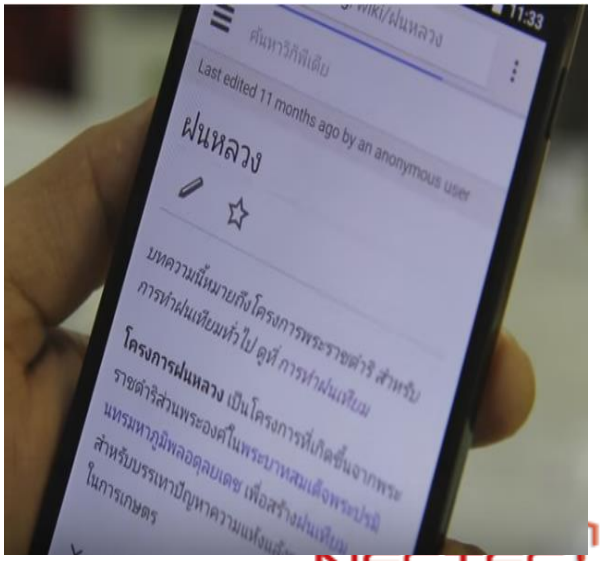
Second Prototype Wireless Transparent Touch Switch with battery



Printed Graphene Invisible Code for Smart Label

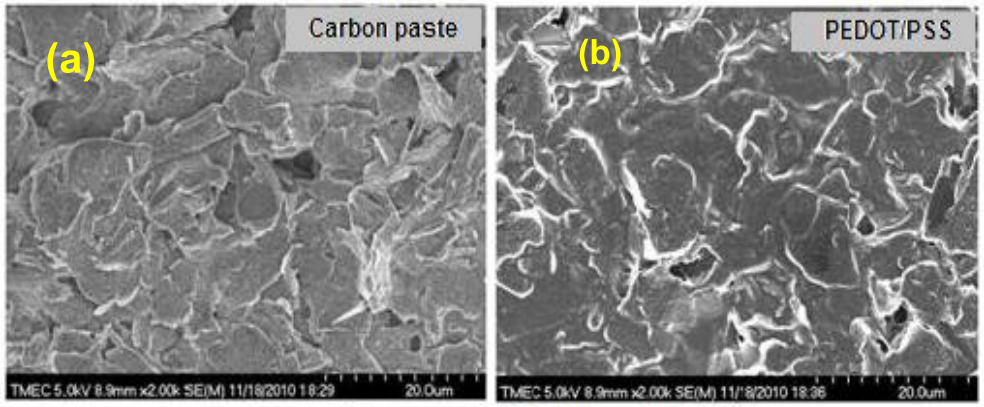
Code printed on packaging

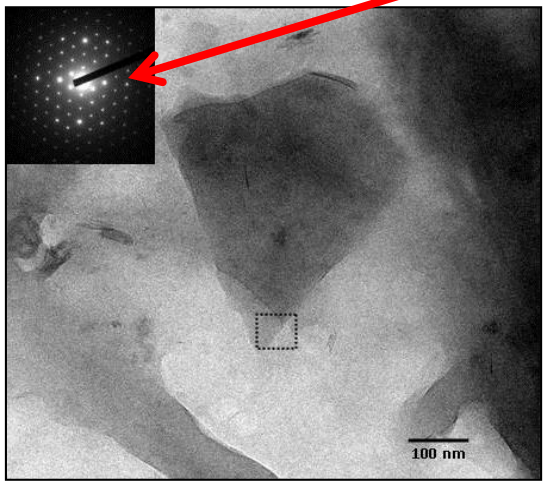




Inkjet-printed Graphene-PEDOT:PSS Applications

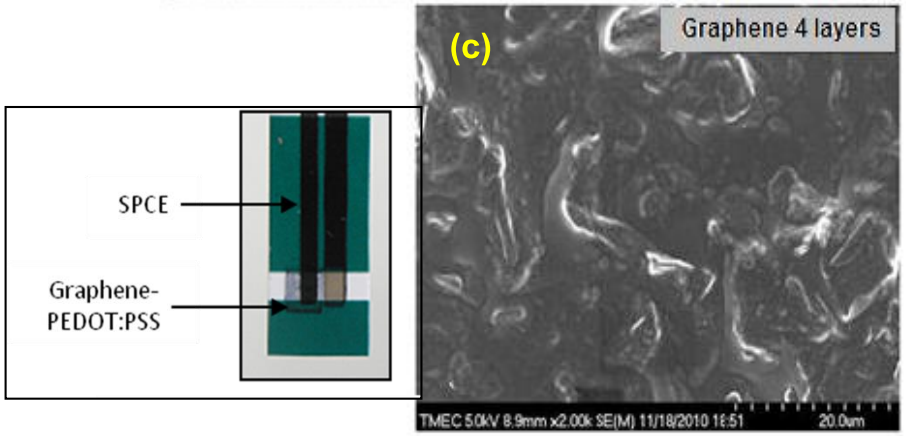
- Salbutamol (**Sensors and Actuators B: Chemical**, 2012)
- NADH, H_2O_2 (***J. Mater. Chem.***, 2012)





SAED pattern of a region near an edge of graphene sheet.

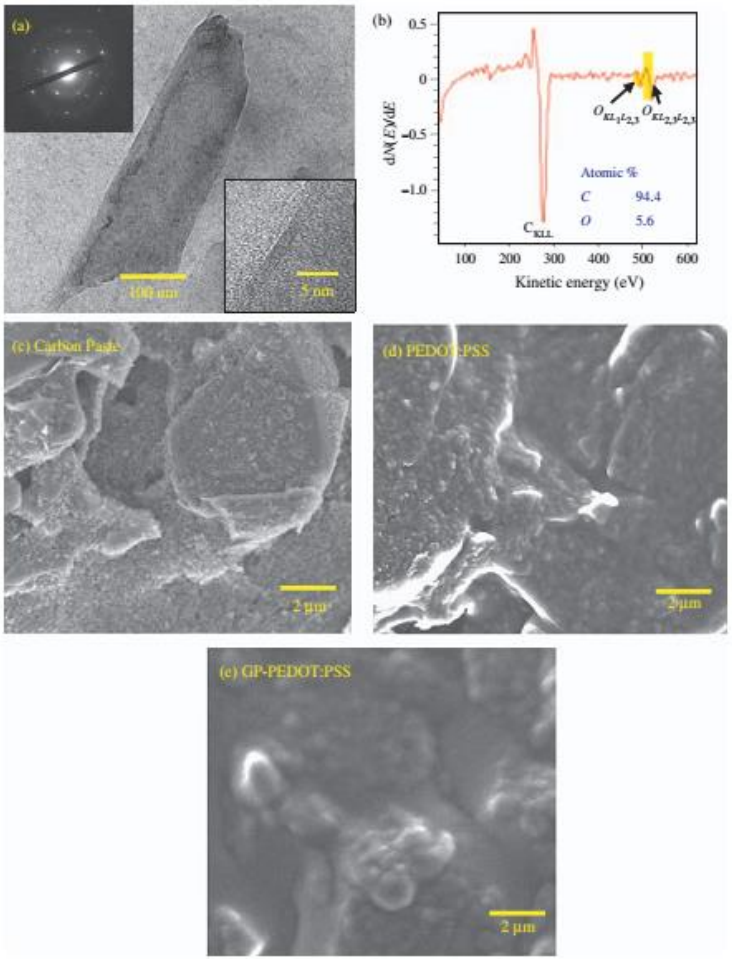
Bright field TEM image of Graphene-PEDOT/PSS composite.

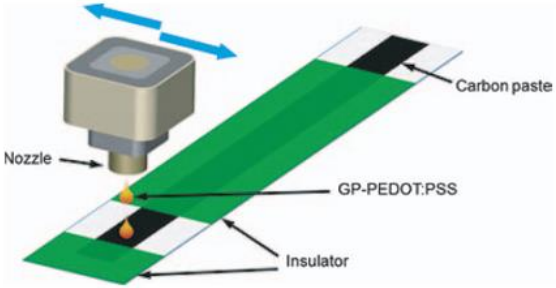


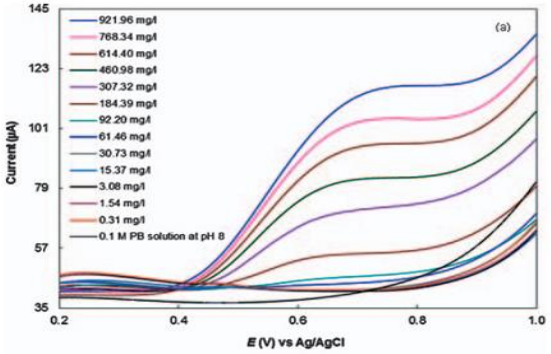
Photograph of fabricated electrode and SEM micrographs of (a) SPCE electrode, (b) inkjet printed PEDOT:PSS on SPCE electrode and (c) printed Graphene-PEDOT/PSS on SPCE electrode.

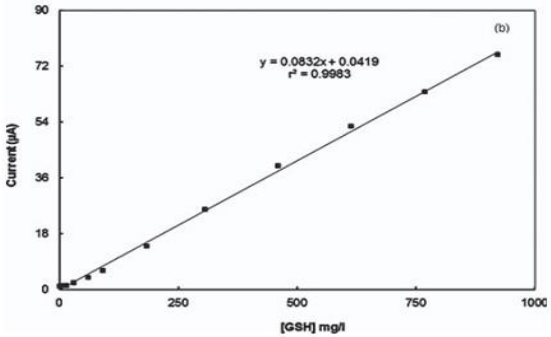
Graphene Hybrid Printing

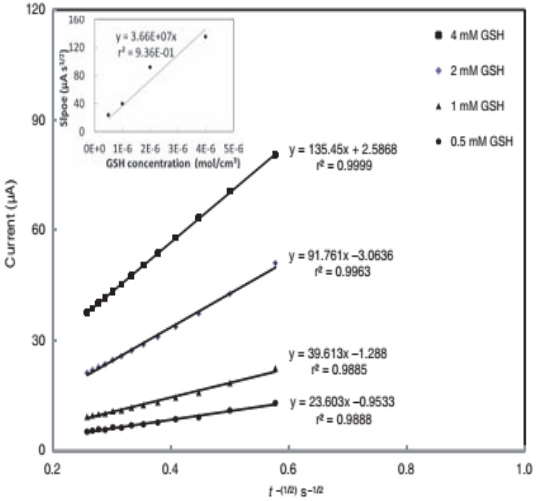
Electrochemical Detection of Glutathione Based on Inkjet-Printed Graphene Modified Screen Printed Carbon Paste Electrode









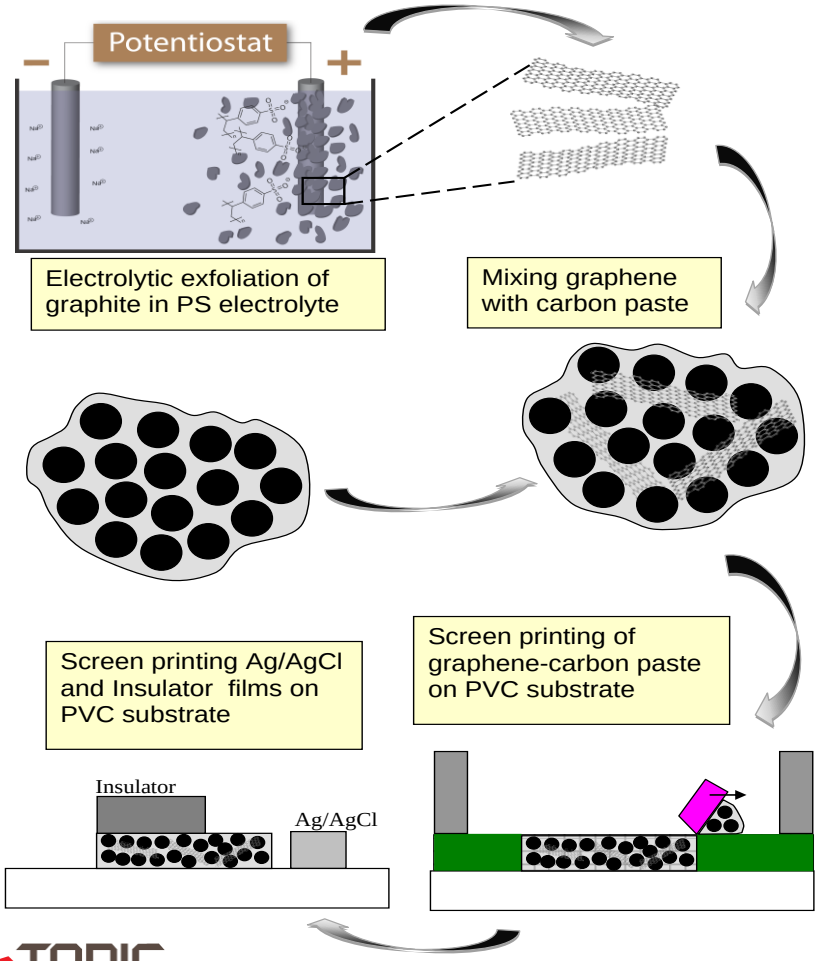


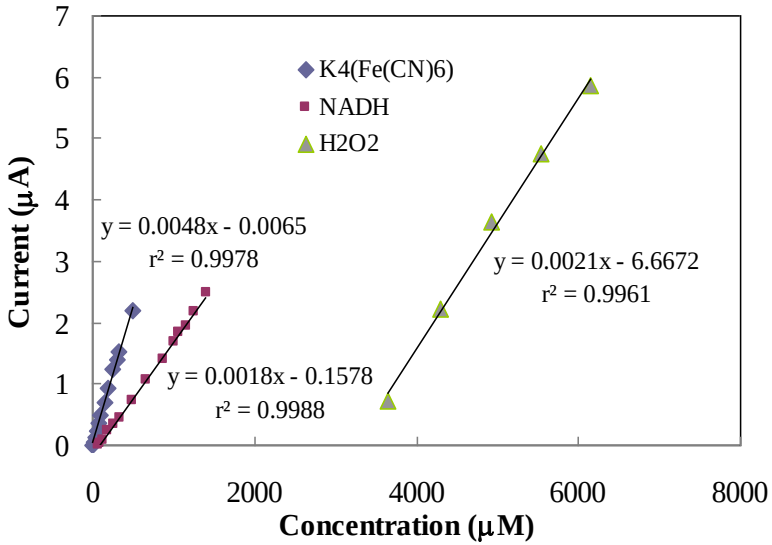
SENSOR LETTERS
Vol. 11, 1–9, 2013

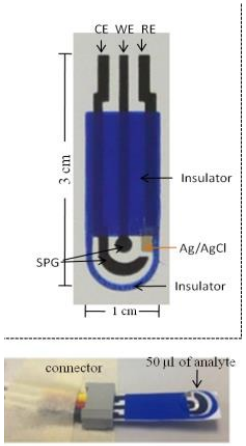
Electrochemical Detection of Glutathione Based on Inkjet-Printed Graphene Modified Screen Printed Carbon Paste Electrode

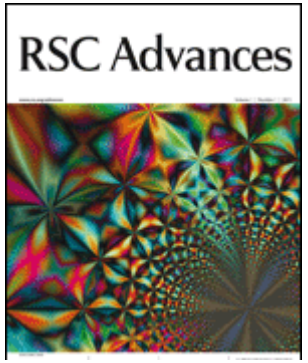
Screen Printed Graphene

Disposable screen printed graphene-carbon paste electrode and its application in electrochemical sensing









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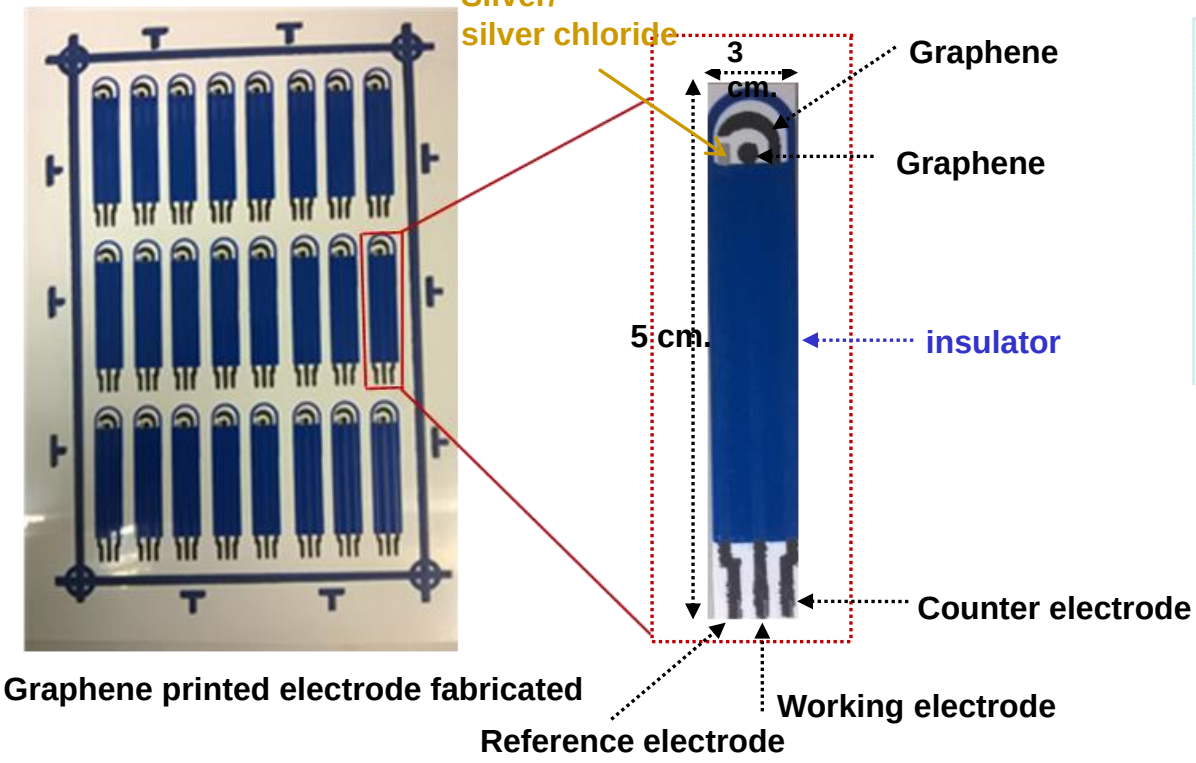
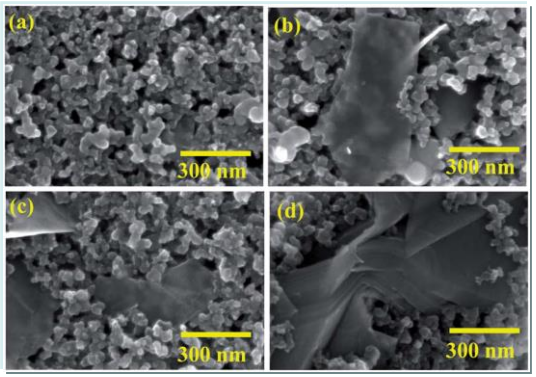
Graphene Screen Printed Electrode



Graphene printed electrode fabricated

Picture of graphene printed electrode and its components.



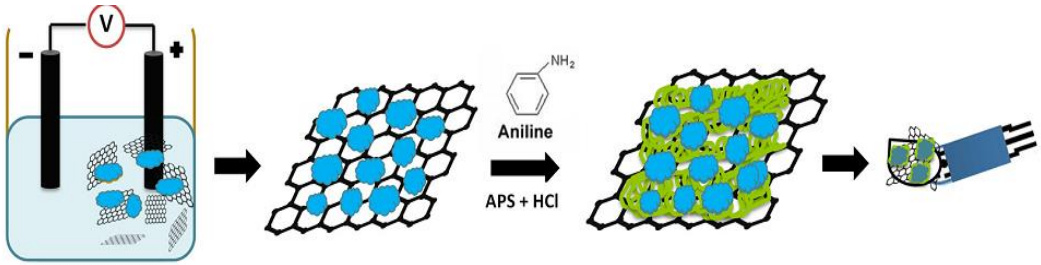
SEM micrographs of SPGEs with (a) 0%, (b) 5%, (c) 10% and (d) 15% graphene concentration.

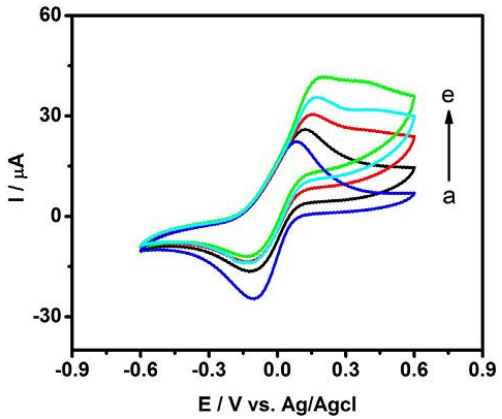
Advantages:

- **Easy to fabricate**
- **Low cost**
- **High electrical conductivity**

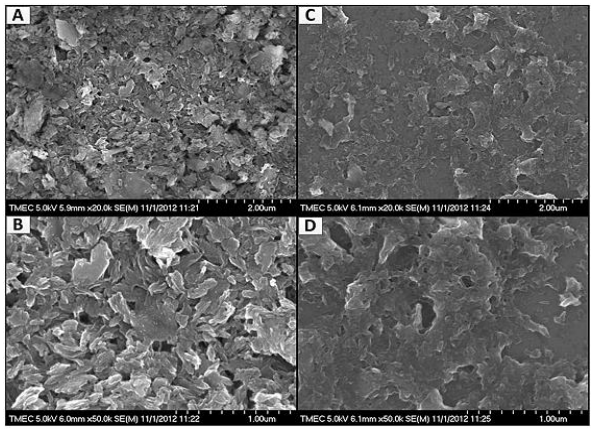
Graphene Hybrid Materials

Highly selective electrochemical sensor for ascorbic acid based on a novel hybrid graphene-copper phthalocyanine-polyaniline nanocomposites

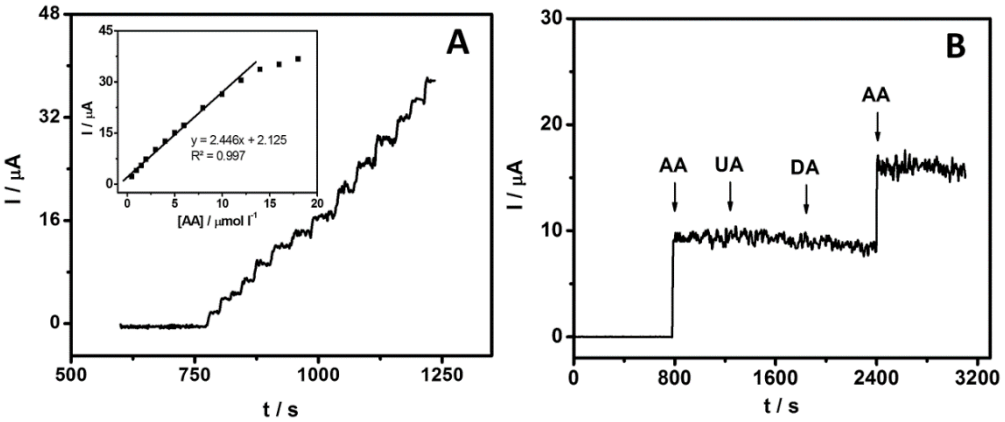




CVs of Gr/CuPc/PANI/SPE in 0.1M PBS pH 7.0 without AA (a) and with 50 μM(b), 0.1 mM (c), 0.25 mM(d) and 0.5 mM (e) AA at a scan rate of 20 mV/s.



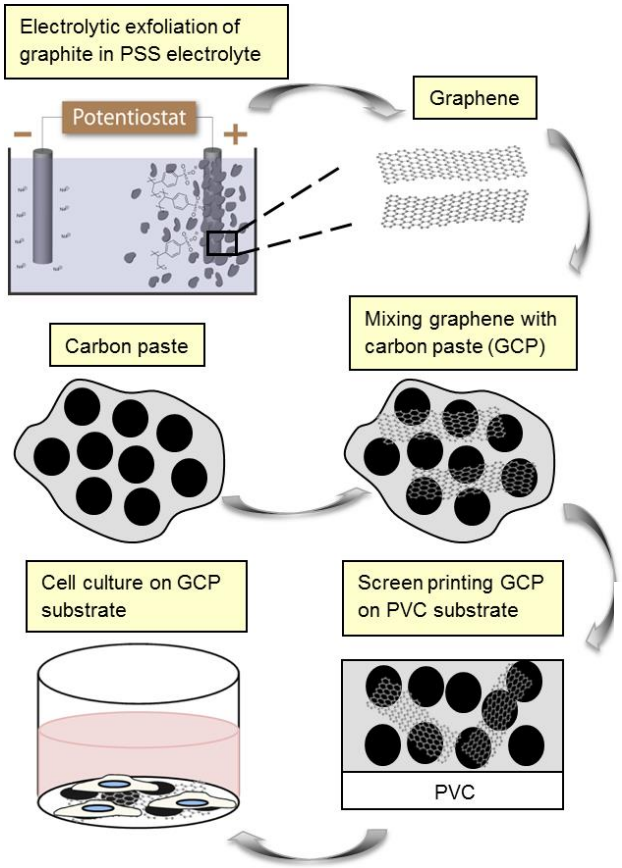
SEM images of (A, B) Gr/CuPc and (C, D) Gr/CuPc-PANI nanocomposites

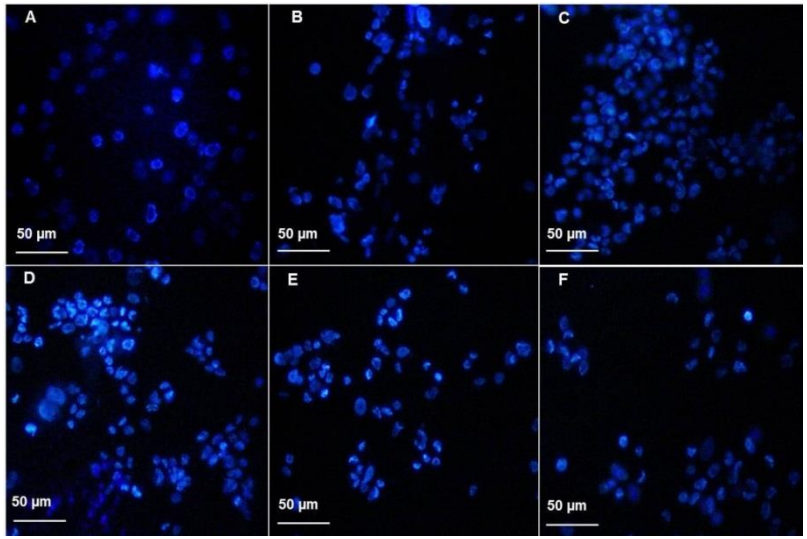


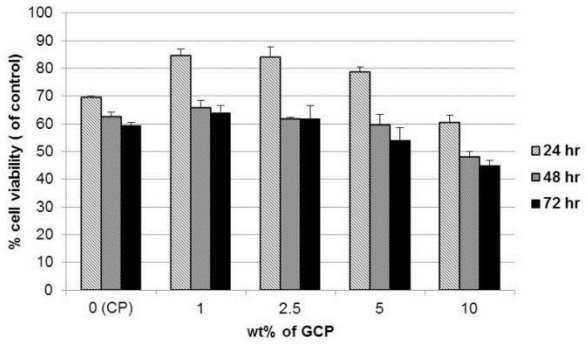
Amperometric current response of Gr/CuPc/PANI modified SPE to successive addition of different AA concentration.

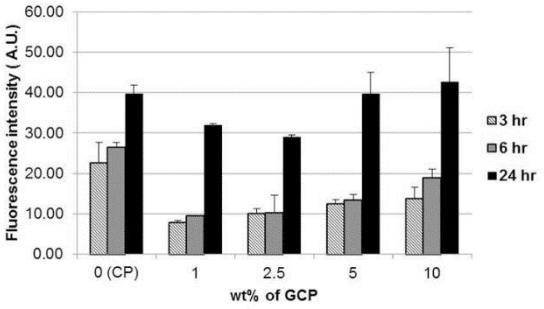
Cytotoxicity Assessment of Graphene Ink

Cytotoxicity assessment of MDA-MB-231 breast cancer cells on screen-printed graphene - carbon paste substrate









TOPIC
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Application: Glucose biosensors

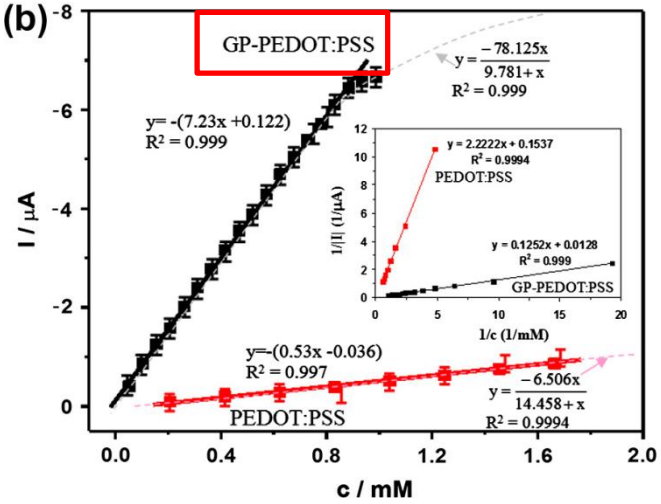
Graphene–PEDOT:PSS on screen printed carbon electrode for glucose biosensing

PEDOT-PSS

Graphen/PEDOT:PSS

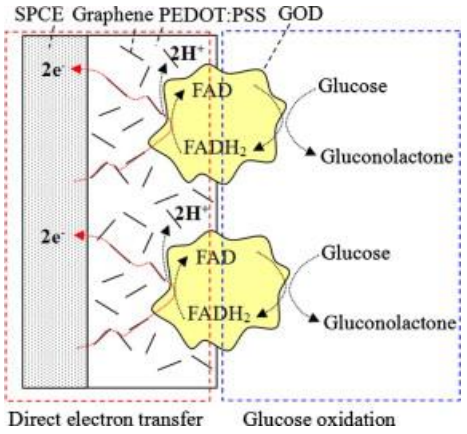
GOD

Enzyme



Glucose biosens

- ✓ Highly selective for glucose sensor
- ✓ High sensitivity
- ✓ Low detection limit



Journal of Electroanalytical Chemistry, 2013

Aflasense

1.First of its kind to the WORLD!

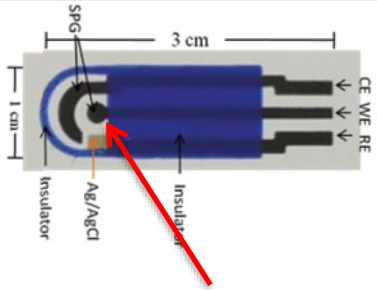
2.Small & Portable

3.Easy, Fast, Accurate

4.Low Cost, High Return

AflaSense: Aflatoxin sensors

Aflasense - A rapid and portable aflatoxin sensor



Graphene-carbon paste

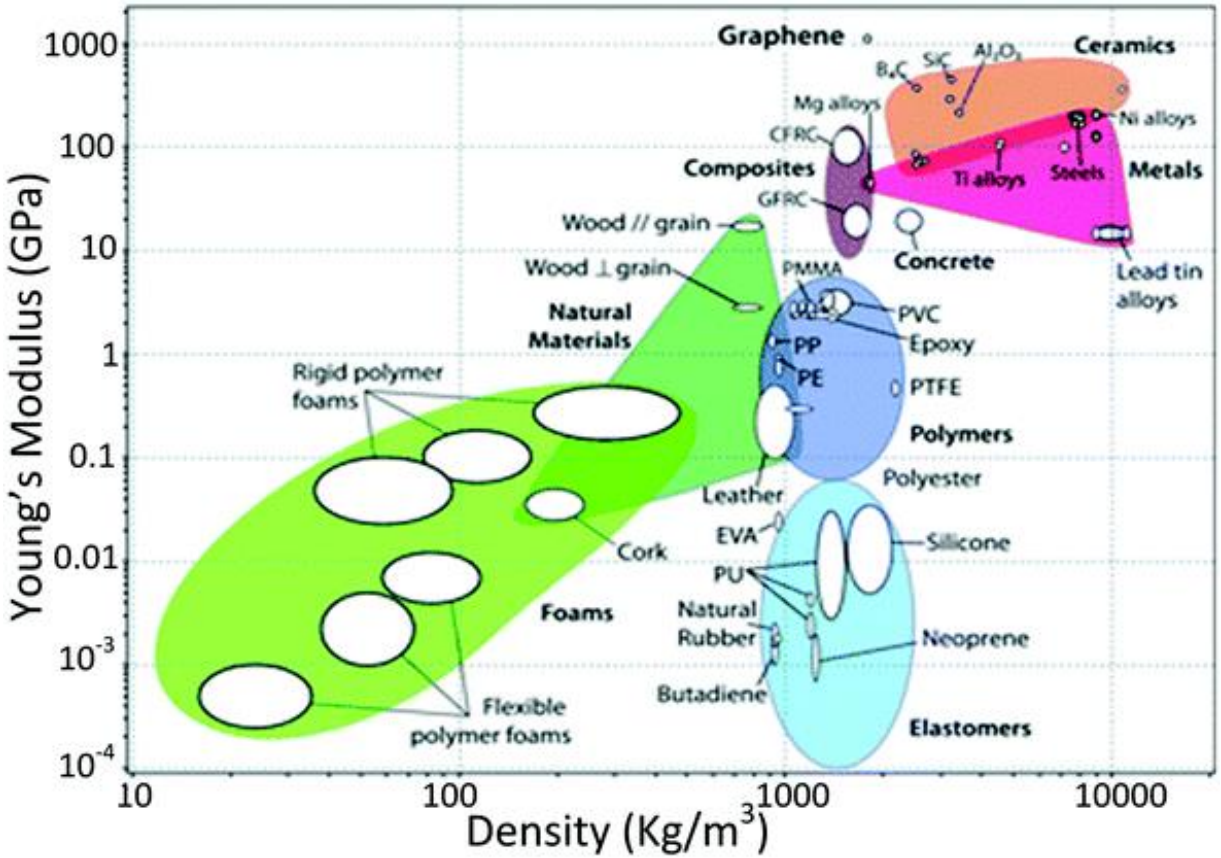
Alfatoxin = 0 to 80 ppb

Analysis process

10-15 min	45 min	10 min	< 1 min	20 sec

Graphene Composite

- *Major developments should be made in the area of chemical derivatives of graphene in order to control electrical conductivity and optical properties of the final products*
- *Incorporation of well-dispersed graphene-sheets into polymers at low filler content improves the mechanical properties*

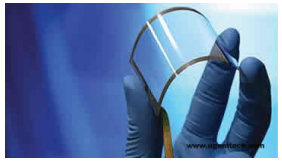


Source: Nanoscale, 2015, 7, 4598

Potential composite improvements with graphene

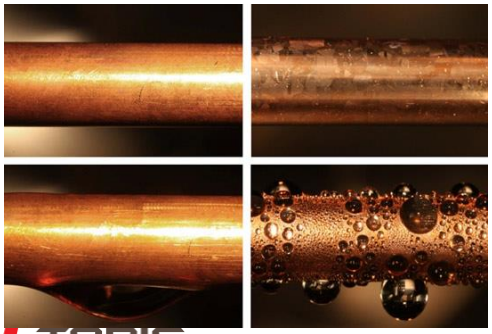
Mechanical properties

Electrical conductivity (σ)



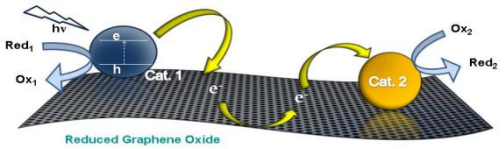
Composite property improvement

Thermal conductivity (κ)

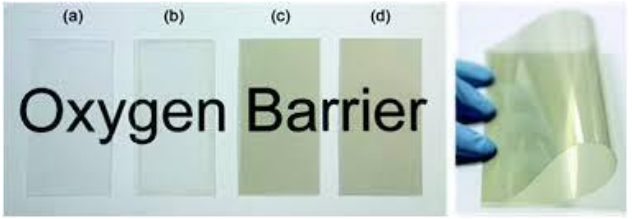


Surface area

Catalysis



Gas permeation



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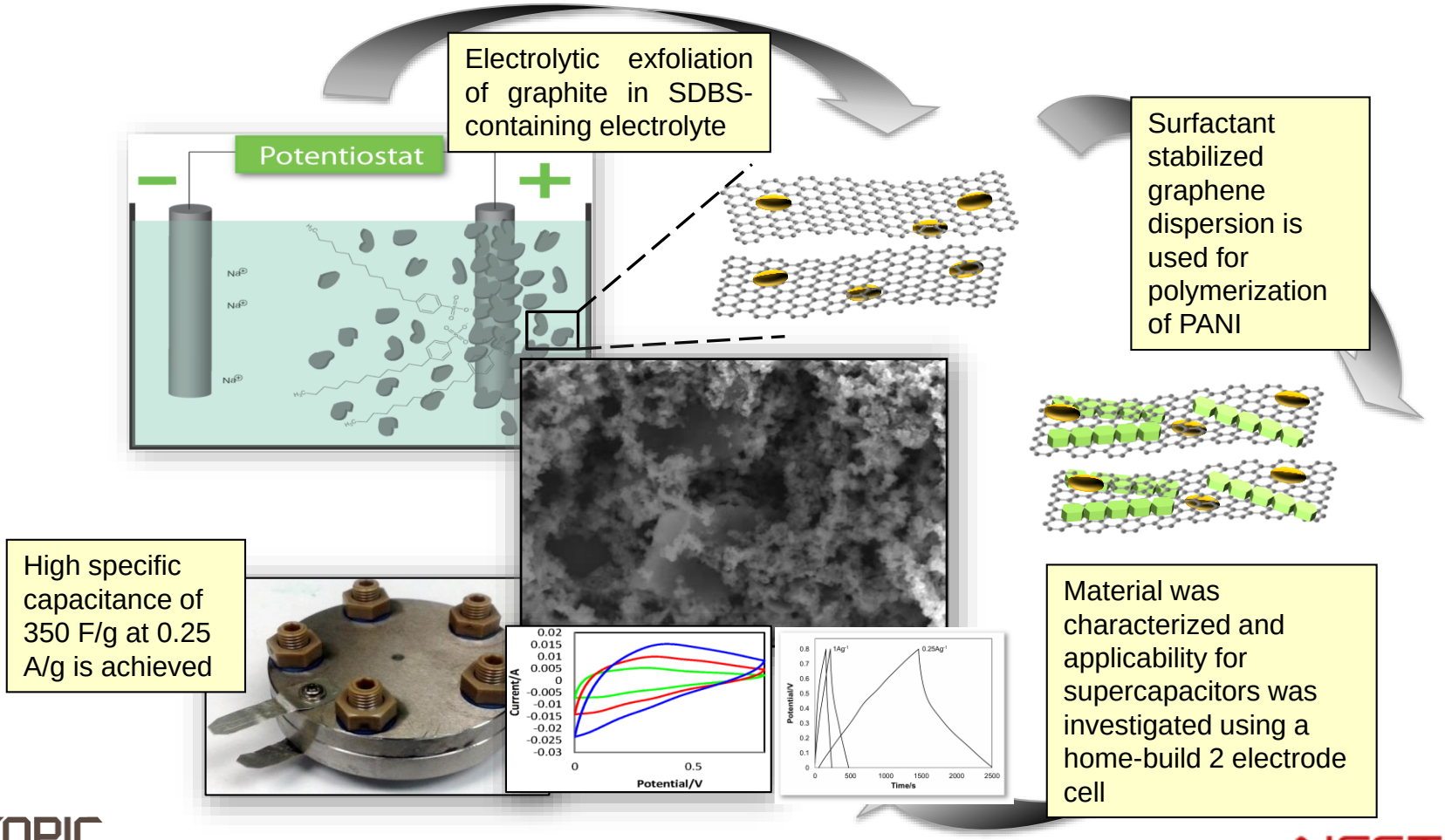
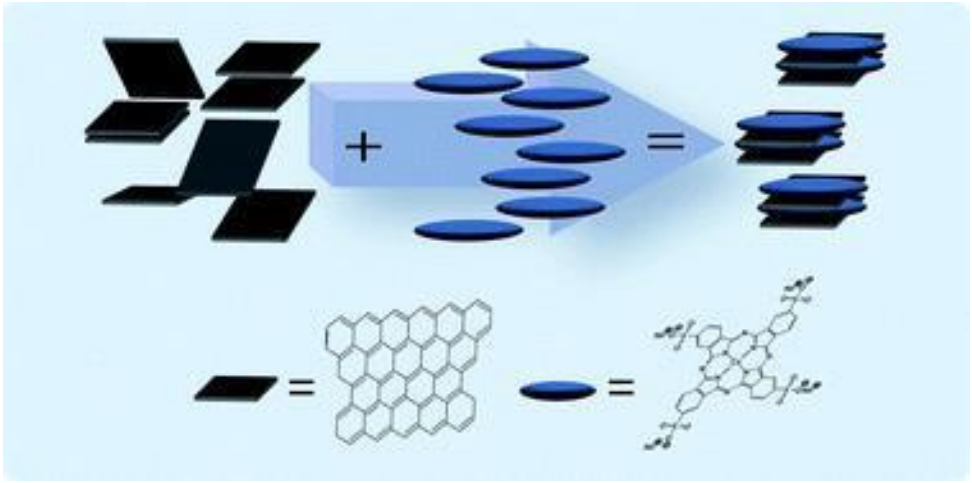
Graphene Composite

Graphene-polyaniline nanocomposite prepared by electrolytic exfoliation for supercapacitor applications



Facile preparation of graphene–metal phthalocyanine hybrid material by electrolytic exfoliation



Journal of Materials Chemistry

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Cite this: *J. Mater. Chem.*, 2012, **22**, 17094

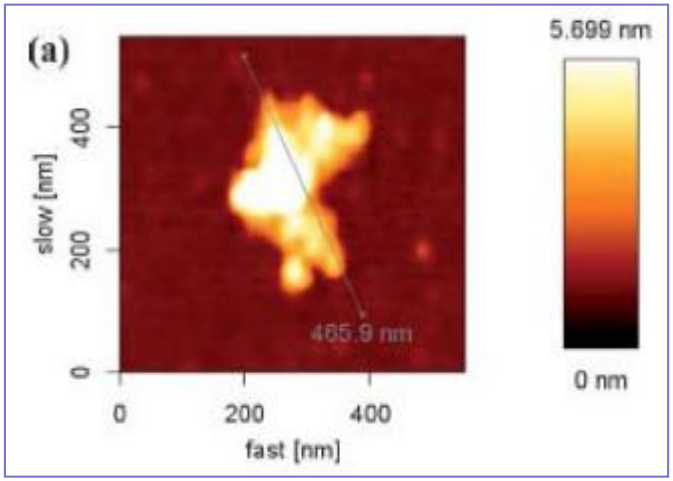
www.rsc.org/materials

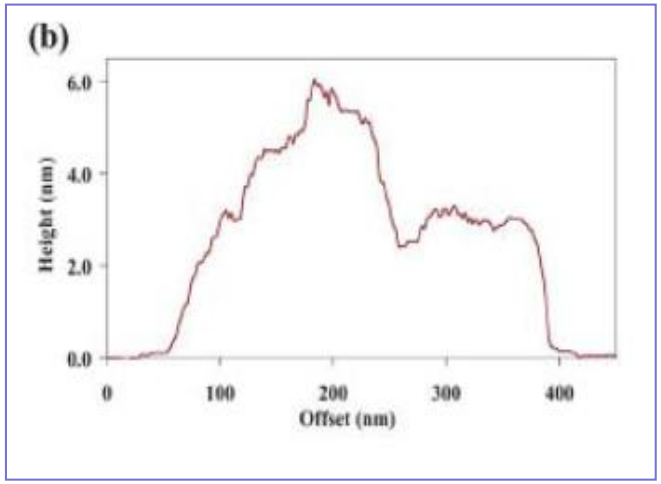
PAPER

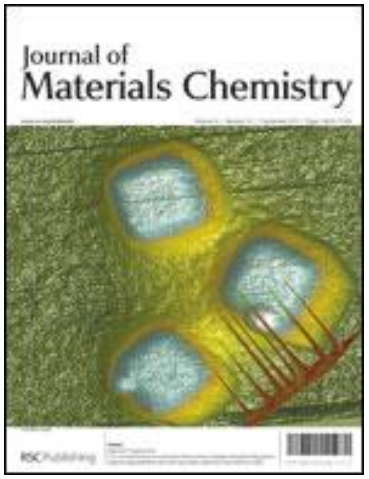
Facile preparation of graphene–metal phthalocyanine hybrid material by electrolytic exfoliation

Johannes Philipp Mensing,[ab] Teerakiat Kerdcharoen,[*b] Chakrit Sriprachuabwong,[a] Anurat Wisitsoraat,[a] Ditsayut Phokharatkul,[a] Tanom Lomas[a] and Adisorn Tuantranont[*a]

Graphene-metal phthalocyanine prepared by electrolytic exfoliation in TSCuPc

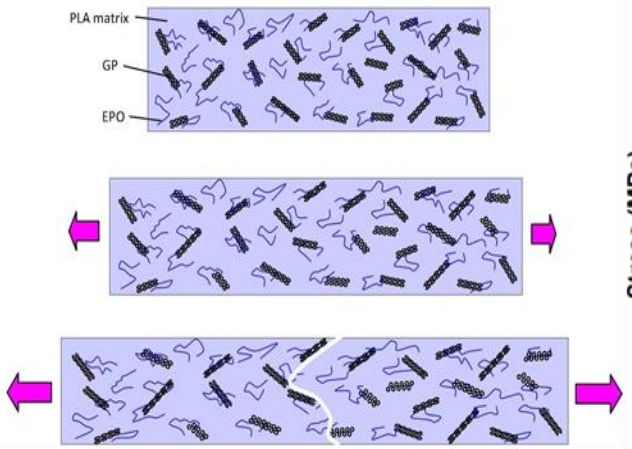




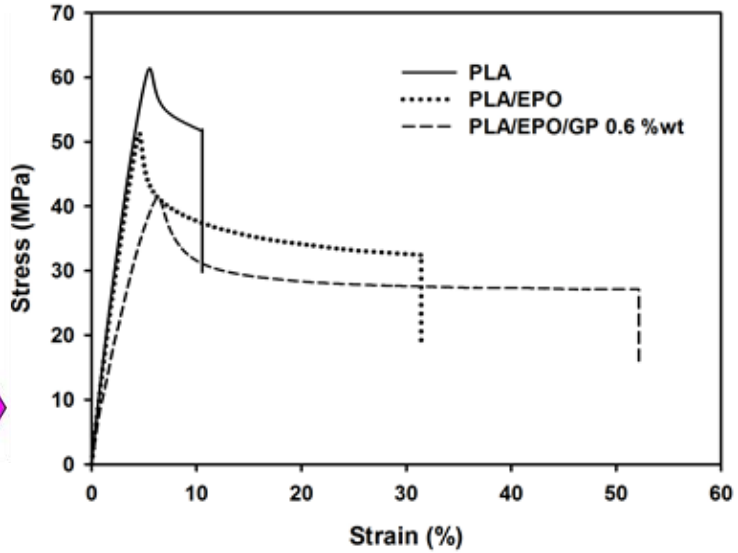


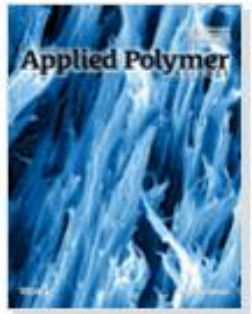
AFM image (a) and height profile (b) of a hybrid particle.

Electrolytically-Exfoliated Graphene-Polylactide based Bioplastic with High Elastic Performance



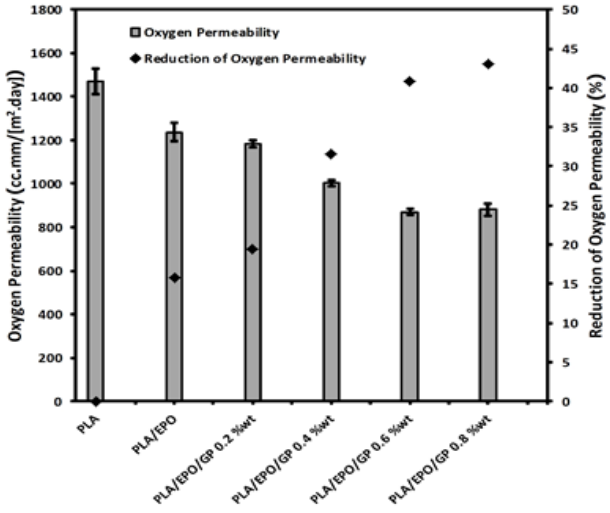
Physical representations of graphene/PLA

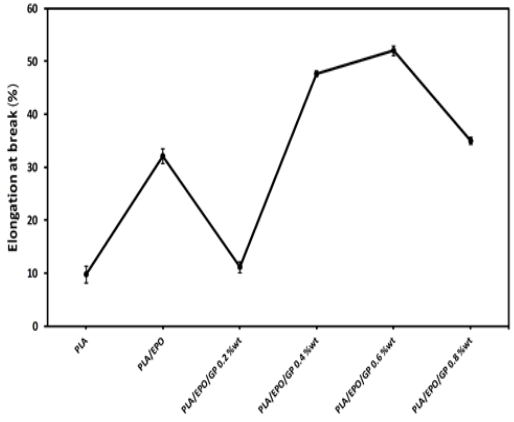




Volume 132, Issue 6
February 10, 2015

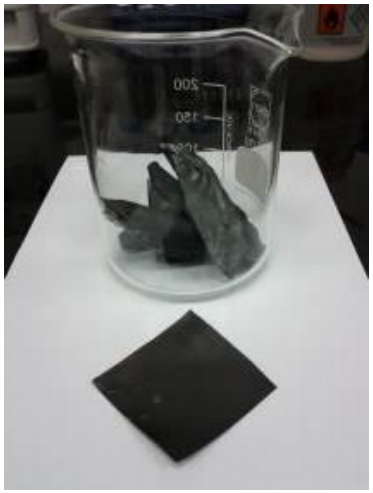
Graphene/PLA tensile test specimen

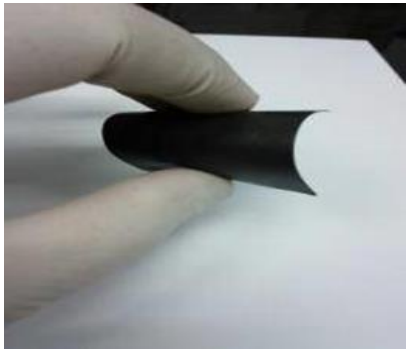


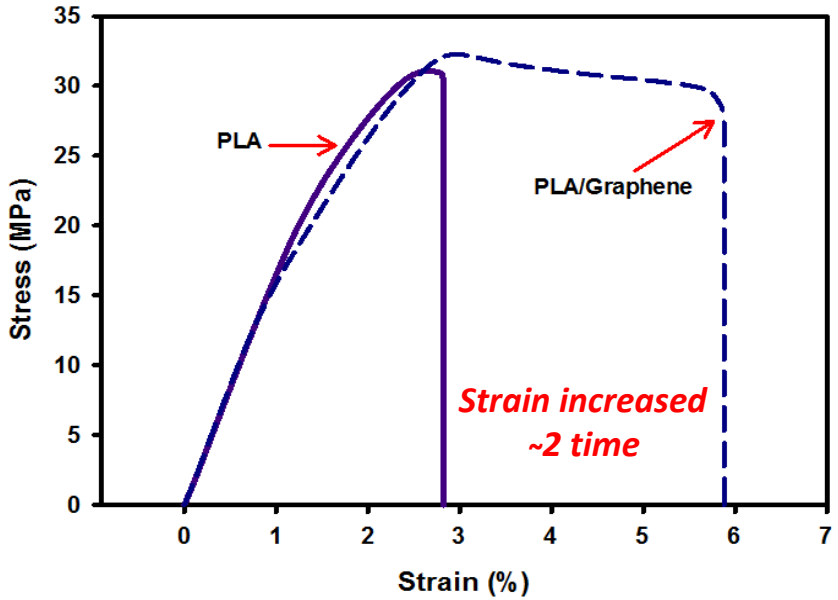


3D Printable Graphene/Polylactide composite for Commercial 3D Printer

Graphene/PLA composite masterbatch

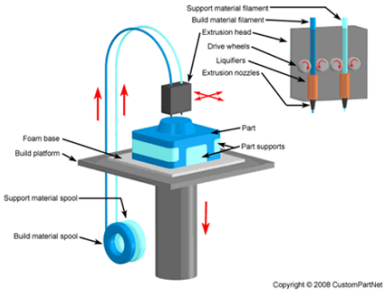






Stress-Strain Curve

Fused deposition modelling (FDM)



Advantages
- Variety of materials
- Machine can be easily set up and used in an office environment
- Cheap

Disadvantages
- Model need support
- Poor strength, vertical direction
- Process is slow on part with large mass
- Poor surface

Graphene/PLA

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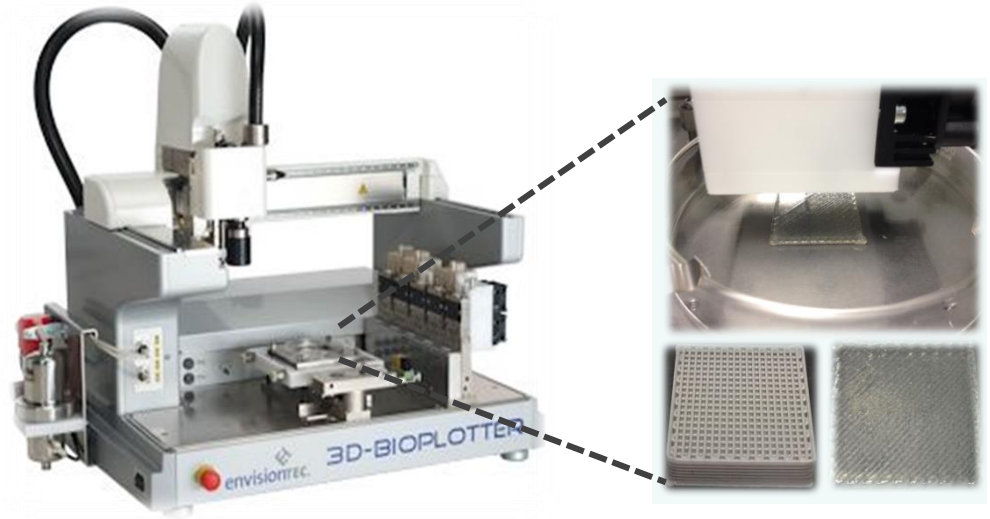
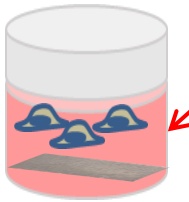
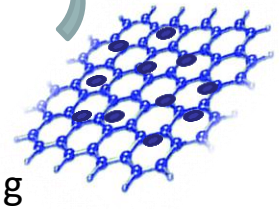
3D-printed graphene electrode cell culture



1.Drug screening



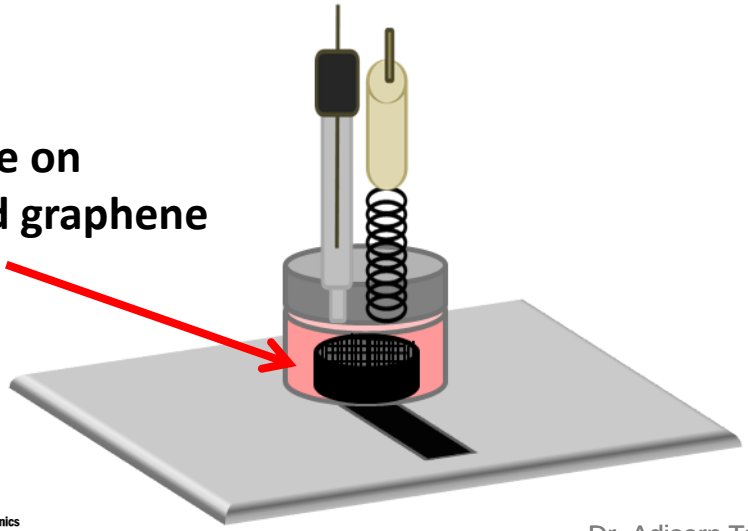
Graphene-hydrogel



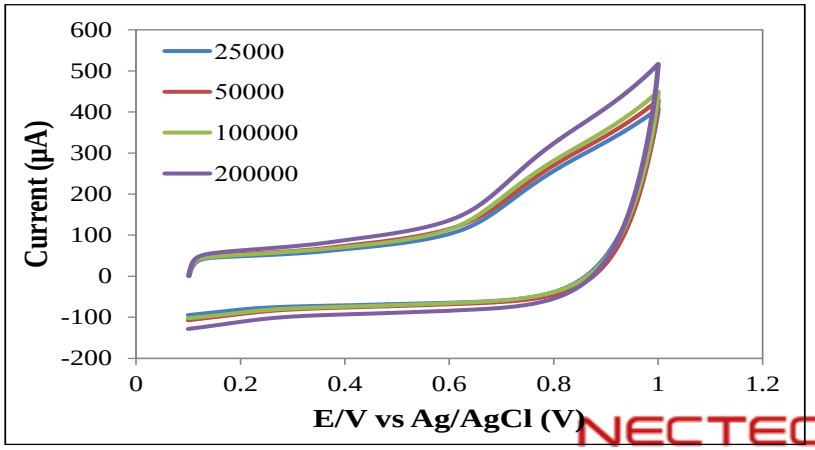
Graphene oxide load anticancer drug

2.Toxicity/Drug testing

Cell culture on 3D-printed graphene electrode

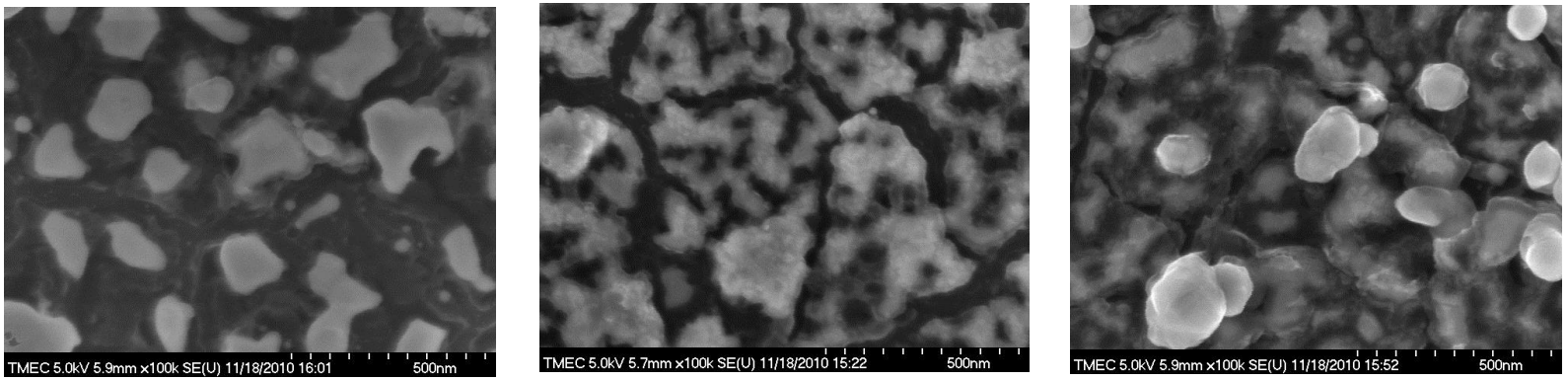


Cellular electrochemical signal

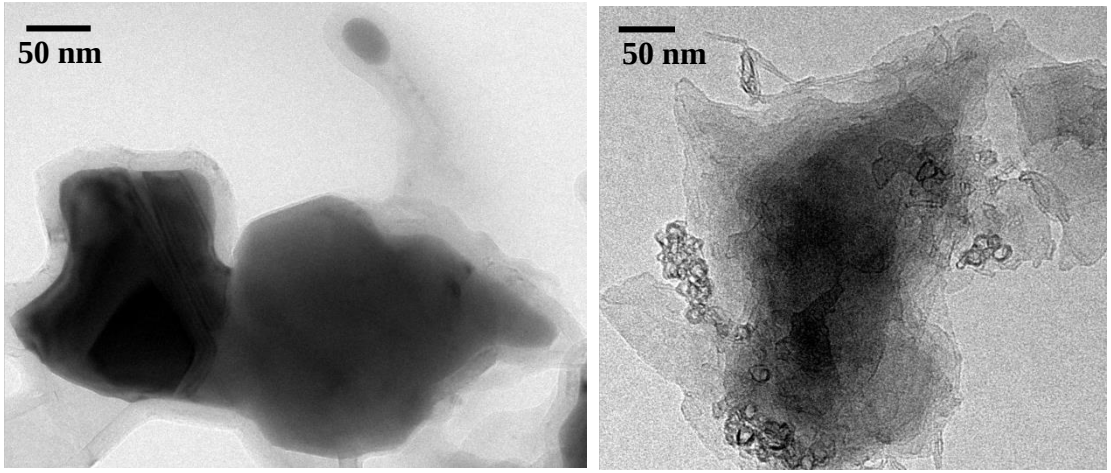


CVD Graphene

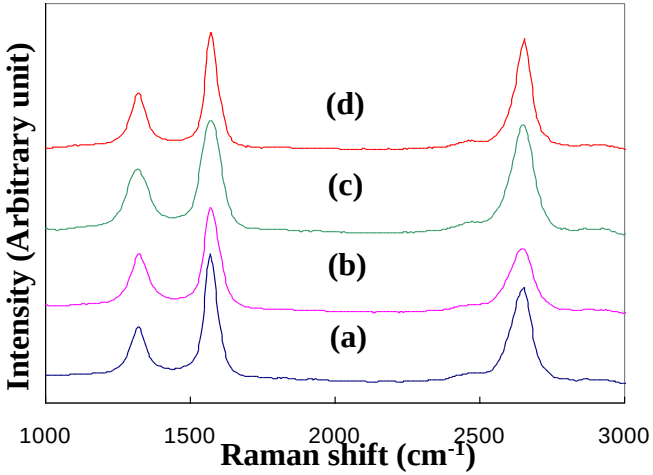
Preliminary development of graphene by CVD



SEM images of CVD graphene thin films synthesized at different CVD conditions



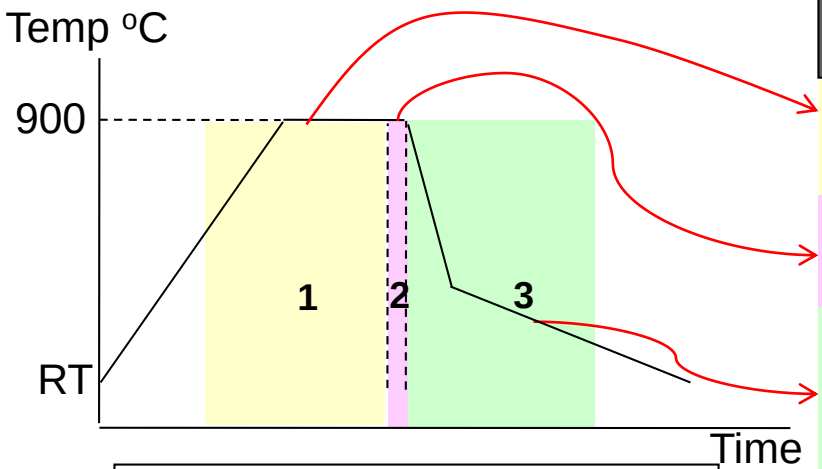
TEM images of CVD graphene thin films synthesized at different CVD conditions



Raman spectra of CVD graphene thin films synthesized at different CVD conditions

3D CVD Graphene

3D Graphene foam and composite



Flow chart for 3D Graphene foam synthesis

➢**1. Nickel foam template cleaning**
Sonicating with acetone for 5 minutes

➢**2. Graphene synthesis on Nickel foam template by CVD**

- **Anneal process**
150 sccm H_2 flow at 900 °C 1 Torr for 30 minutes

- **Graphene layer deposition**
C_2H_2/H_2 gas mixture (2/16 sccm) at 900 °C 0.6 Torr for 1

- **Fast cooling**
150 sccm H_2 flow at 1 Torr with fast cooling rate of >50°C/min

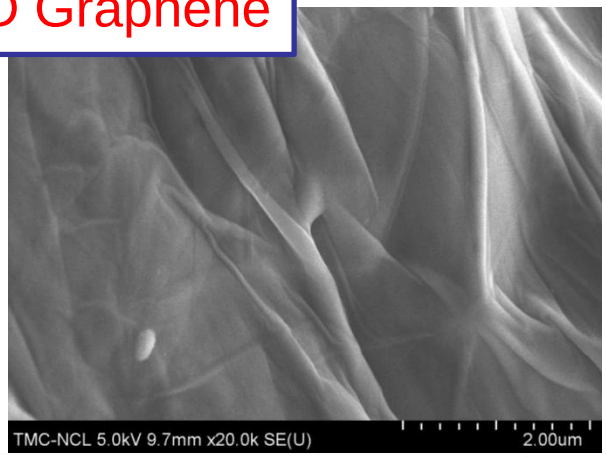
➢**3. PDMS coating**
Nickel foams template covered with graphene were drop-coated with PDMS

➢**4. Nickel Foam template etching**
Etching with 3 M HCl solution at 70°C for 12 hours

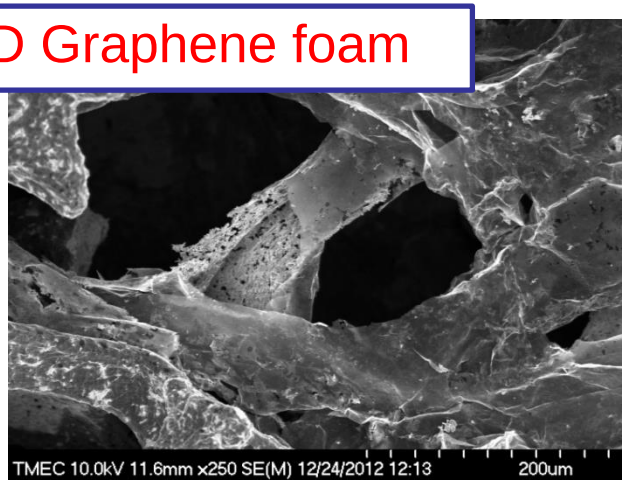
3D CVD Graphene

3D Graphene foam and composite

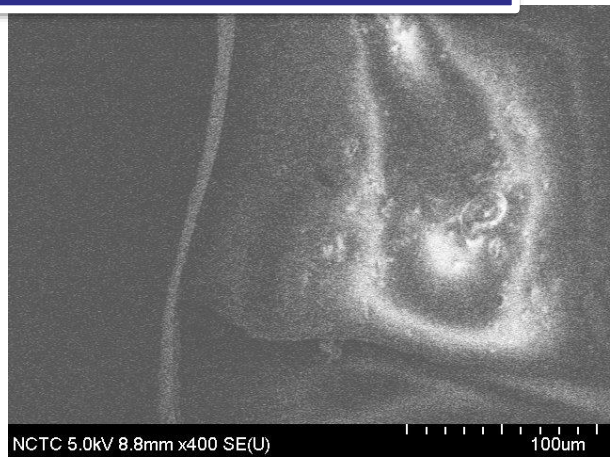
2D Graphene



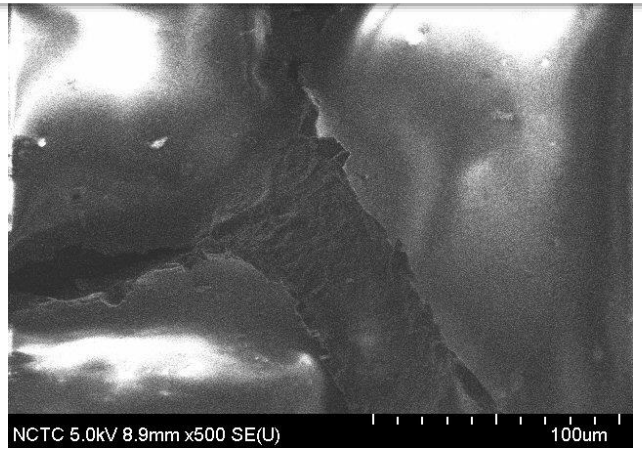
3D Graphene foam



Nickel foam - PDMS



3D Graphene foam - PDMS



SEM images

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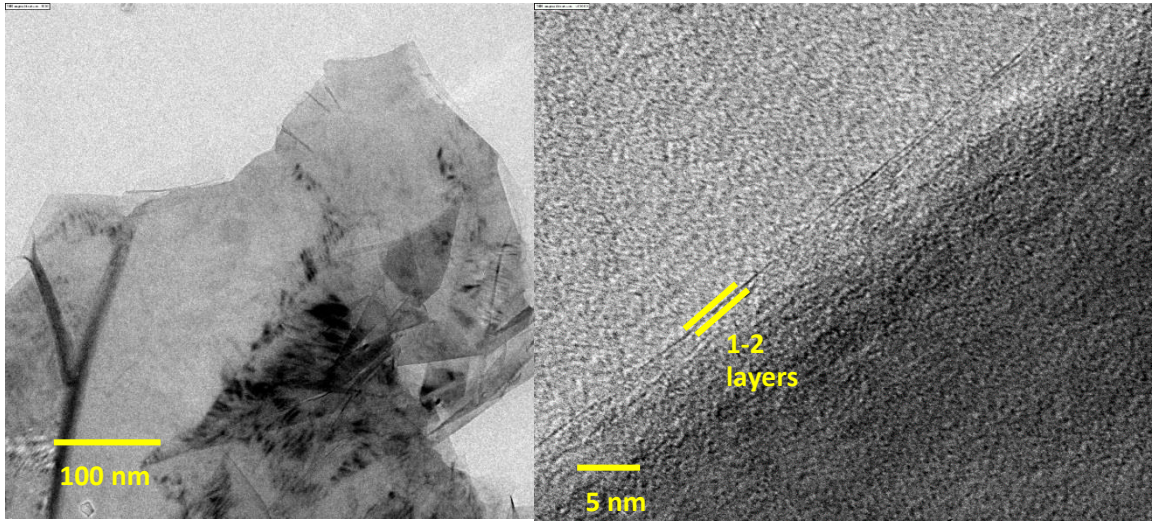
3D Graphene

3D Graphene foam and composite

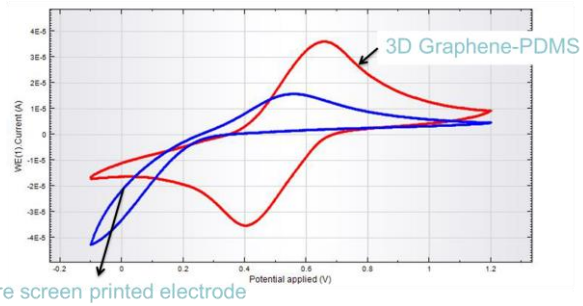
Ni foam

Graphene/Ni foam

Graphene/PDMS foam



3D Graphene foam and composite



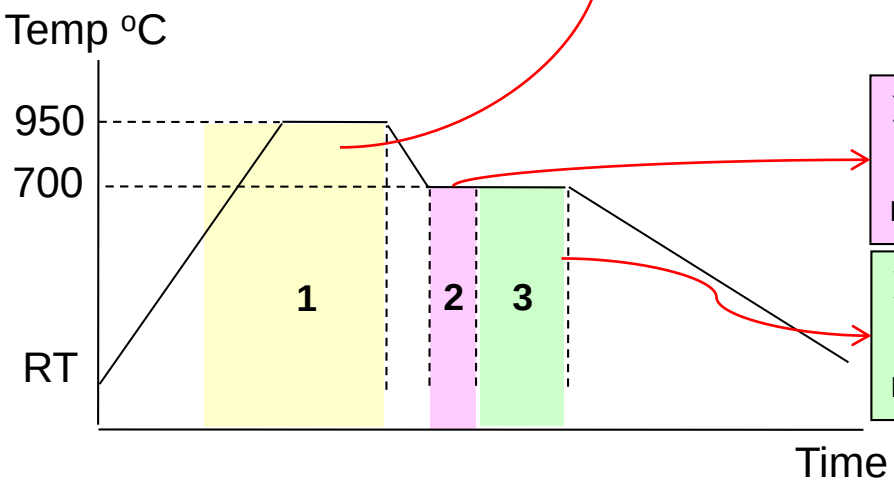
Cyclic voltammograms of 3D Graphene to ferricyanide ([Fe(CN)6]$^{3-}$)

TEM images

Novel 3D Graphene Nanostructure

3D Hollow Graphite Nanotetrapods by Vapor Phase Transport and In-situ Chemical Vapor Deposition/Etching

Typical condition



Flow chart for Graphite Decorated ZnO Nanotetrapods

➢**1. ZnO nanotetrapod synthesis by vapor phase transport**

- ZnO/CNTs mixture powder (1/1 by mole)
- 1000 sccm Ar flow at 950 °C for 40 minutes
- Air exposure at 1 atm for 1 minute

➢**2.CVD graphitic layer deposition**

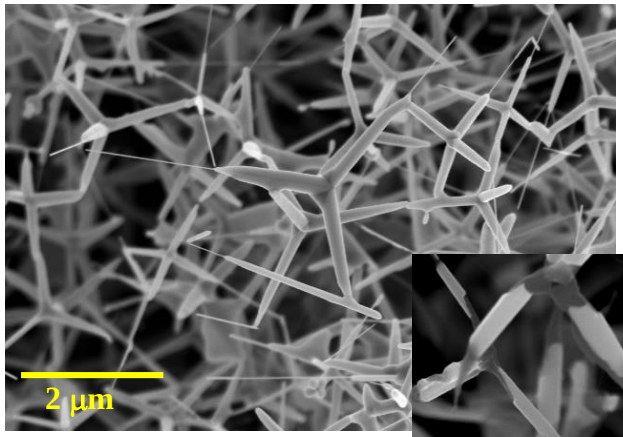
C_2H_2/H_2 gas mixture (3/24 sccm) at 700 °C for 3 minutes

➢**3.Vapor phase ZnO etching**

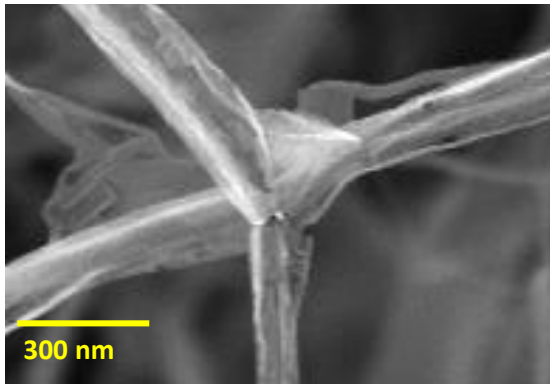
Reduction in H_2 flow of 500 sccm at 700 °C for 30 minutes

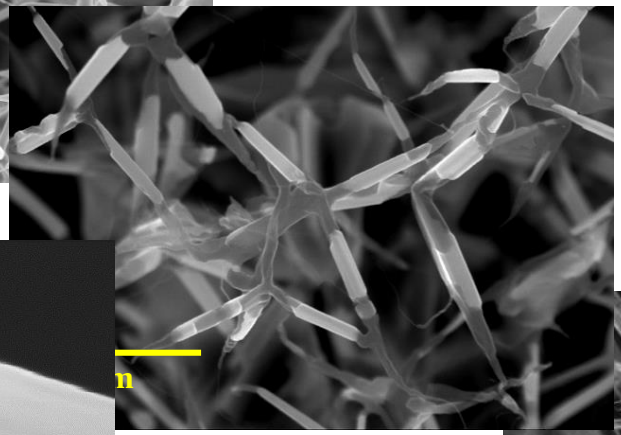
Novel 3D Graphene Nanostructure

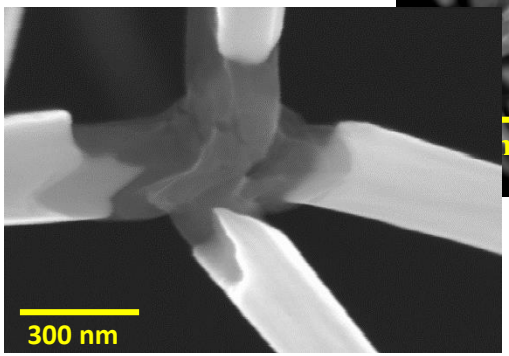
ZnO Nanotetrapods



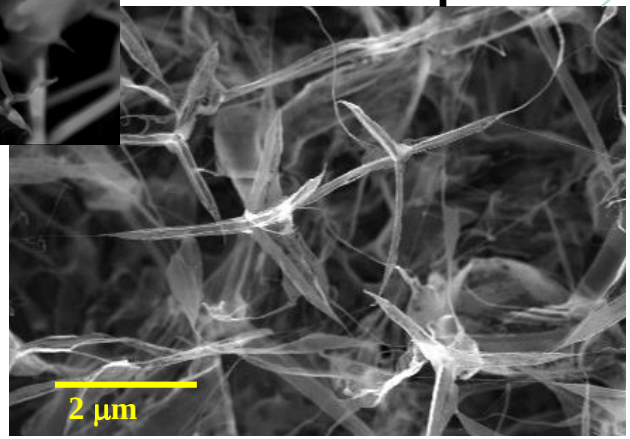
Graphite coated ZnO Nanotetrapods







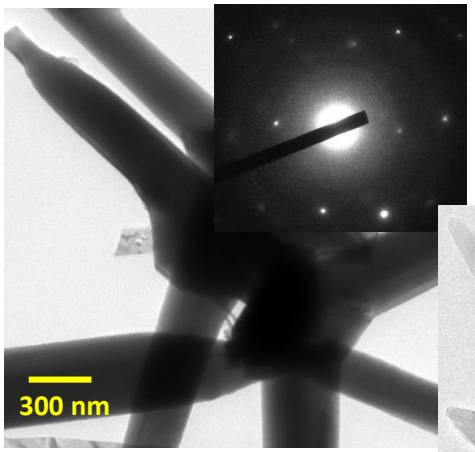
3D Hollow Graphite Nanotetrapods

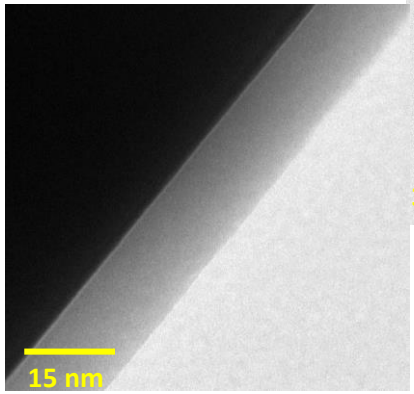


SEM images

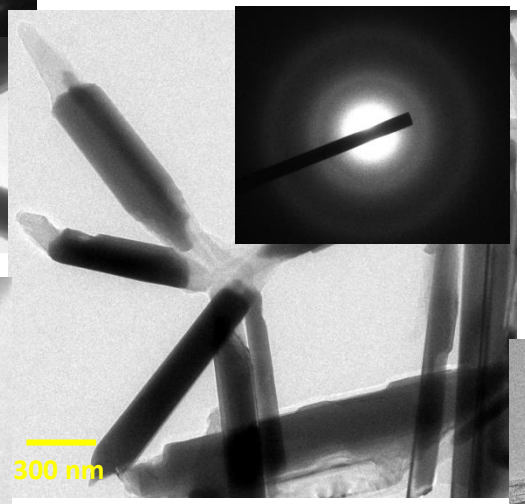
Novel 3D Graphene Nanostructure

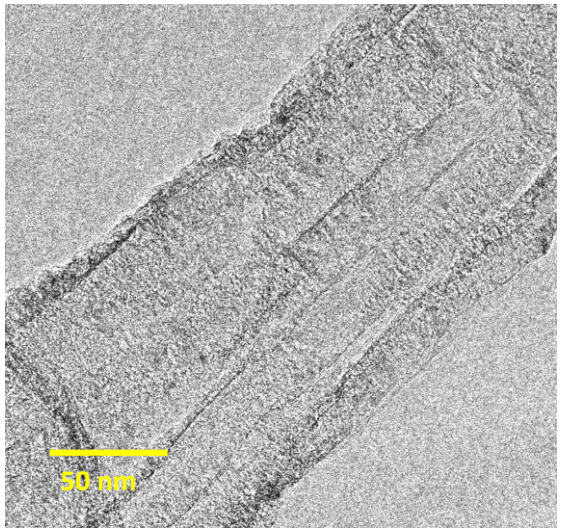
ZnO Nanotetrapods



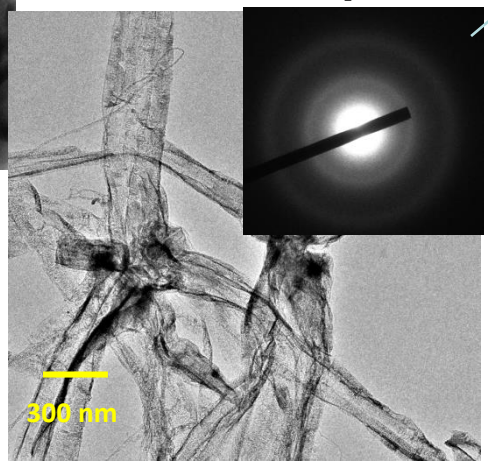


Graphite coated ZnO Nanotetrapods





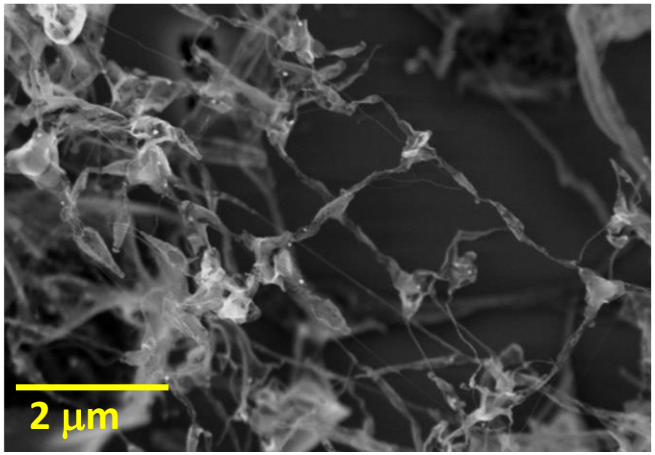
3D Hollow Graphite Nanotetrapods



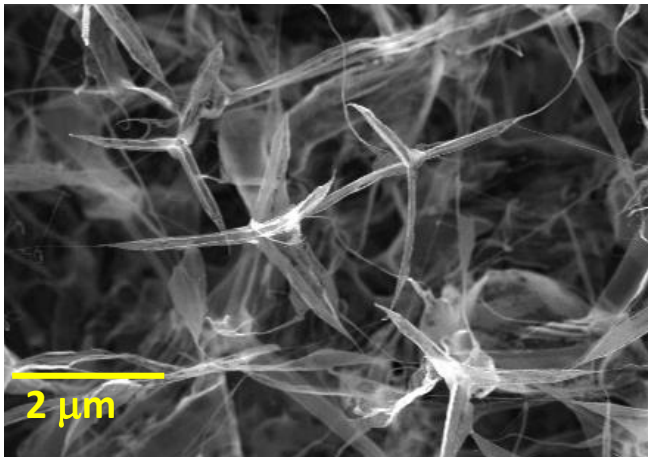
TEM images

Novel 3D Graphene Nanostructure

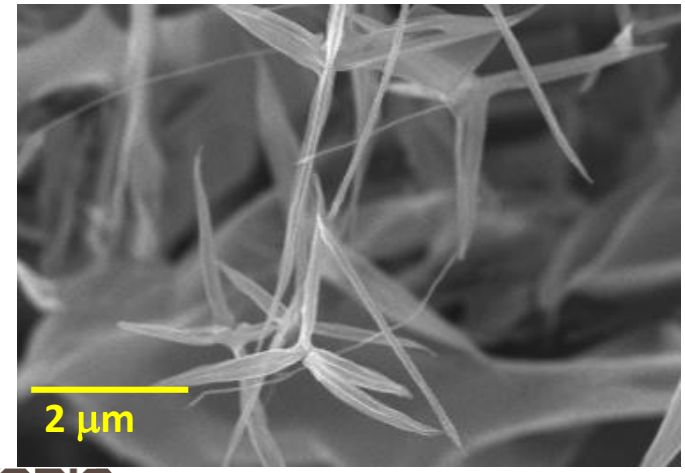
Effect of Carbon synthesis time



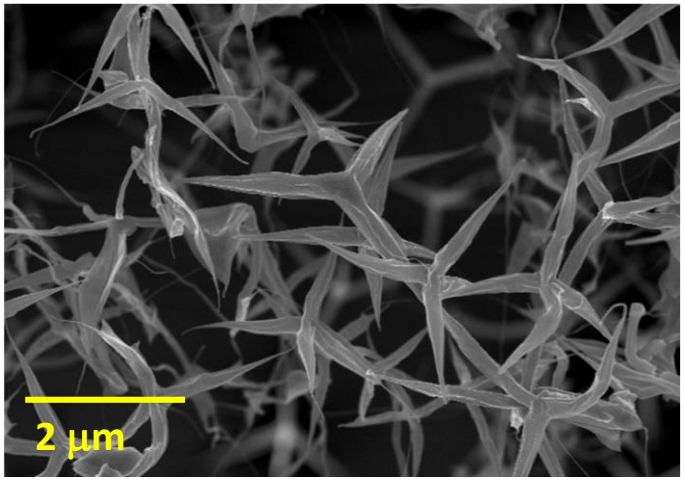
1 min



3 min



5 min

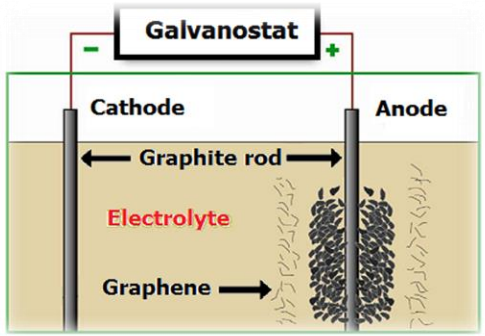


10 min

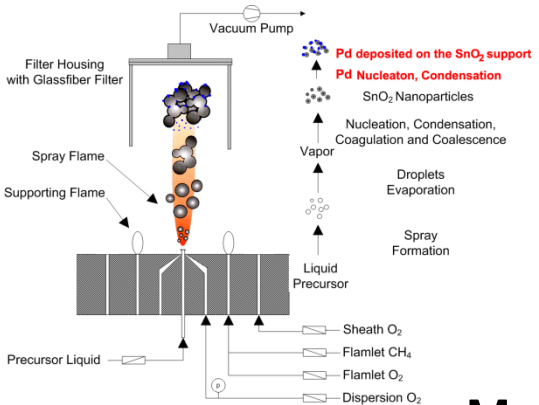
Graphene-composite gas sensors

Graphene-SnO_2 nanocomposite prepared by electrolytic exfoliation and flame spray pyrolysis

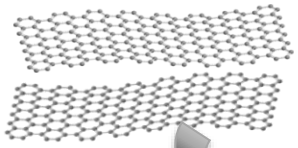
Electrolytic exfoliation



Flame spray pyrolysis

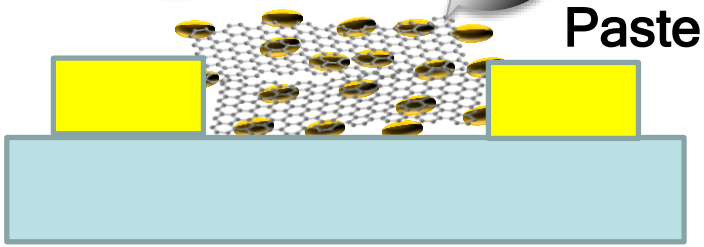


Graphene



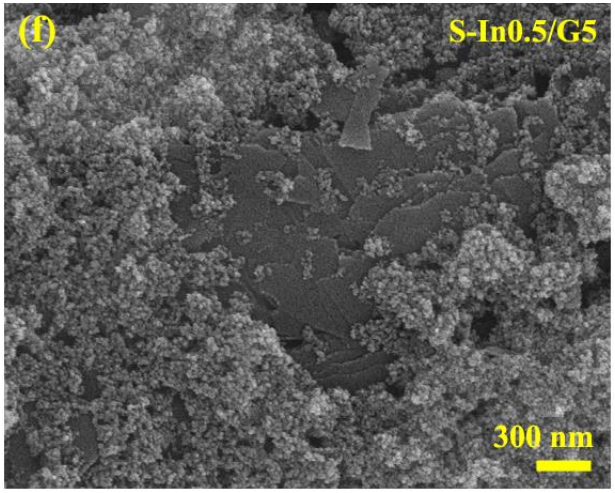
Metal oxide nanoparticles

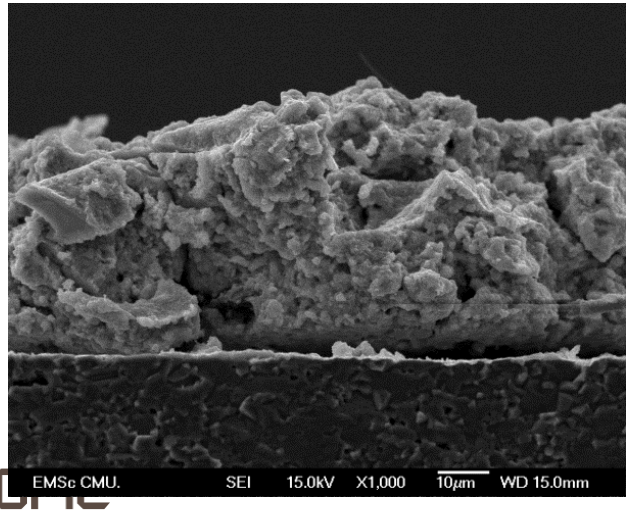
Paste mixing and spin coating

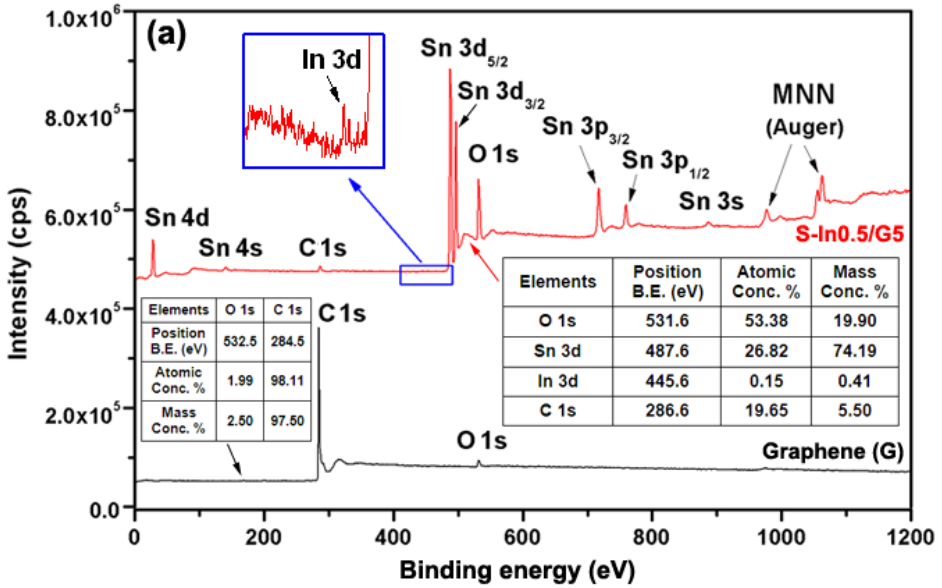


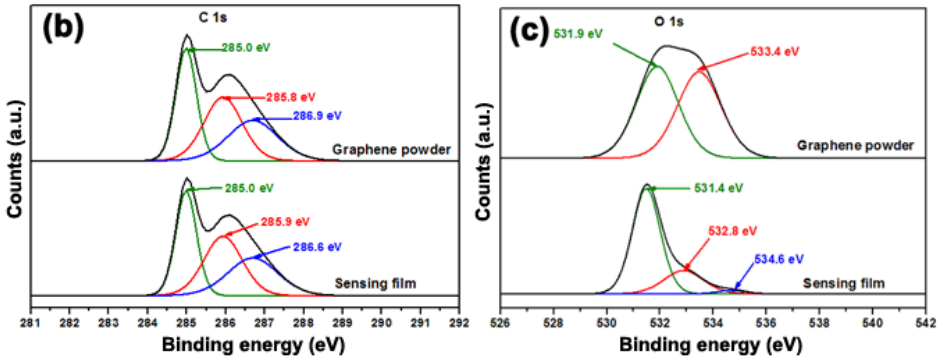
Graphene-composite gas sensors

Graphene-In-doped SnO_2 nanocomposite

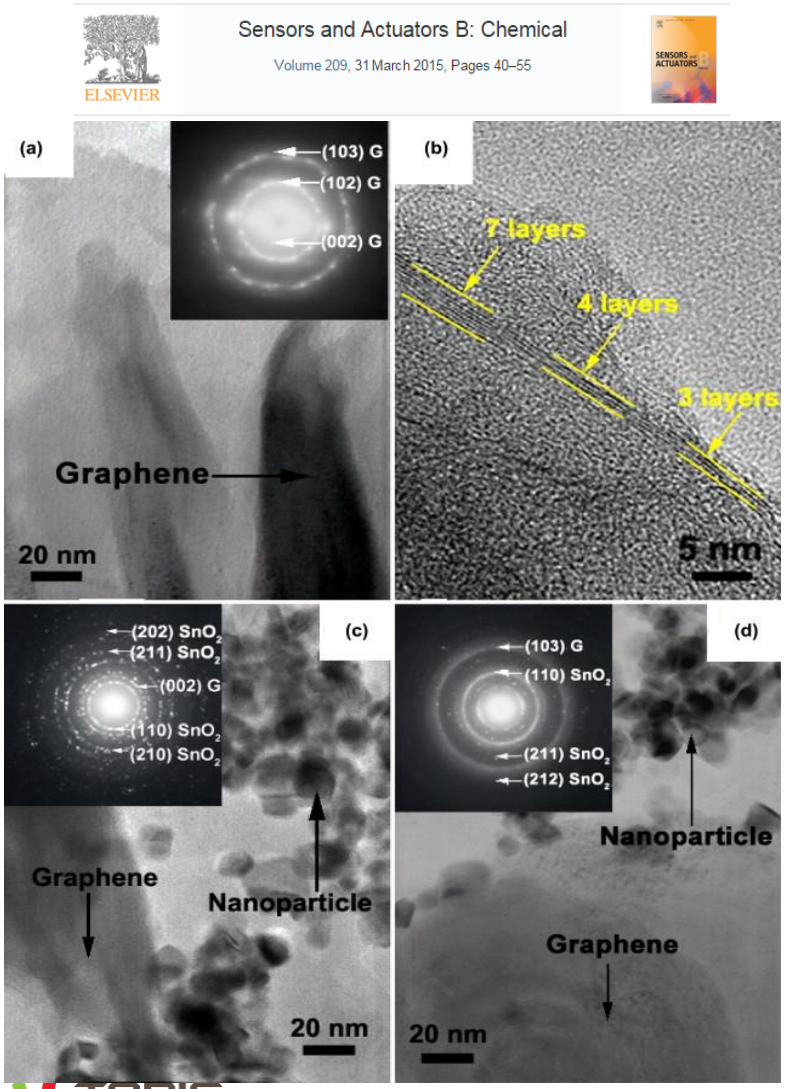


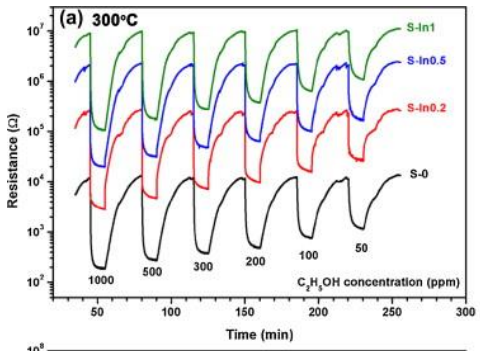


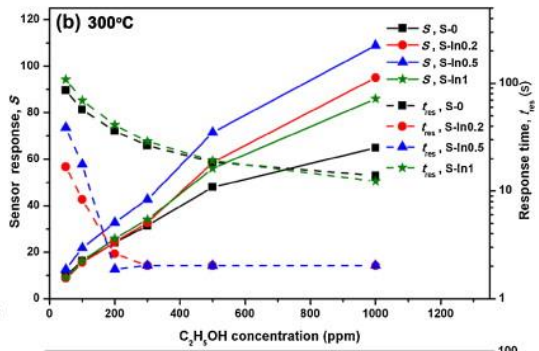


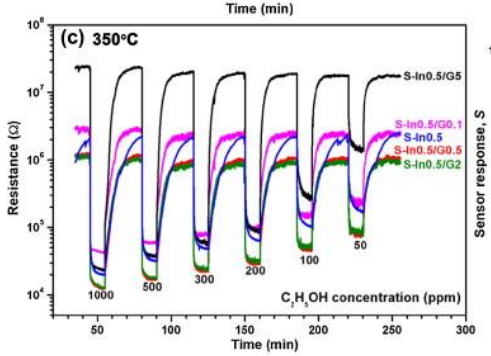


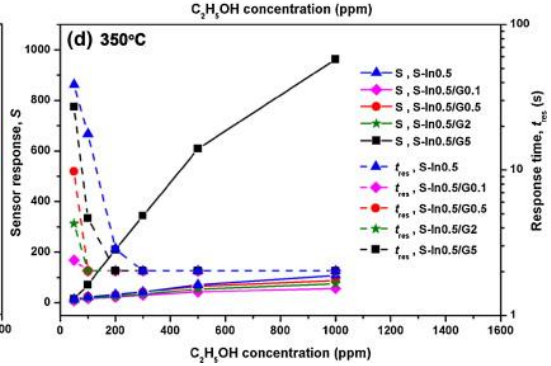
Rapid ethanol sensor based on electrolytically-exfoliated graphene-loaded flame-made In-doped SnO_2composite film

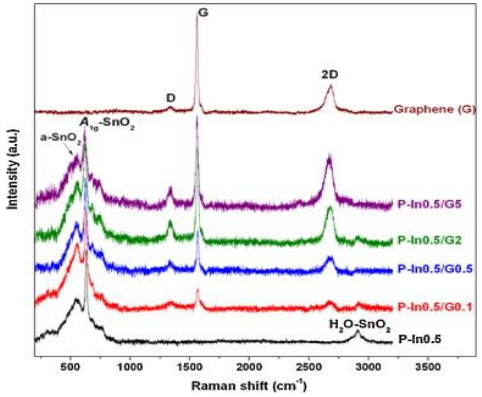


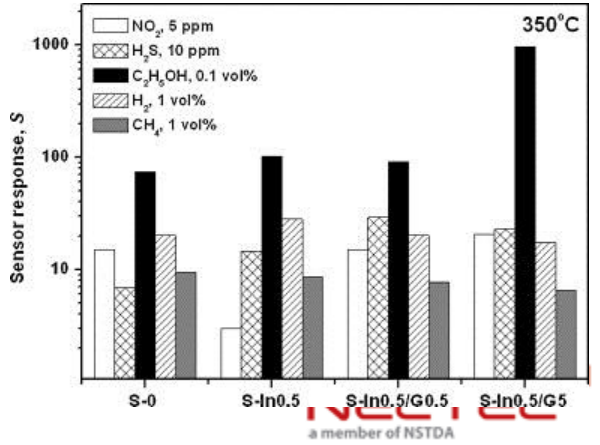








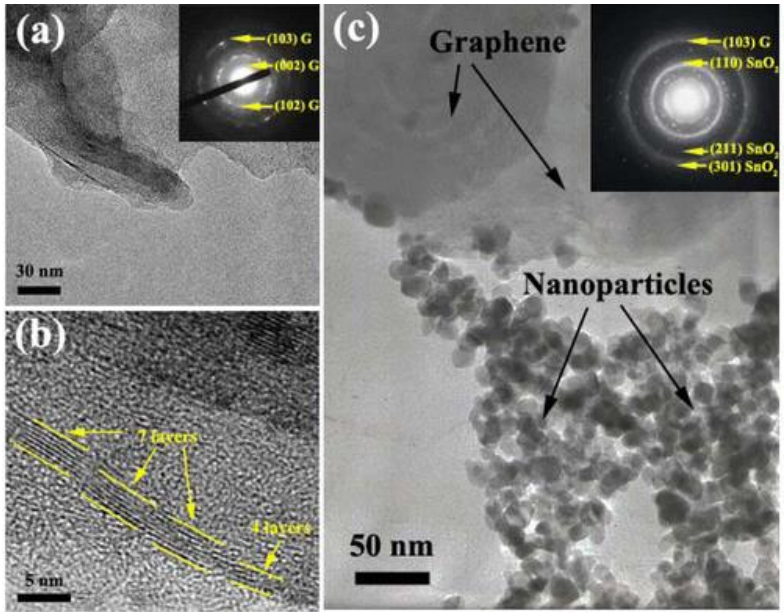


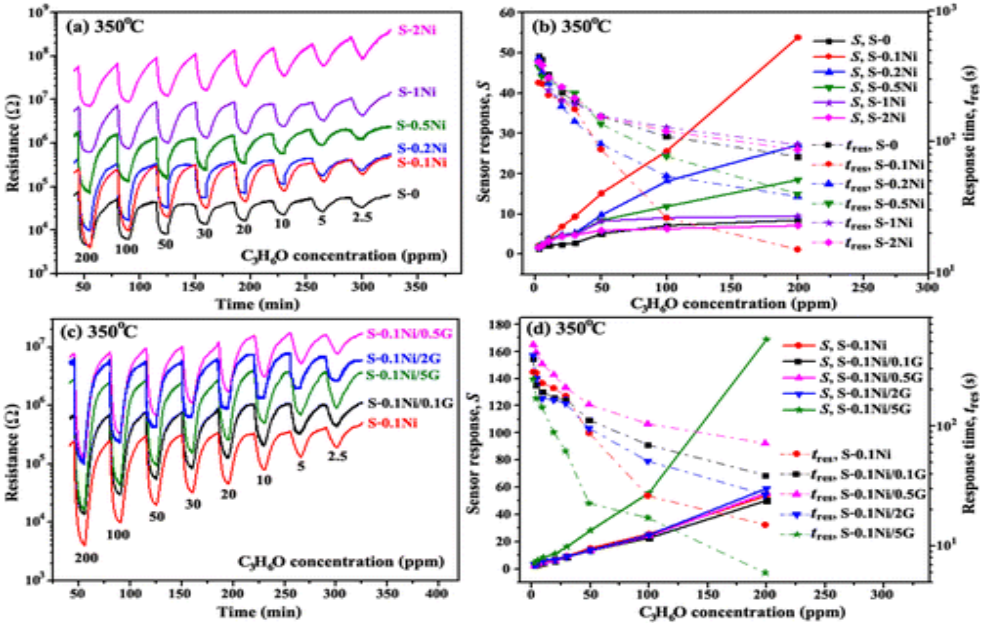


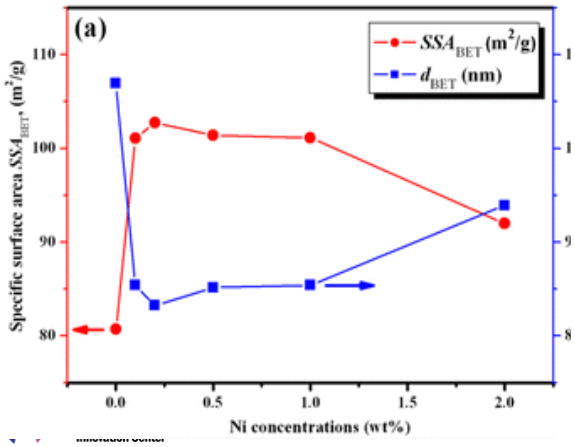
Electrolytically Exfoliated Graphene-Loaded Flame-Made Ni-Doped SnO_2 Composite Film for Acetone Sensing

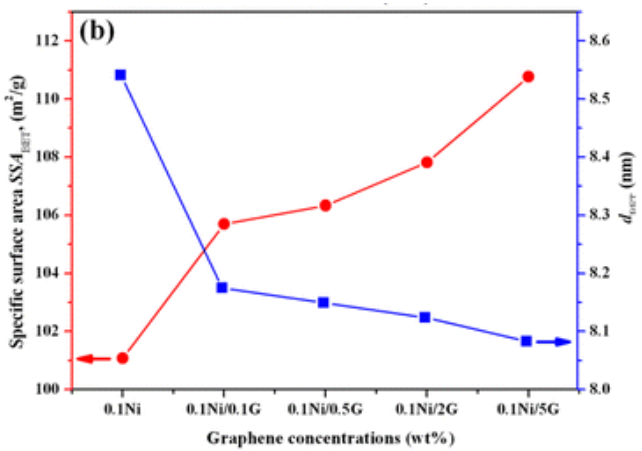
ACS APPLIED MATERIALS & INTERFACES

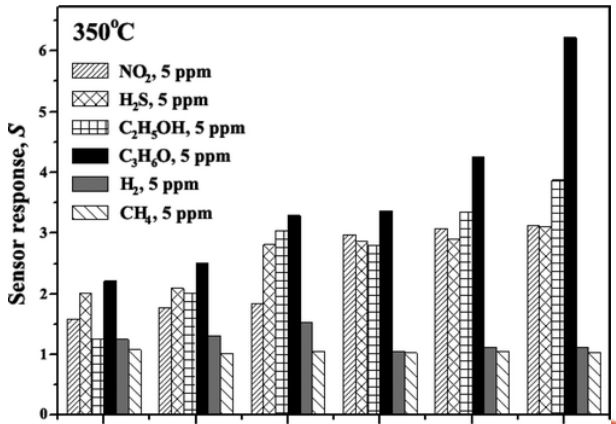
ACS Appl. Mater. Interfaces, 2015, 7 (5), pp 3077–3092











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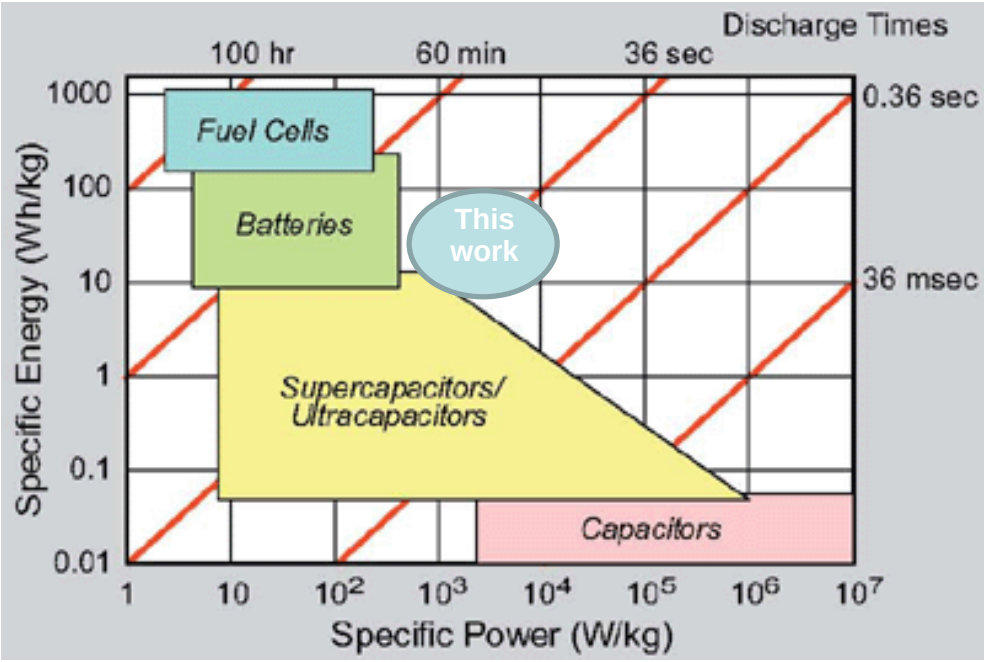
a member of NSTDA

Graphene-based Supercapacitors

- ***Supercapacitors*** also called ***ultracapacitors*** and ***electric double layer capacitors (EDLC)*** are capacitors with capacitance values greater than any other capacitor type available today.

Ultracapacitors vs. Battery and Conventional Capacitors

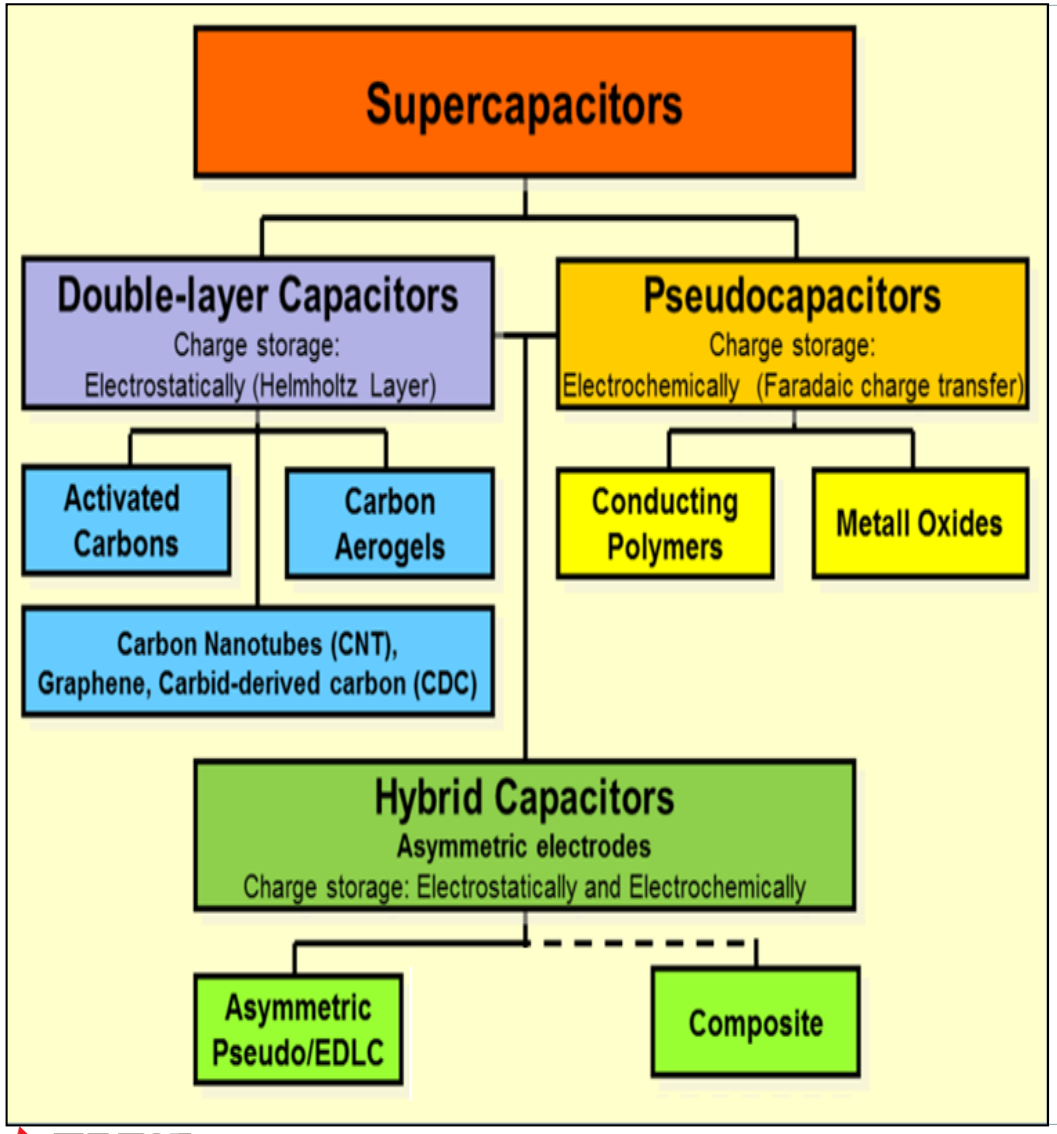
Available Performance	Lead Acid Battery	Ultracapacitor	Conventional Capacitor
Charge Time	1 to 5 hrs	0.3 to 30 s	10^{-3} to 10^{-6} s
Discharge Time	0.3 to 3 hrs	0.3 to 30 s	10^{-3} to 10^{-6} s
Energy (Wh/kg)	10 to 100	1 to 10	< 0.1
Cycle Life	1,000	>500,000	>500,000
Specific Power (W/kg)	<1000	<10,000	<100,000
Charge/discharge efficiency	0.7 to 0.85	0.85 to 0.98	>0.95
Operating Temperature	-20 to 100 C	-40 to 65 C	-20 to 65 C



Benefits of Supercapacitors

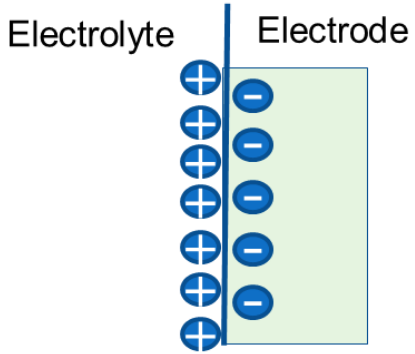
- High Power Density
- Very Fast Charge/Discharge Time
- Long Cycle Life (1,000x > Battery)
- High Reliability and Low Maintenance
- Wide Working Temperature

Type of supercapacitors



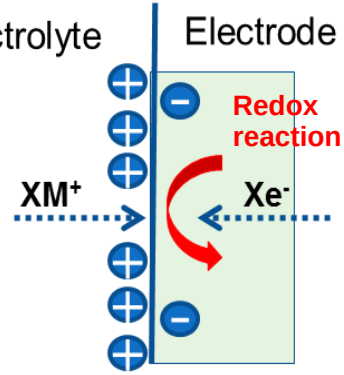
Electric double-layer capacitance (EDLC)

- Physical charge storage



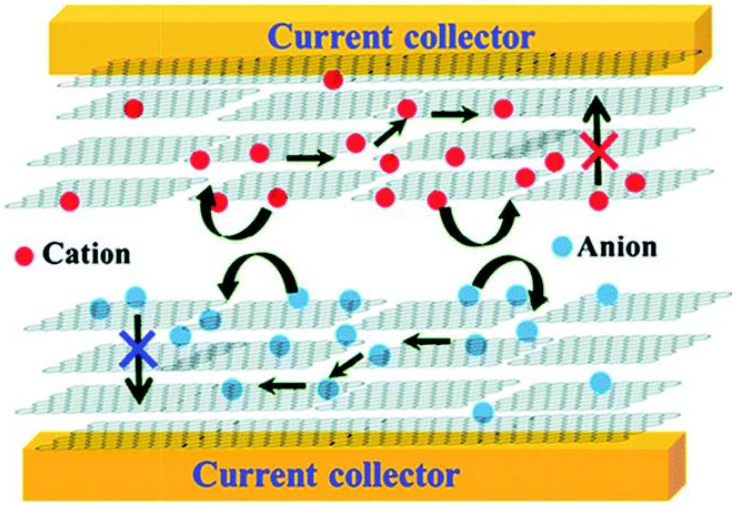
Pseudocapacitance

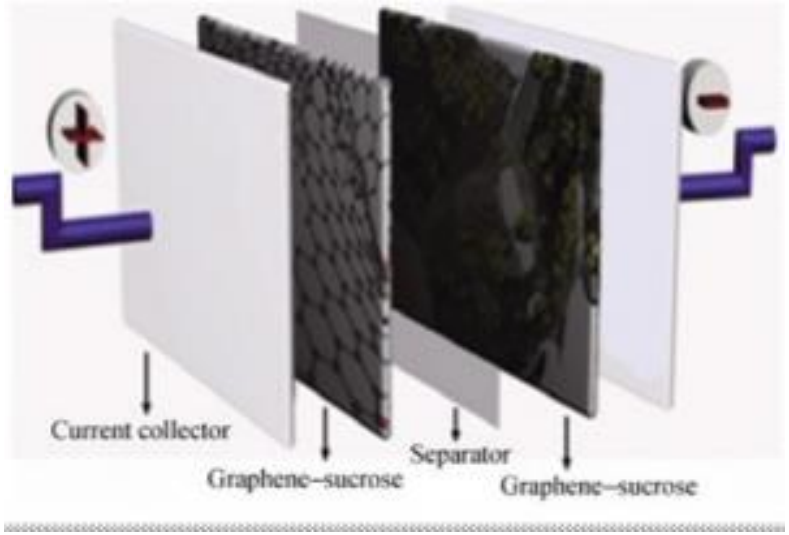
- Charge transfer through surface redox reactions



Advantage of graphene for supercapacitor

- ✓ High electrical conductivity *(R~ 1.5 x10^{-6} ohm-cm)*
- ✓ Large surface area *(about 2630 m^2/g)*
  - Activated carbon 1000 m^2/g
  - SWCNT 1300 m^2/g
  - Carbon aerogel 400-1200 m^2/g
- ✓ Graphene is based on graphite *(low cost and scalable)*

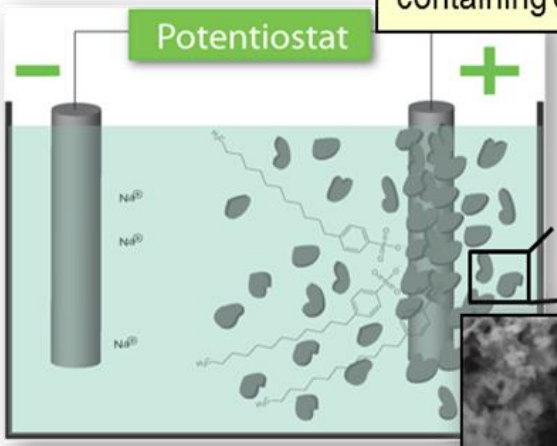


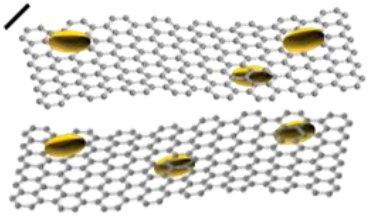


Novel surfactant-stabilized graphene-polyaniline composite nanofiber for supercapacitor applications

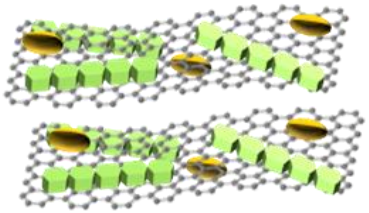
Composites Part B: Engineering
Volume 77, August 2015, Pages 93–99

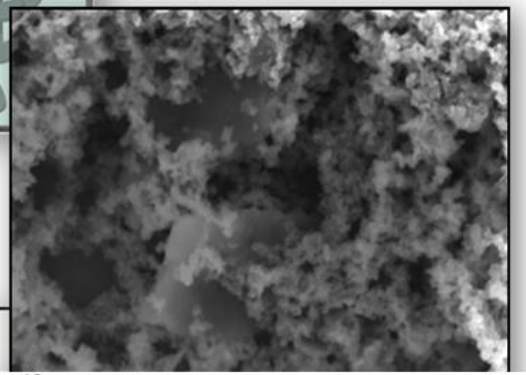
Electrolytic exfoliation of graphite in SDBS-containing electrolyte



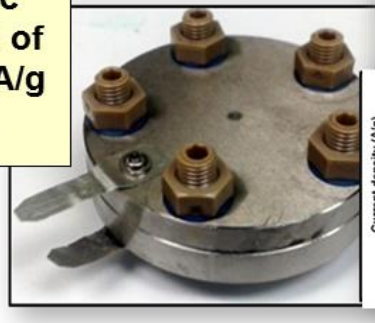


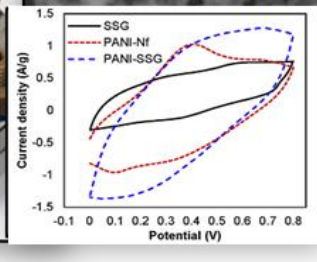
Surfactant stabilized graphene dispersion is used for polymerization of PANI

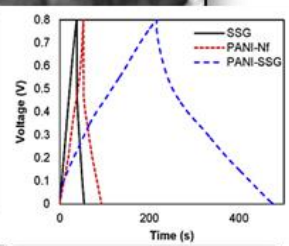




High specific capacitance of 690 F/g at 1A/g is achieved

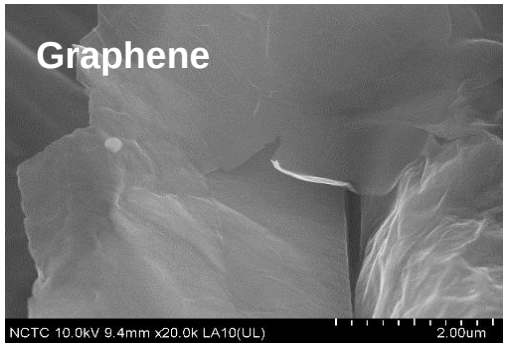


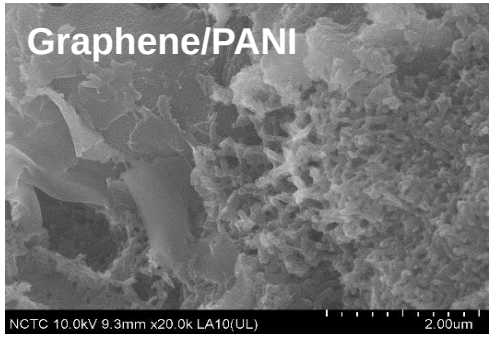


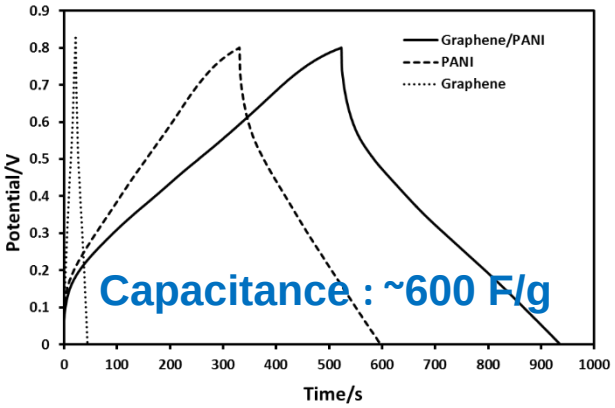


Material was characterized and applicability for supercapacitors was investigated using a home-build 2 electrode cell

Printed Graphene/PANI supercapacitor electrode



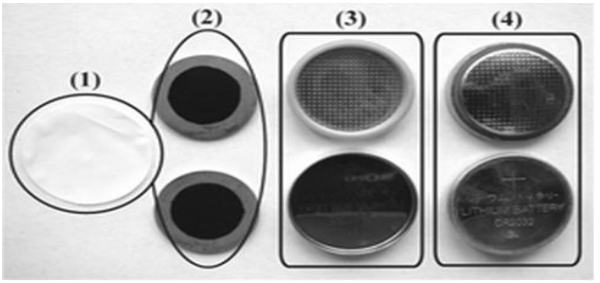


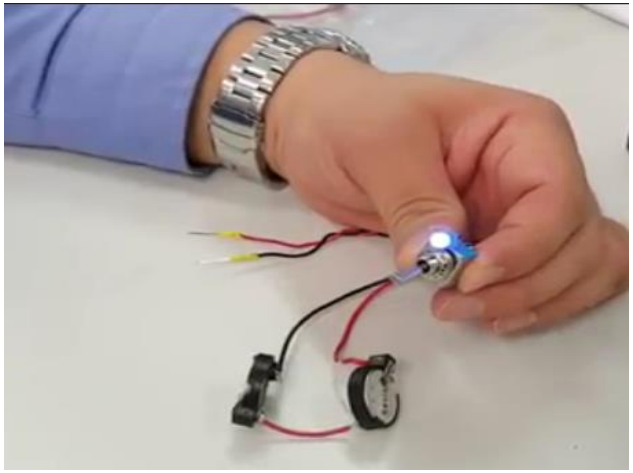


$$C = \frac{I\Delta t}{m\Delta V}$$

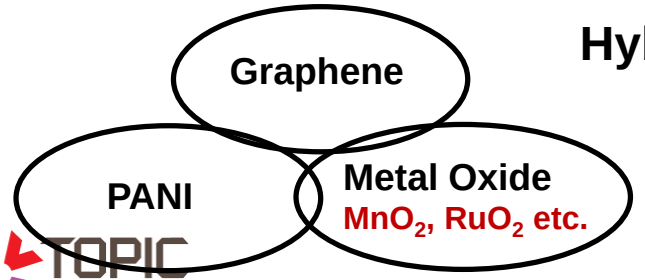
- I : charge-discharge current (A)
- Δt : discharge time (s)
- Δv : potential charge during discharge process (v)
- m : mass of active material (g)

Coil cell preparation





Further work



Hybrid Supercapacitors

- High specific capacitance ≥ 1,000 F/g
- High power density
- High energy density

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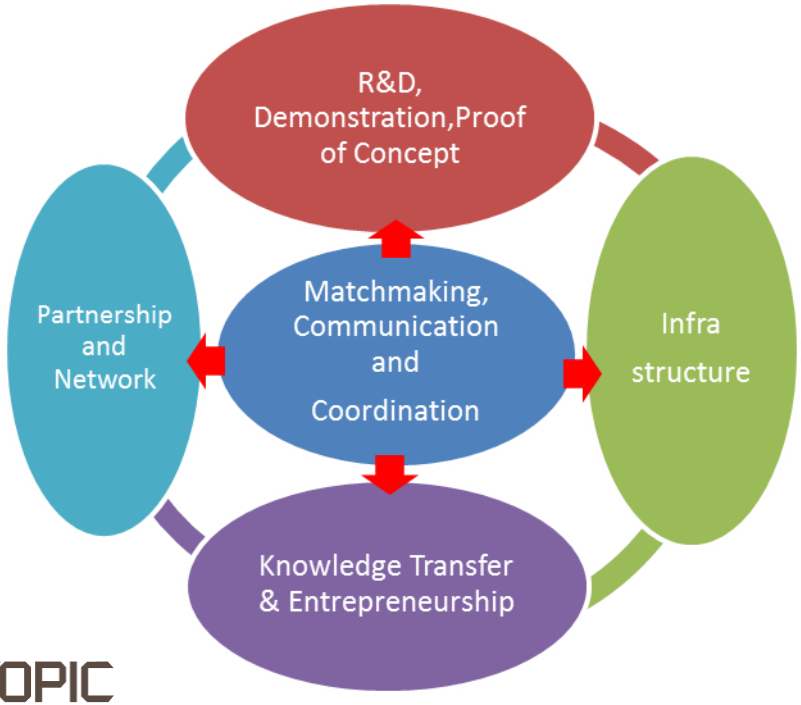
Organic Electronics Association



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BRIA

Mobilis Automata Co., Ltd.

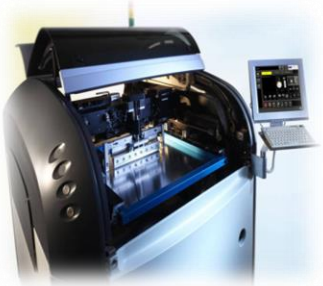
INNOPHENE
GRAPHENE PRINTED ELECTRONICS

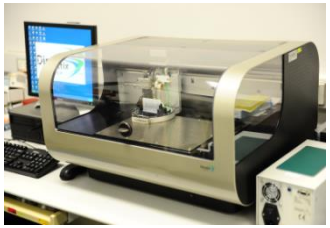
EPC corporation

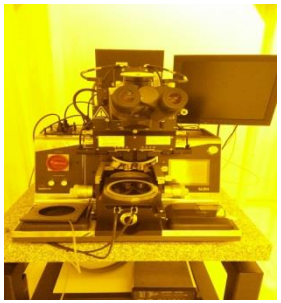
NECTEC
a member of NSTDA

TOPIC's Infrastructure: Material Printing/Deposition

- ✓ **Inkjet printer : DMP-2831, Fujifilm Dimatix**
- ✓ **DEK Automated Screen Printer**
- ✓ **Semi-auto screen printer : MK mini, Minami**
- ✓ **Gravure printer : Labratester, Norbert Schläfli Maschinen**
- ✓ **Flexo printer : F1-UV, IGT Testing system**
- ✓ **Spin coater : WS-650Sz-6NPP/LITE, Laurell**
- ✓ **Chemical Vapor Deposition (CVD) : planarGROW-4E, Planar tec**
- ✓ **Mask aligner : MJB4, SUSS MicroTech**
- ✓ **Plasma system : Pico, Diener electronic …**





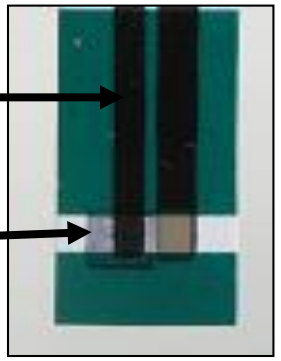


Spin-off Company: Innophene Graphene Conductive Ink for Transparent Electrode and Sensors

Inkjet Printing

Screen printed carbon electrode

Graphene-Polymer



Graphene-polymer dispersed solution was used as an ink for inkjet printing (PCT Patent licensed to Innophene)

The inkjet printed graphene-polymer on carbon electrode

INNOPHENE
GRAPHENE PRINTED ELECTRONICS

www.innophene.com

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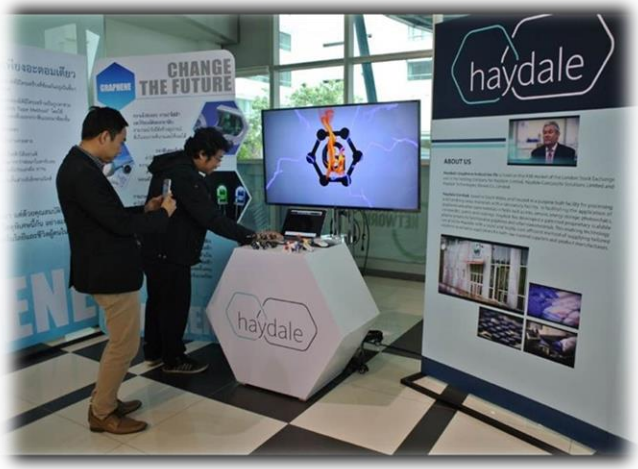
NECTEC
a member of NSTDA

Haydale Graphene Industries establish research center in Thailand

Haydale Technology Thailand (HTT)

www.haydale.com

- Graphene composite
- Graphene ink and coating



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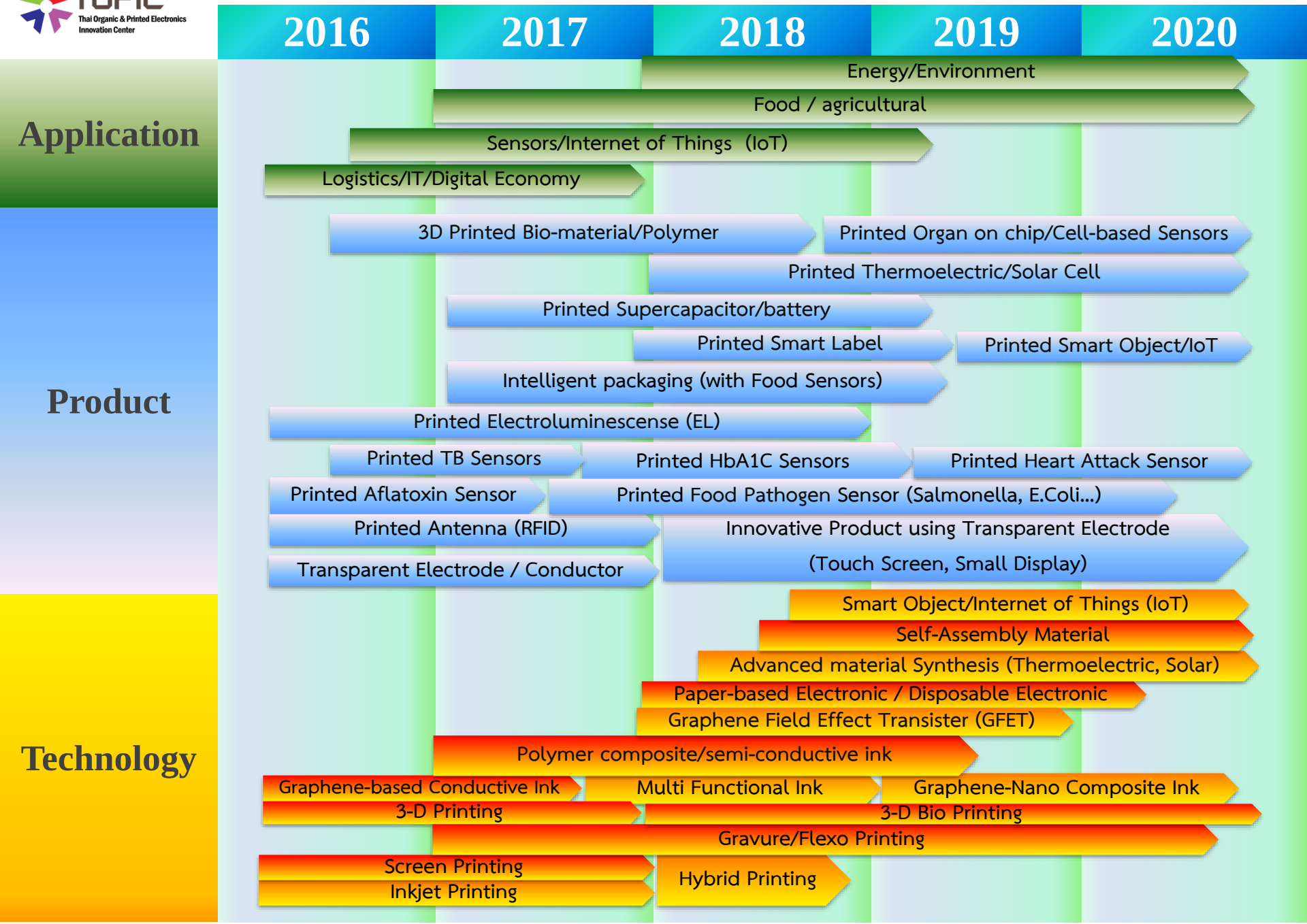

Acknowledgement

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"Thank you for your attention"

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