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Graphene based supercapacitors

Paolo Bondavalli
Thales Research and Technology

31 Mars 2016, NanoInnov Saclay

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Mission

- THALES Research & Technologies is a portal for emerging technologies into THALES Group
- Open organisation, co-located close to or within some of the best research campus in our fields, according to the Group worldwide map of locations

  - **France (Palaiseau) : 350 p + 70 PhD + 80 CNRS-Universities**
    - **Ecole Polytechnique – Plateau de Saclay**
  - **UK (Reading) : 130 p**
    - **University of Surrey**
  - **Netherlands (Delft) : 15 p**
    - **Technological University of Delft - University of Twente**
  - **Singapore : 15 p**
    - **Nanyang Technical University**

The actors of research

THE JOINT LABS

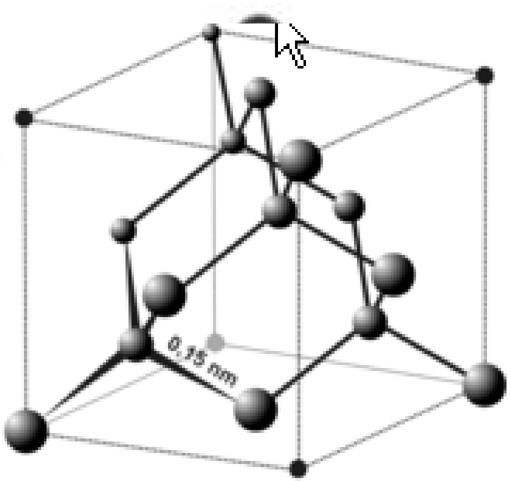
TRT	**INNOVATION PLATFORMS** Work on concrete cases of technology implementation in the products of the group.	DIVISIONS
TRT	**ACADEMICS JOINT LABS** Those common labs have common personnel, common equipment and shared research agenda	ACADEMICS CNRS-Ecole Polytechnique…
TRT	**INDUSTRIAL JOINT LABS** Those common labs have common personnel, common equipment and shared research agenda	INDUSTRIALS ALCATEL

What's graphene?
What are carbon nanotubes?

Allotropic forms of Carbon (until 80s)

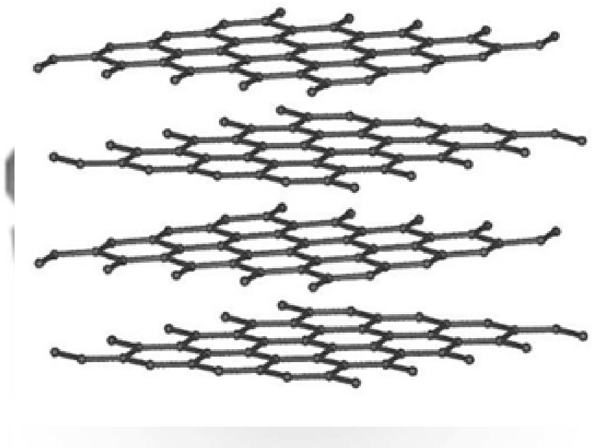
These different ways of rearrange themselves, change the properties of the final "macromaterials". For example graphite is black and quite soft, inversely diamond is transparent and one of the hardest materials discovered up to now.

Diamond



4 Bondings

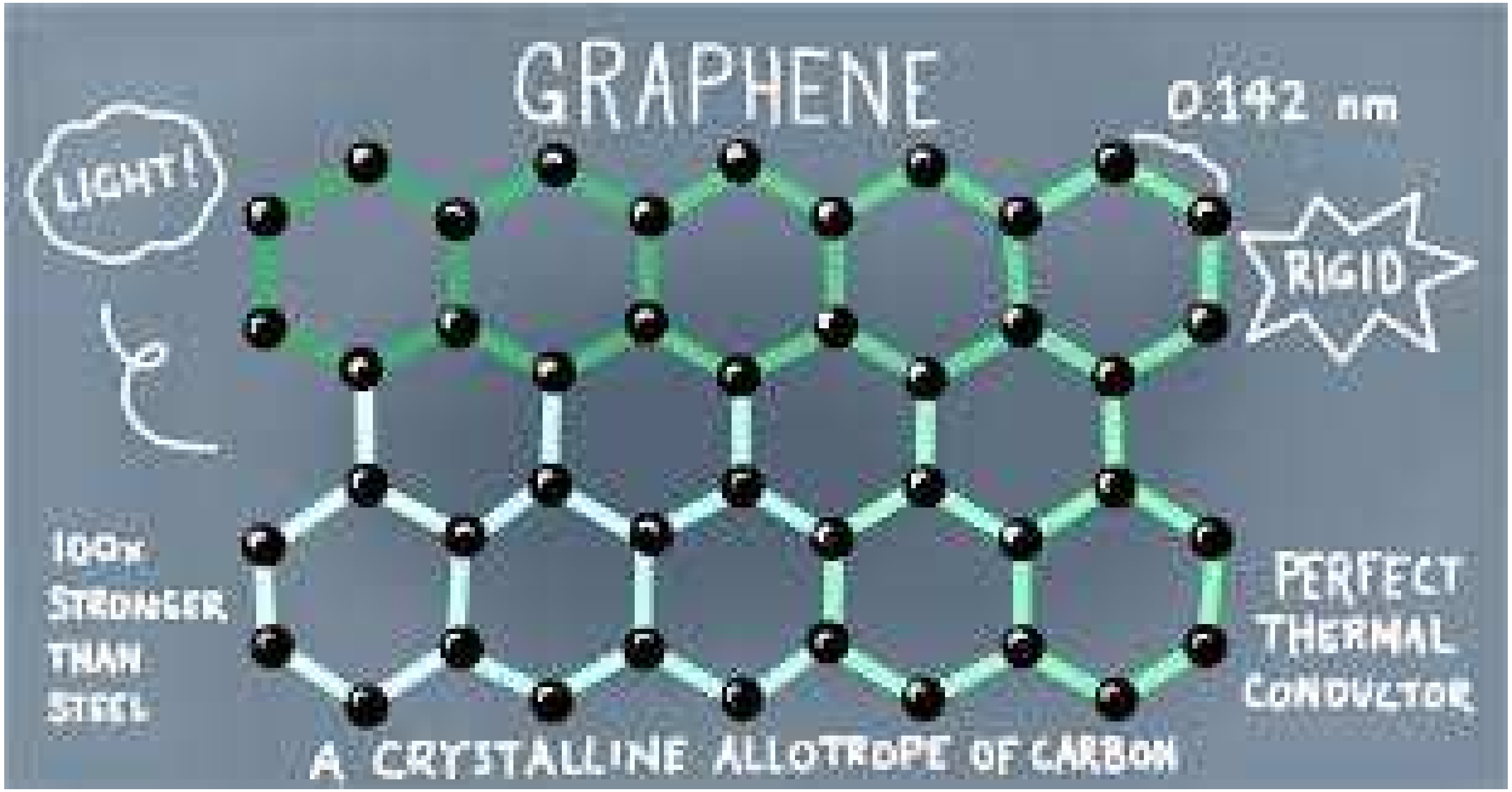
Graphite



3 Bondings

After 80s….

What's graphene ?



The Nobel Prize in Physics 2010

Photo: U. Montan

Andre Geim

Prize share: 1/2

Photo: U. Montan

Konstantin Novoselov

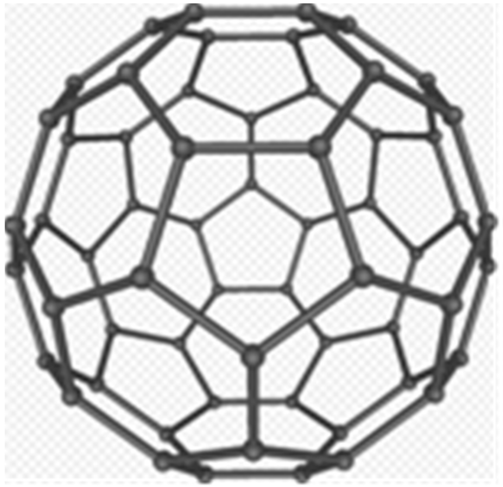
Prize share: 1/2

The Nobel Prize in Physics 2010 was awarded jointly to Andre Geim and Konstantin Novoselov *"for groundbreaking experiments regarding the two-dimensional material graphene"*

1946 Theoretical Prediction (P. R. Wallace, Un. Ontario)

1961First observation of "2D Graphite Lamellae" (Hanns-Peter Boehm, Ludwig-Maximilians-Universität in Munich)

Fullerene : beautiful…but no real application…



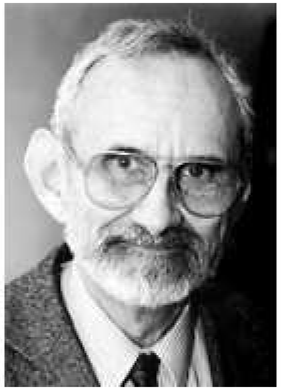
Buckminsterfullerene C60, where 60 is the number of carbon atoms, is the most known form of the fullerene family.

It was first generated in 1985 by Harold Kroto, James R. Heath, Sean O'Brien, Robert Curl, and Richard Smalley at Rice University

1996 Nobel Prize in Chemistry

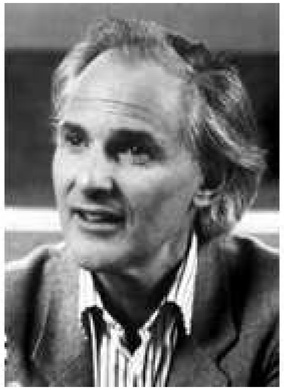
Richard Buckminster Fuller

The Nobel Prize in Chemistry 1996



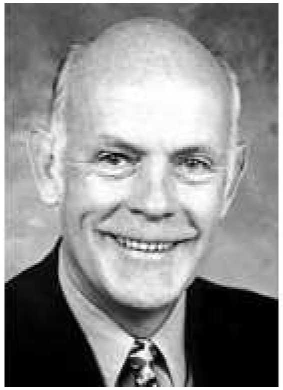
Robert F. Curl Jr.

Prize share: 1/3



Sir Harold W. Kroto

Prize share: 1/3



Richard E. Smalley

Prize share: 1/3

The Nobel Prize in Chemistry 1996 was awarded jointly to Robert F. Curl Jr., Sir Harold W. Kroto and Richard E. Smalley *"for their discovery of fullerenes"*.

Fullerene/Buckyballs   Nanotubes de carbone   Graphite

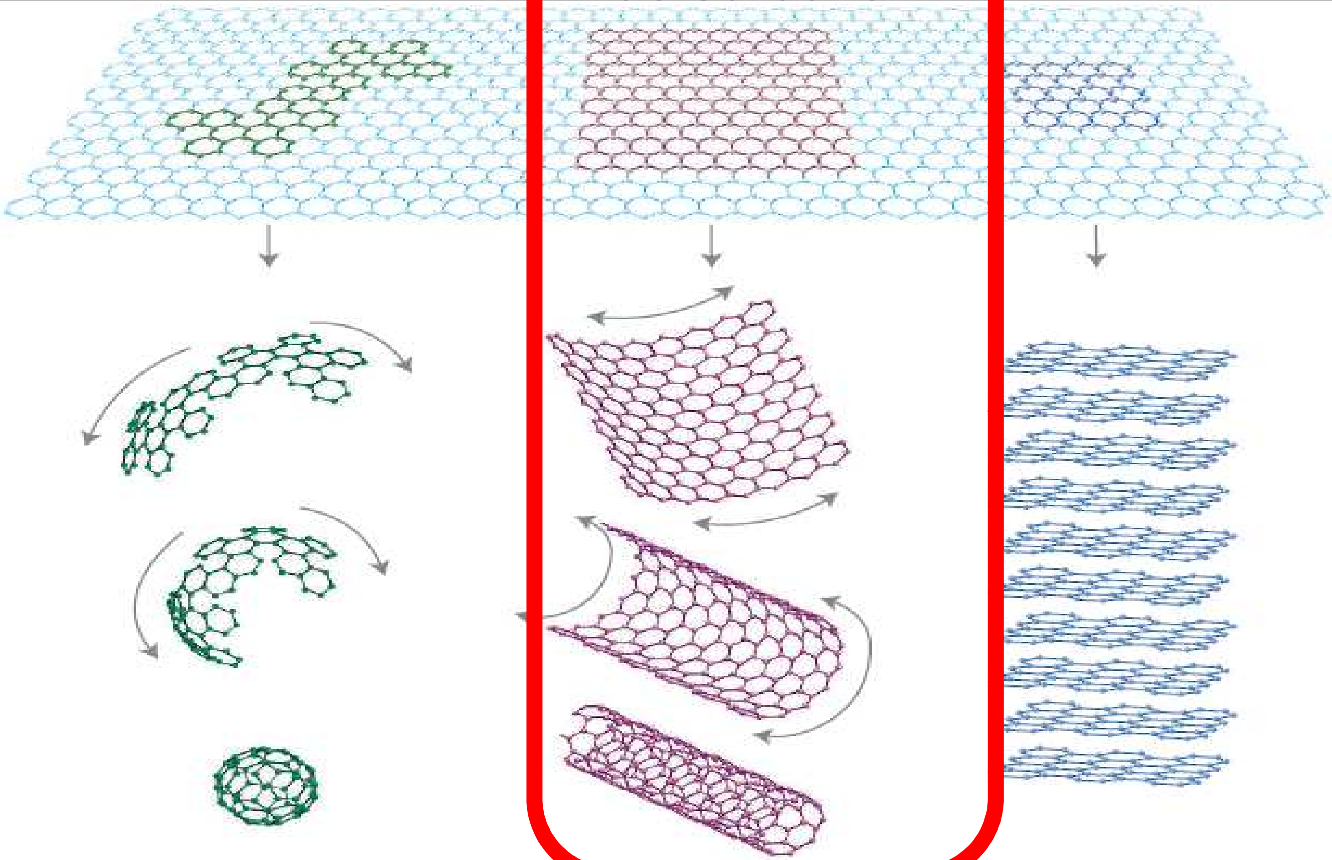


Figure 1 Mother of all graphitic forms. Graphene is a 2D building material for carbon materials of all other dimensionalities. It can be wrapped up into 0D buckyballs, rolled into 1D nanotubes or stacked into 3D graphite.

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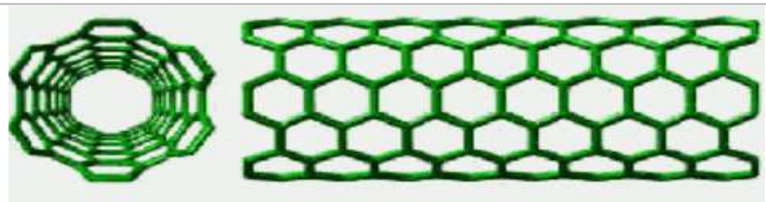
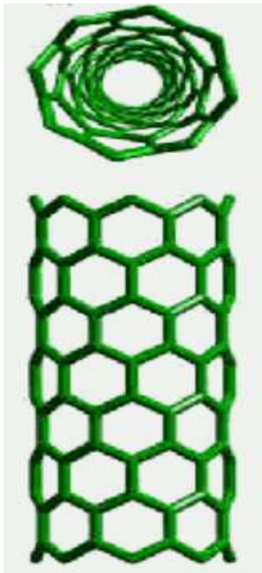
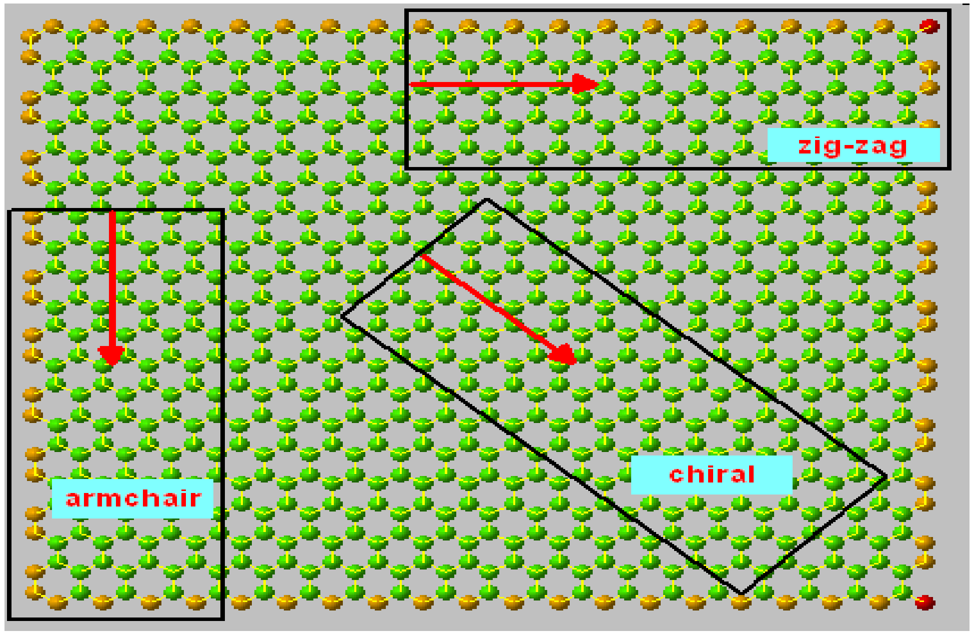
1991 Carbon Nanotubes Discovery

Carbon nanotubes properties : how to roll up graphene?

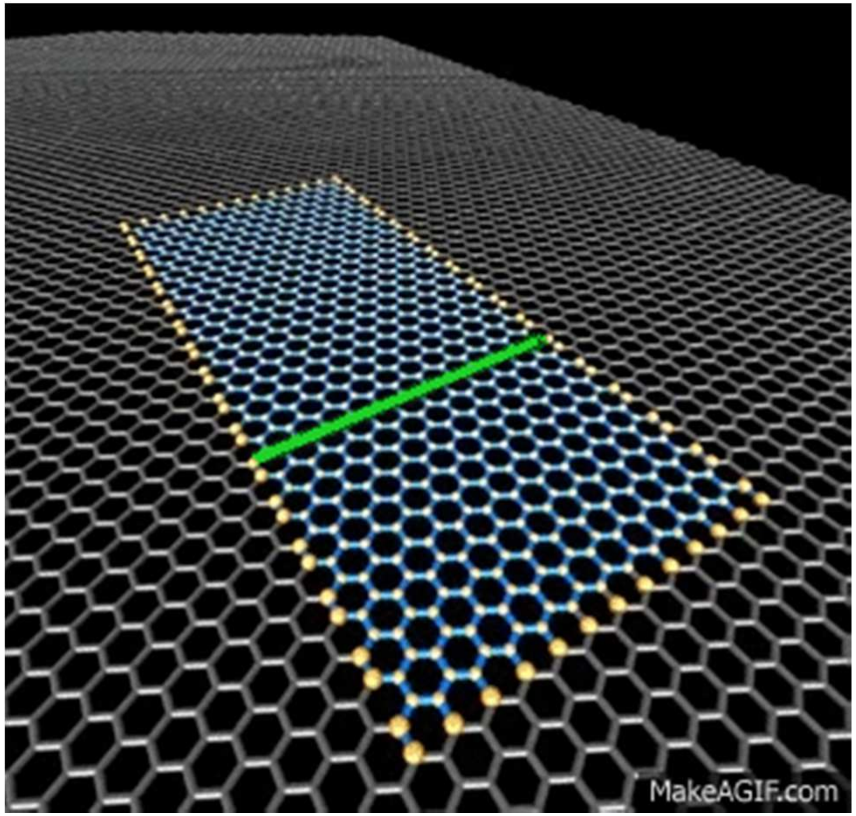




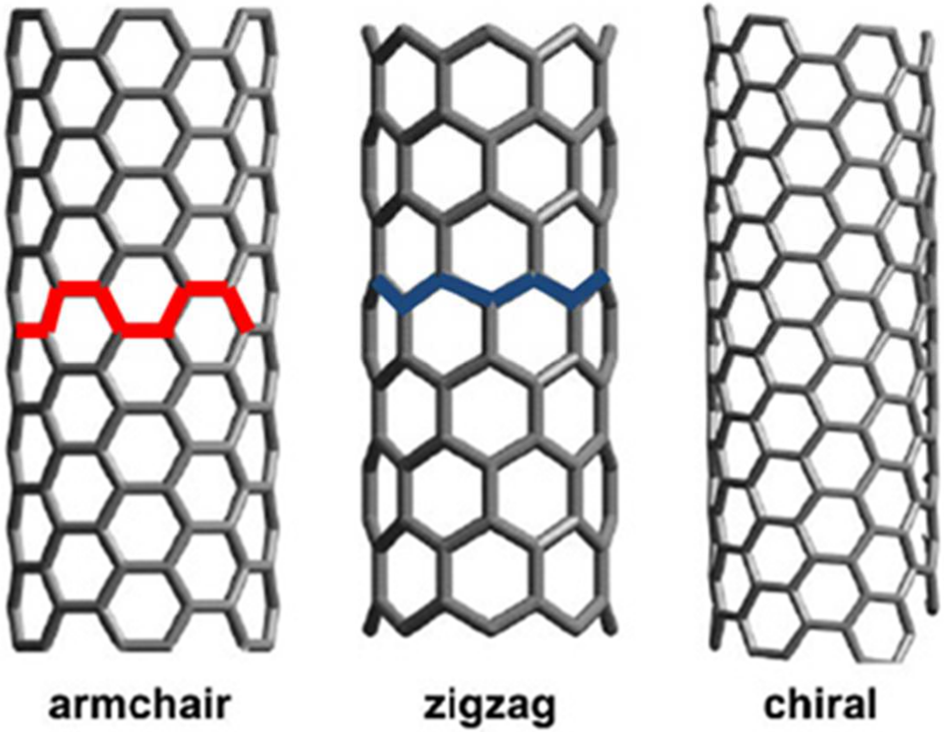


Graphene

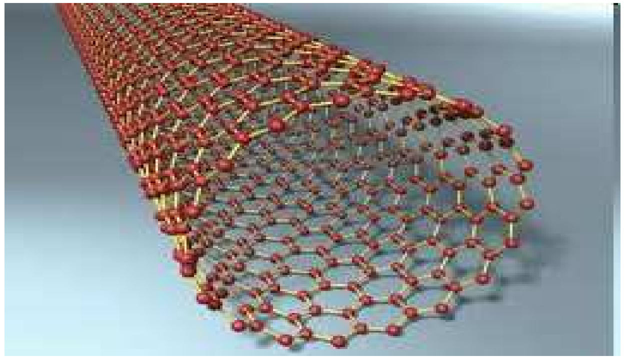
Rolling up Graphene to Obtain Carbon Nanotubes



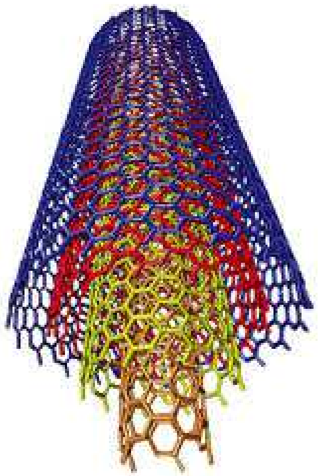
Carbon nanotubes properties : how to roll up graphene?



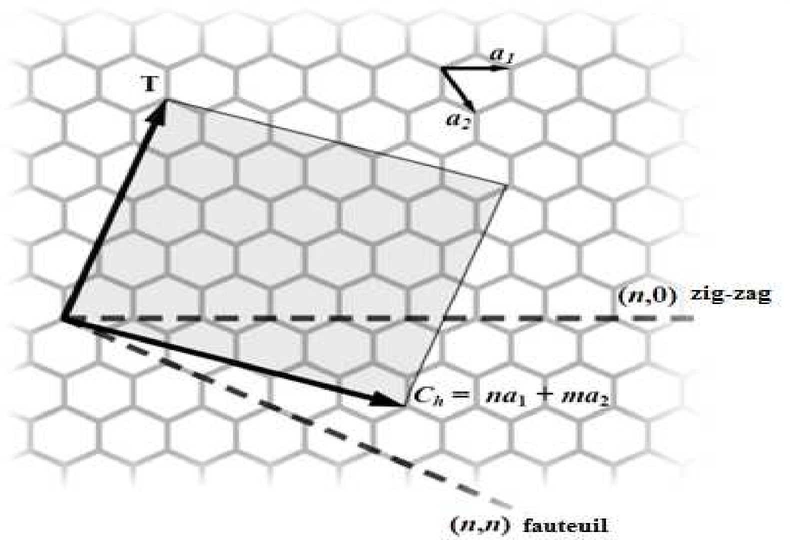
MultiWalled Carbon Nanotubes, SingleWalled Carbon Nanotubes



SWCNT



MWCNT



$$\underline{C}_h = n.\underline{a}_1 + m.\underline{a}_2$$

Saito Law :

$$n\text{-}m \neq 3p \text{ (p entier)}$$

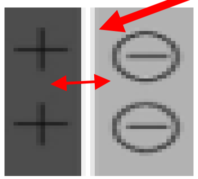
NO NOBEL PRIZE

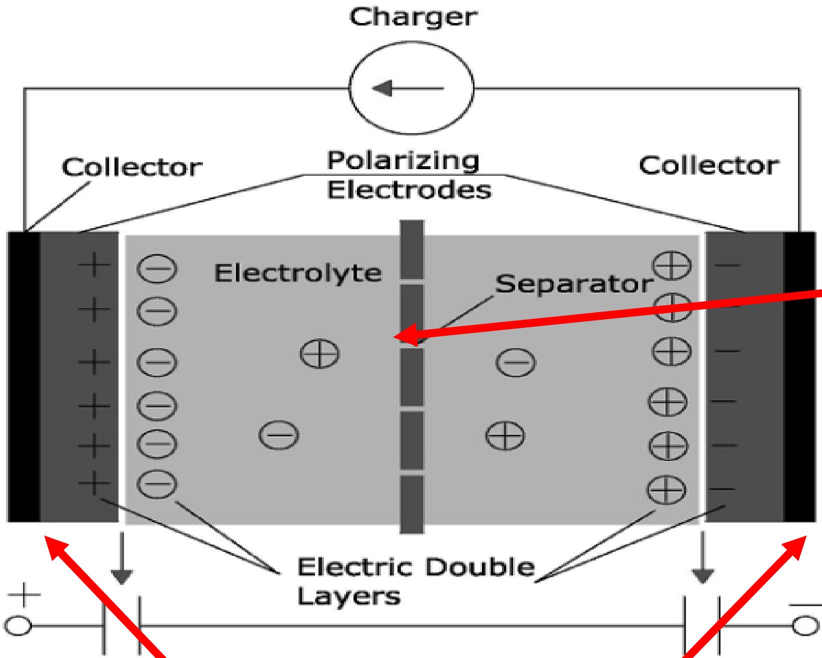
Supercapacitor is the name done by NEC in 1971
Technically is defined Electrical Double Layer Capacitor (EDLC)

$$C=(\varepsilon/\delta)A$$
$$E=(1/2)CV^2$$
$$P=V^2/(4R)$$

Helmotz’s model

δ





separator

Only non faradic reactions

Two electrodes

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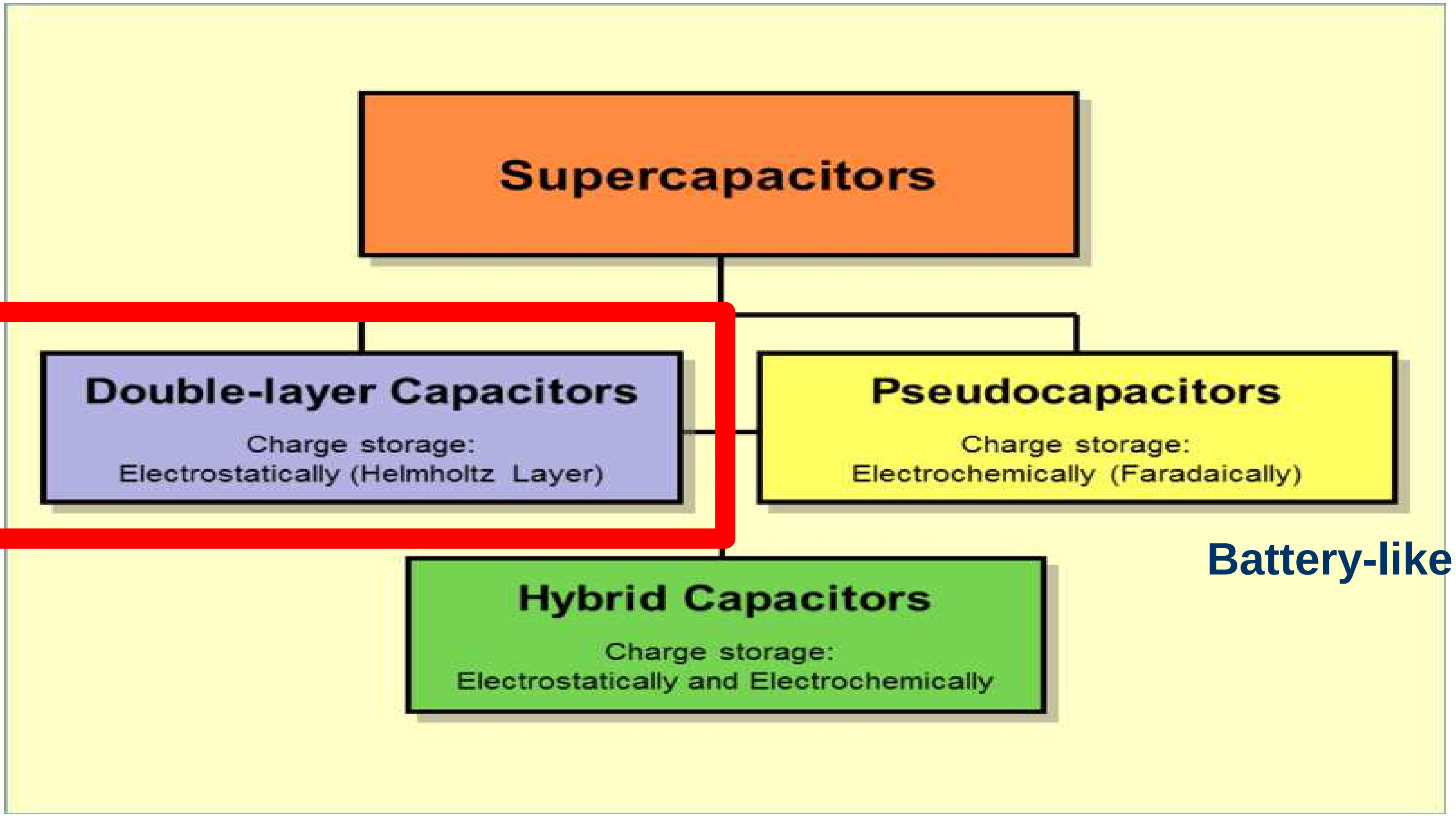

Battery-like devices

Advantages

- Very high rates of charge and discharge
- Higher life cycle (>500000, rechargeable batteries can attain 10000)
- Good reversibility
- Low toxicity of material used
- High cycle efficiency
- Low internal resistance (Higher output power)
- Extremely low heating levels

Drawbacks

- Low amount of energy stored (3-5 Wh/Kg vs 30-40 Wh/Kg for batteries)
- It requires sophisticated control and switching equipment (from batteries to supercaps)

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1957 : the first patent on EDLC (Electrical Doucle Layer Capacitance)
General Electric : Electrolytic capacitor with porous carbon electrodes

2,800,616

LOW VOLTAGE ELECTROLYTIC CAPACITOR

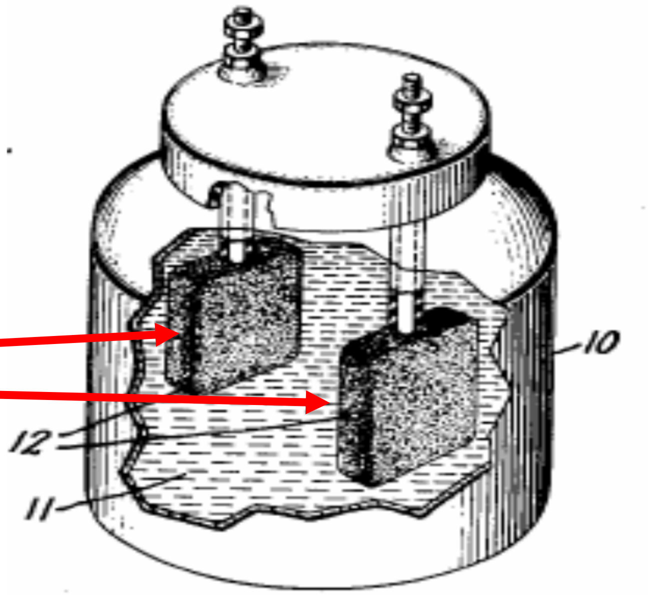
Howard I. Becker, Vischers Ferry, N. Y., assignor to General Electric Company, a corporation of New York

Application April 14, 1954, Serial No. 423,042

4 Claims. (Cl. 317—230)

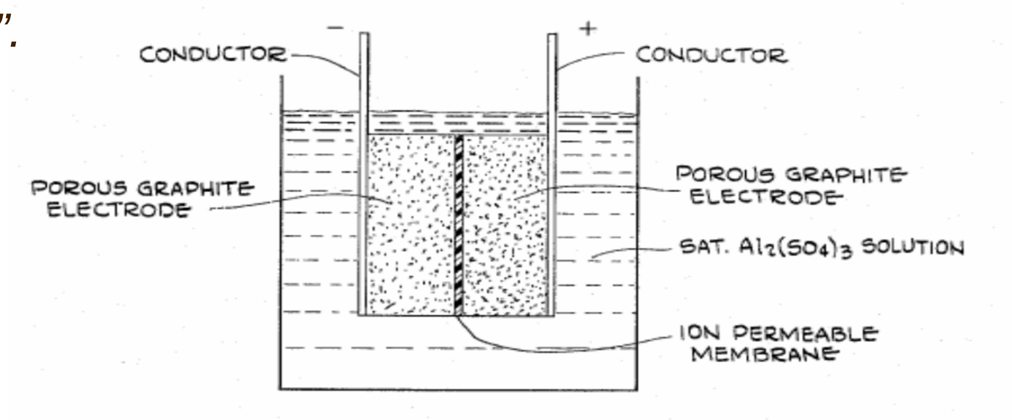
1

Two porous graphite electrodes



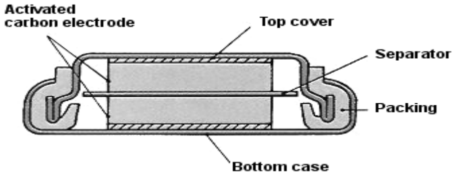
1966 : SOHIO (Standard Oil company, OHIo)

At this time SOHIO acknowledged that "*the double-layer at the interface behaves like a capacitor of relatively high specific capacity*".



1971 : NEC : first commercial device called « supercapacitor » based on technology suggested by SOHIO: Memory backup applications (low voltage, high internal resistance)

<u>1978 : Panasonic : « gold capacitor » </u>: back-up energy source for microprocessors and solar batteries (power failures include video recorders, DVD players, fax machines, telephones, digital still cameras, mobile phones, audio stereo systems, etc.)



(Fig.2)

<u>1982 : Pinnacle Research Institute </u>: « PRI **<u>ultracapacitor</u>** » (first time that the term « ultracapacitor » is used) incorporated metal-oxide electrodes

Applications : laser weaponery and missile guidance (military applications)

Some visible supercap applications

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Activated carbon: parameters

Non-faradic carbon nanotubes based supercapacitors : state of the art, **P.Bondavalli,** et al. , Eur. Phys. J. Appl. Phys. 60,10401, 2012

Main parameters

- Surface (energy)
- High breakdown voltage (energy)
- Pore size (to exploit surface completely and to promote easy ion diffusion)

Activated Carbon

- Large surfaces ($3000 m^2/g$)
- Low-cost material

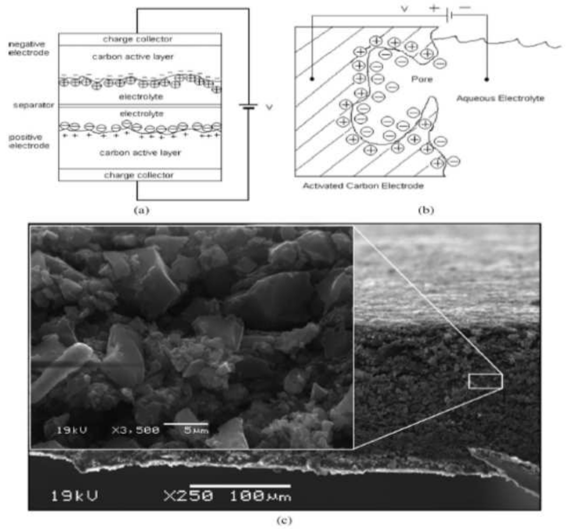

Fig. 1. *(a) Schematic of an activated carbon-based EDLC. (b) Representation of pore in carbon electrode active layer. (c) Electron micrograph of activated carbon electrode.*

The main issue :

- Very bad mesoporous distribution!!!

  2/3 of the pore size are smaller than 2 nm and so are unpercolated)

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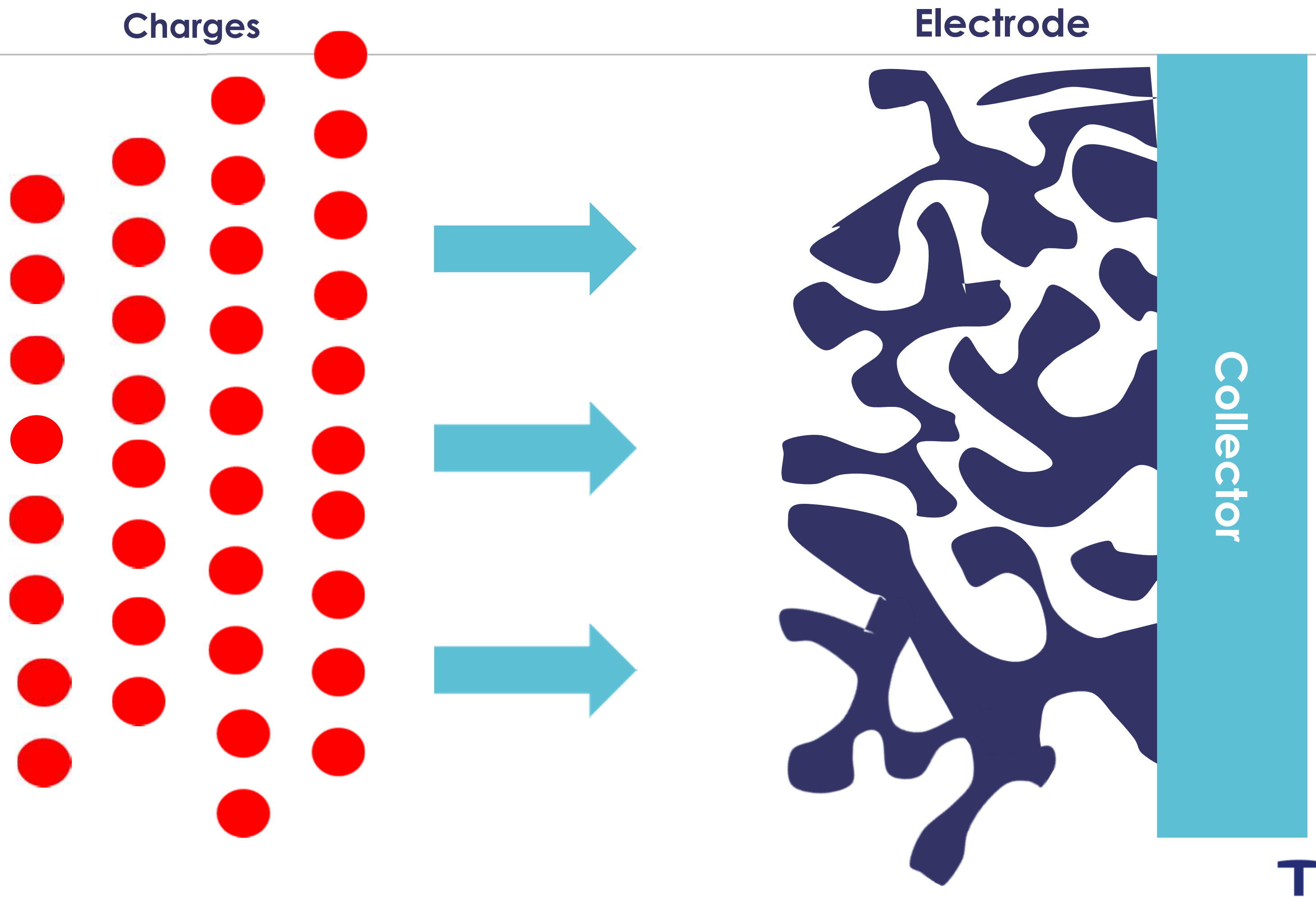

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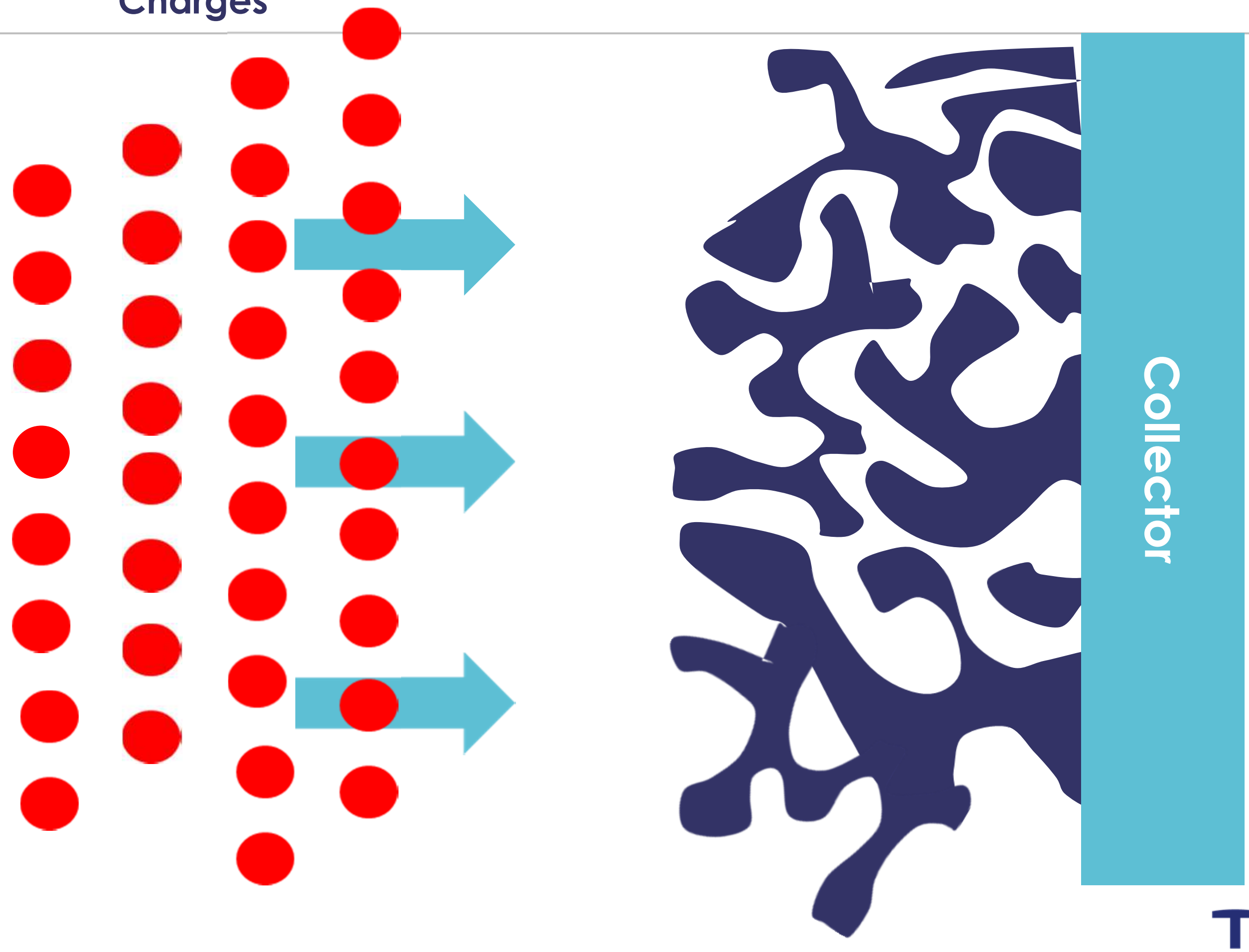
Charges



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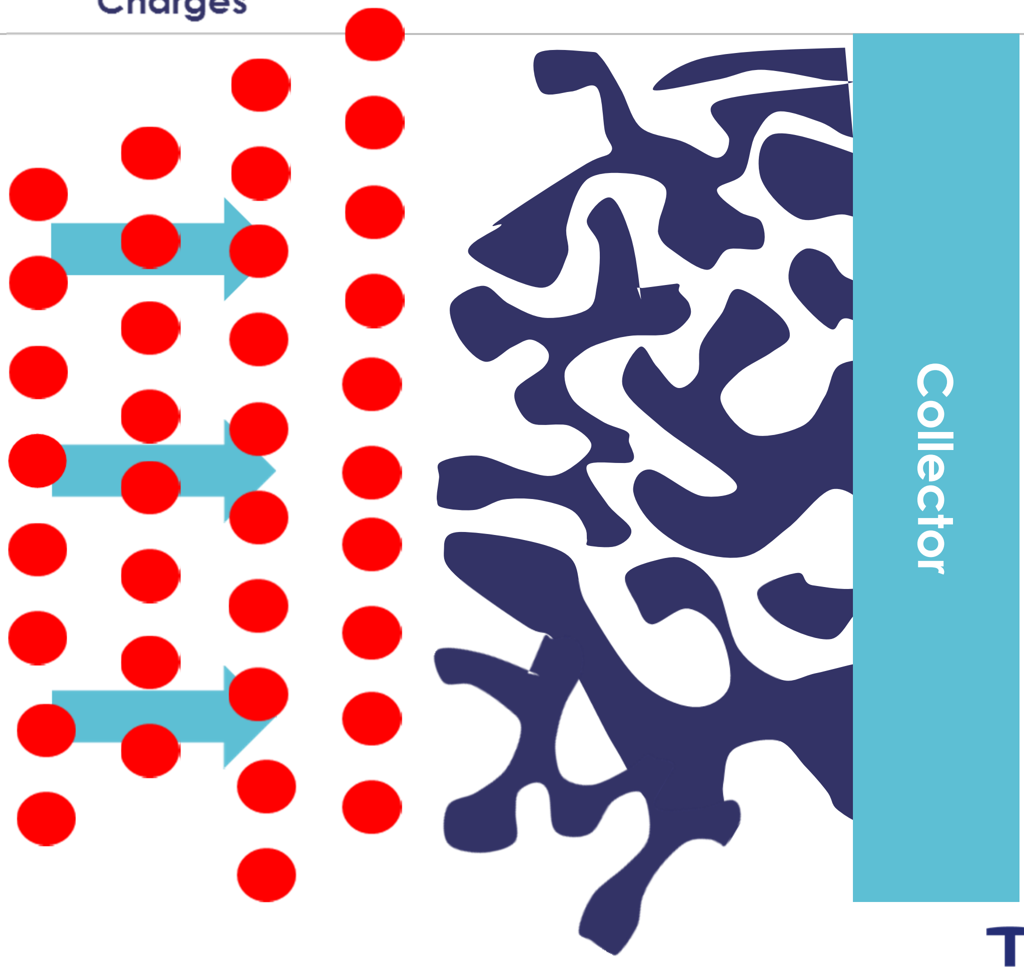
Charges



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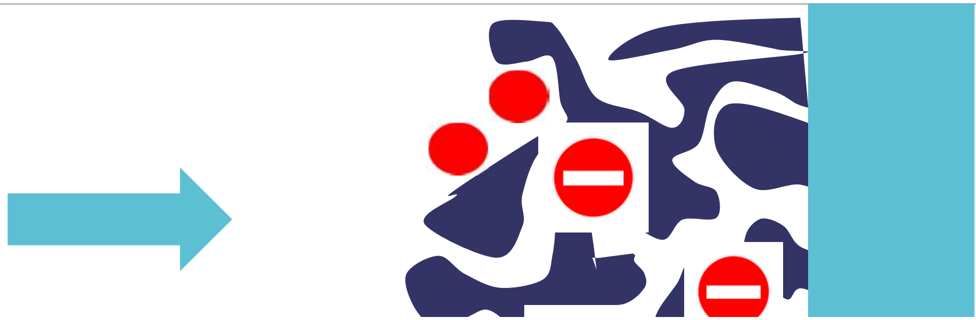
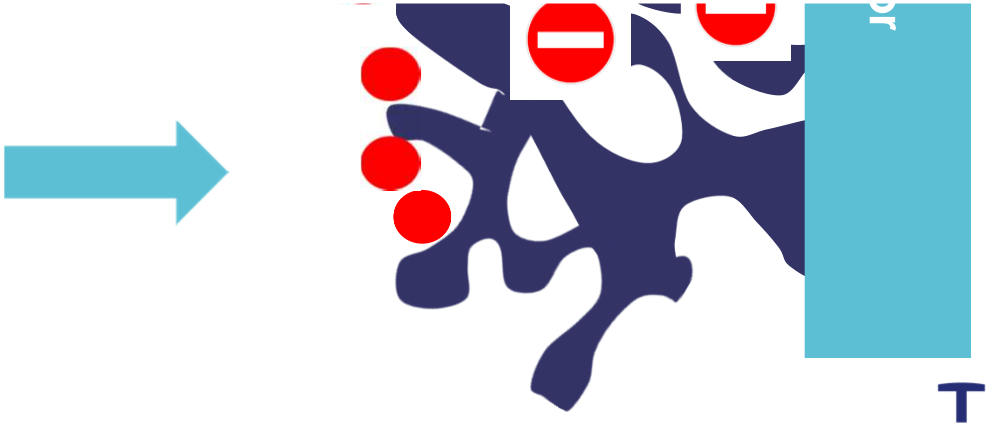
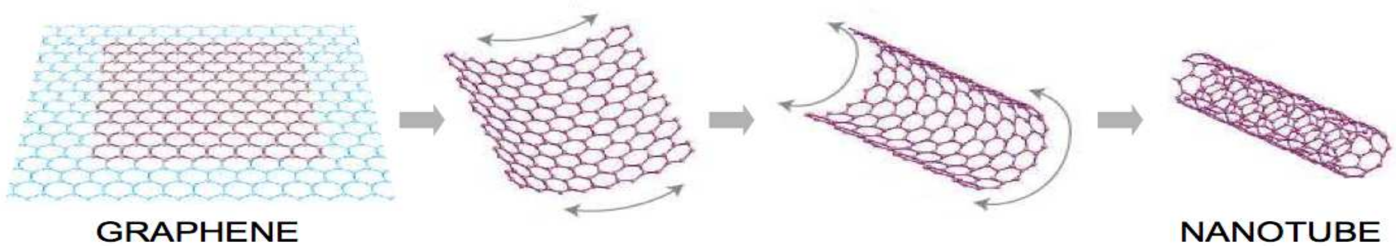
2/3 of the surface is not exploited

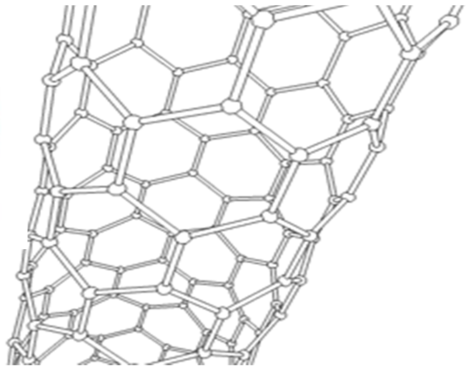


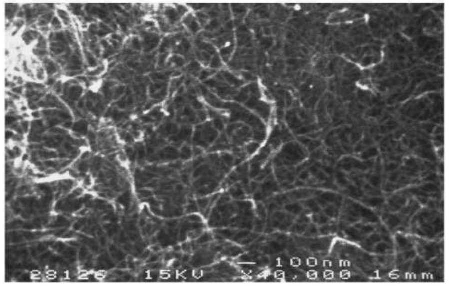
PORE SIZE IS NOT OPTIMIZED AND SURFACE IS NOT ADEQUATELY EXPLOITED



What are carbon nanotubes and why carbon nanotubes for Supercaps?







Niu, C.; Sichel, E.K.; Hoch, R.; Moy, D.;
Tennent, H., *Appl. Phys. Lett.* 1997, 70(11)
1480-1482 Hyperonc Inc.

- **Randomly entangled nanotubes for electrodes can be fabricated easily**
- **Highly surface specific surface area (300m²/Kg)**
- **High mesoporous distribution (2-5nm) and so electrolyte accessibility**
- **Low resistivity (they can be used as electrode and collector)**
- **We can fabricate electrodes without binder (higher breakdown voltage)**
- **Total weigth is very low (enhancement energy and power density)**
- **High stability (long life-time)**

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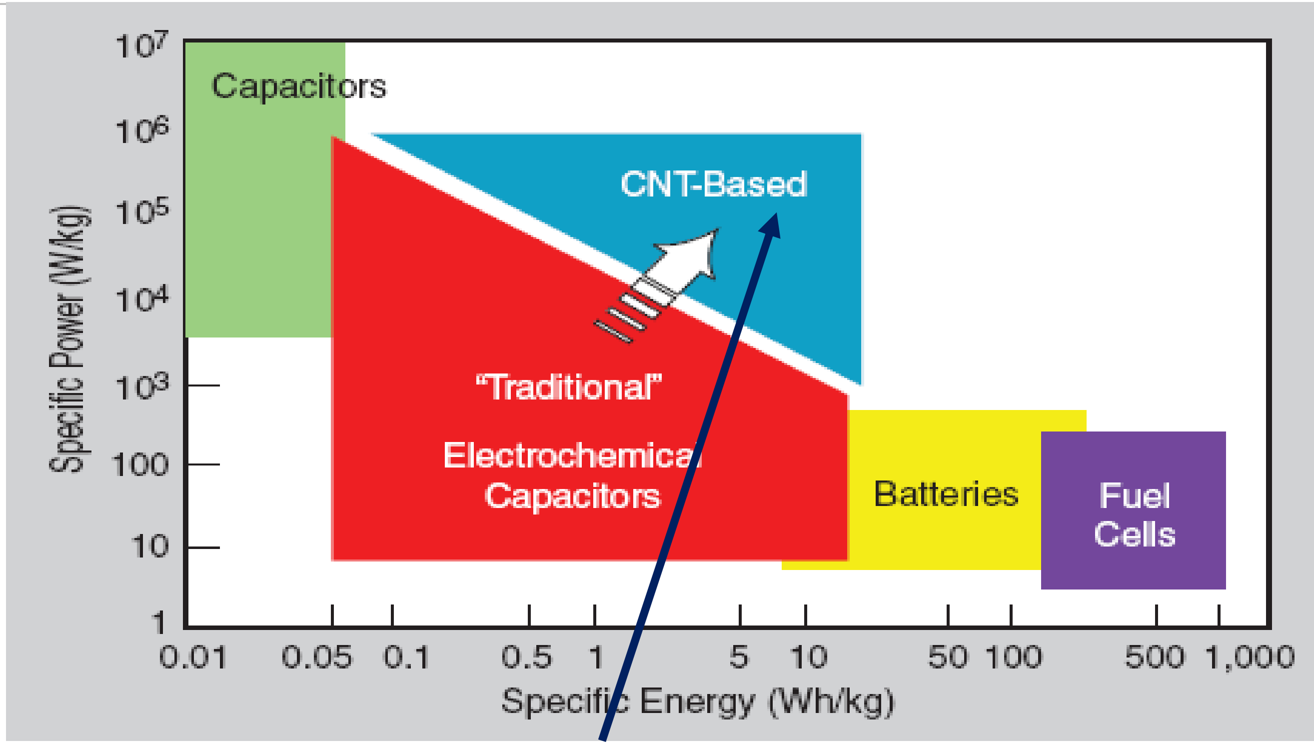
CNTs (and Graphene related materials) based Supercapacitors



Considering that supercapacitors bridge the gap of capacitors and batteries performances we have to attain performances in this zone

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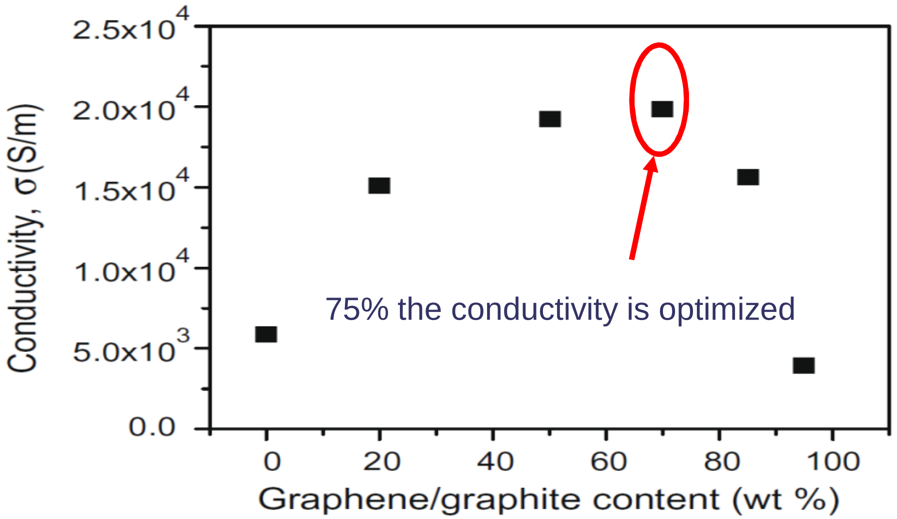
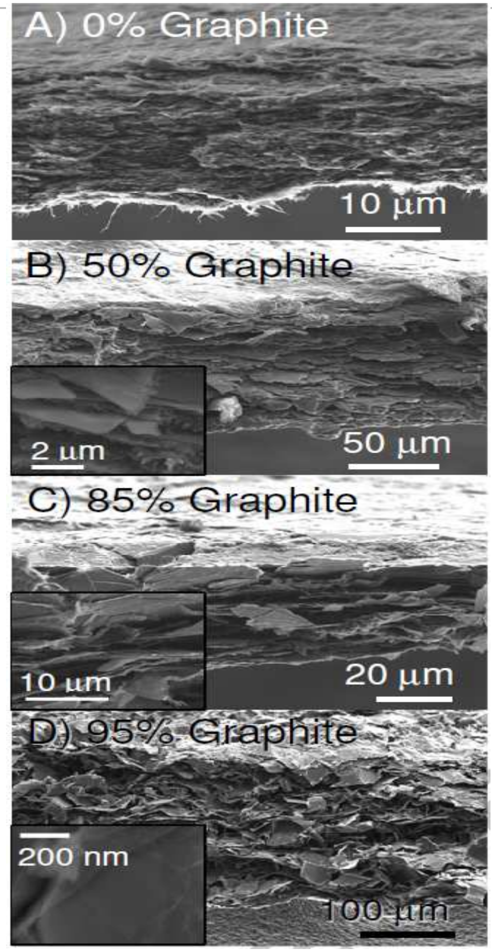
Why to use Graphene related materials and CNTs mixings?

- CNT/graphene/graphite composite



U. Khan, J. N. Coleman et al Carbon (2010)



Resistance is reduced by a factor of 4 compared to bare CNTs layers

Can we improve the Power output (P∝ 1/R)?

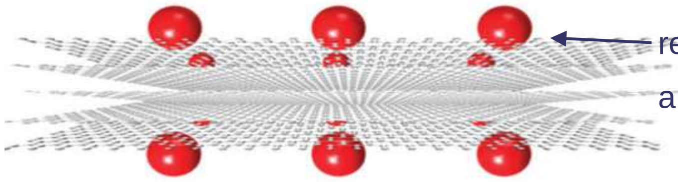
Why to use Graphene related materials and CNTs mixings?

Cite this: *Phys. Chem. Chem. Phys.*, 2011, 13, 17615–17624

www.rsc.org/pccp **PAPER**

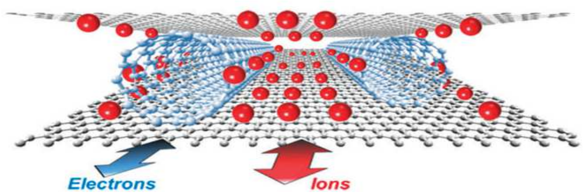
Graphene and carbon nanotube composite electrodes for supercapacitors with ultra-high energy density

Qian Cheng,[ab] Jie Tang,*[ab] Jun Ma,[a] Han Zhang,[a] Norio Shinya[a] and Lu-Chang Qin[c]



reduced charge diffusion and surface

Pristine graphene/graphite



Graphene/graphite/CNTs mixing

- **CNTs prevent restacking (higher surface, higher energy stored)**
- **CNTs/graphite/graphene improve conduction (higher power delivered)**
- **CNTs prevent the disintegration of the composite**

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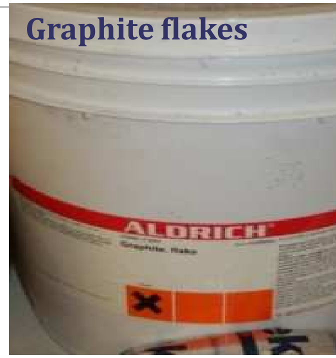
Our Approach

CNTs



Separated weighing

↓

Separated dispersion
(solvent = NMP)

↓

Dilution to get C_{solide} = 0,5g/l

Initial sonication
- CNT : 10’ high power
- Graphite : 18h low power

Centrifugation
10 minutes x2

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Final sonication of the mixture :
18h low power

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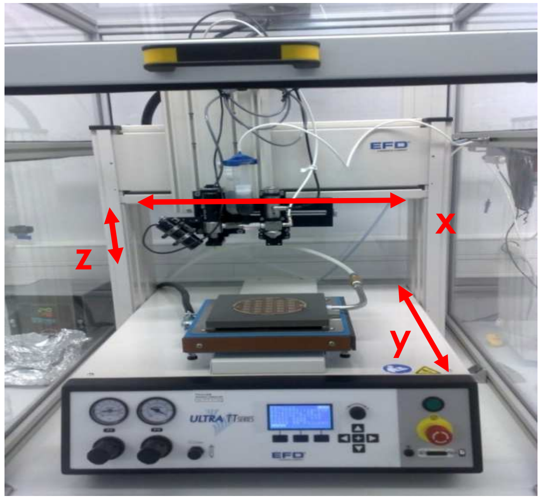
Deposition method

- Excellent reproducibility
- Versatile, easily scalable for large-area applications
- Extremely uniform deposition with no “coffee-ring” effect



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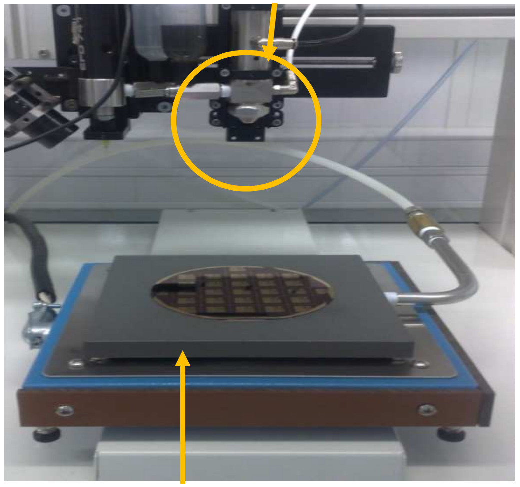

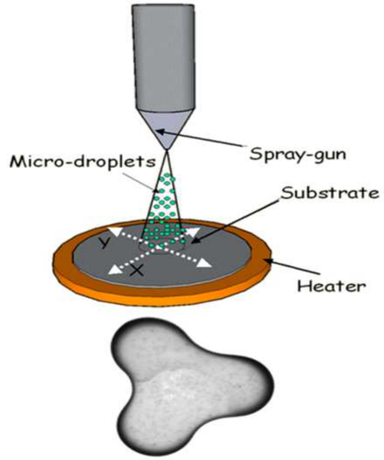

Process patented

3-axes displacement

Heating plate

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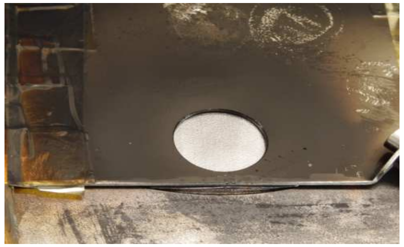
Supercapacitor electrode based on mixtures of graphite and carbon nanotubes deposited using a new dynamic air-brush deposition technique, **P Bondavalli**, JECS 160 (4) A1-A6, 2013

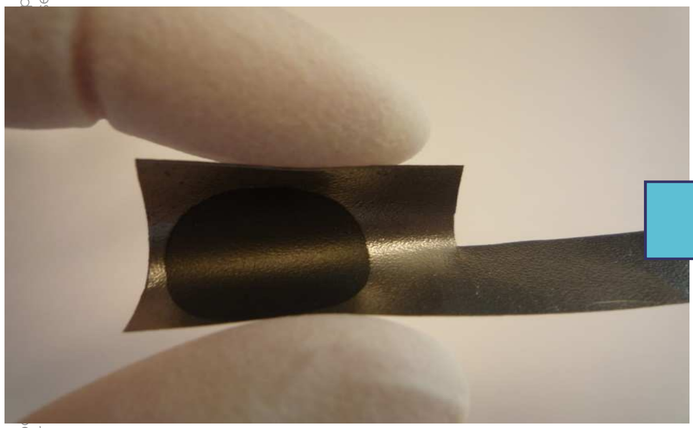
Air-brush deposition

- **Gun spraying**
- **Masking**
- **Several samples fabricated at the same time**

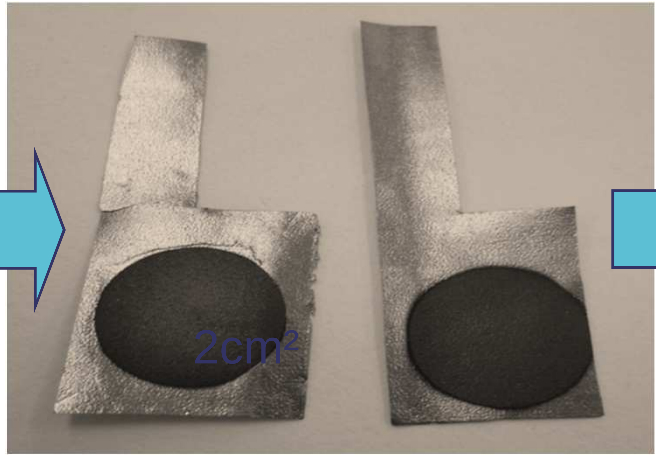
Panasonic Graphite bucky paper



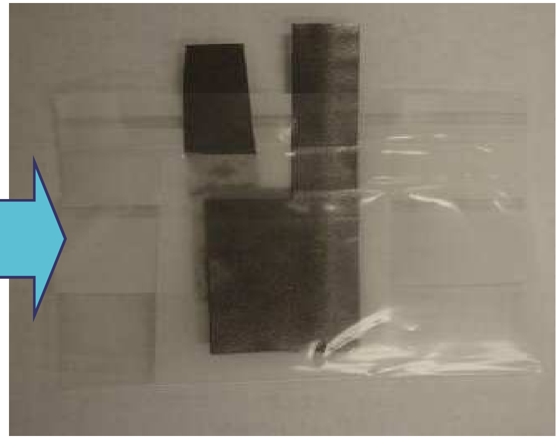
Flexible electrodes



Electrode design

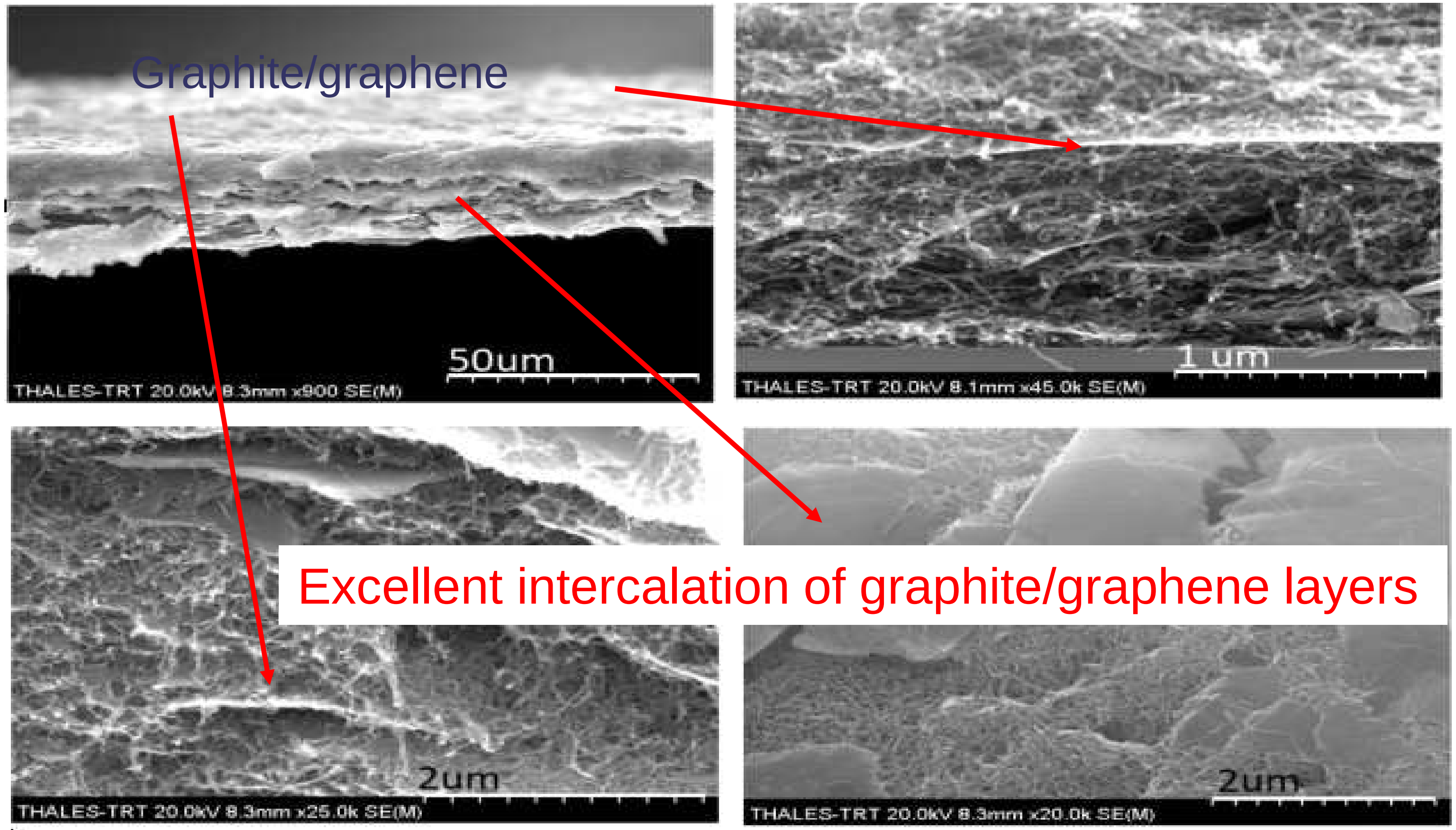


Supercapacitor Cell



Sample Morphology (cross section)

Supercapacitor electrode based on mixtures of graphite and carbon nanotubes deposited using a new dynamic air-brush deposition technique, **P Bondavalli**, JECS 160 (4) A1-A6, 2013



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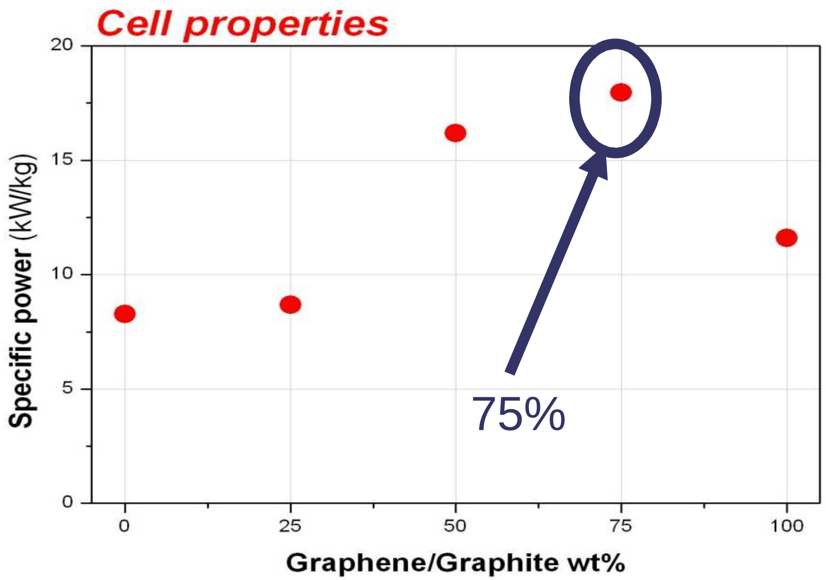
Results : Energy and Power as a function of the concentration

Sample characteristics :
- weight = 1.8mg
- surface = 2cm² (circular design)
- thickness ~ 20µm

A - Influence of the CNT concentration (Electrodes)

- **Energy max. ~4,5Wh/kg for 75wt%CNT**
- **Power max. ~15 kW/kg for 25wt%CNT (enhancement of 2,5)**



Supercapacitor electrode based on mixtures of graphite and carbon nanotubes deposited using a new dynamic air-brush deposition technique, **P Bondavalli**, JECS 160 (4) A1-A6, 2013

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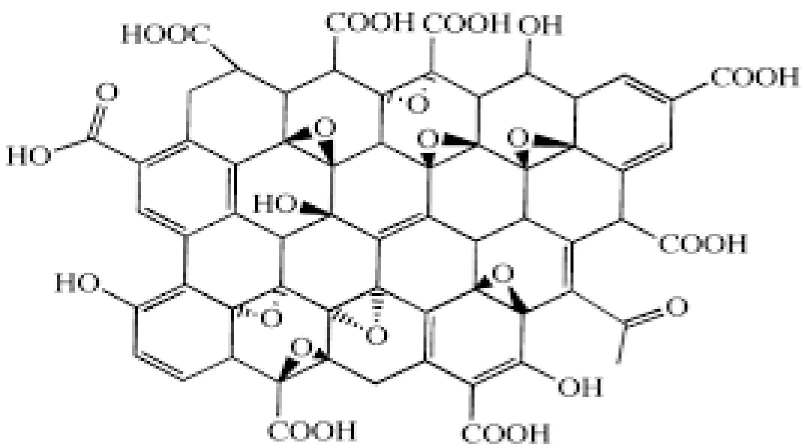
Last measurements : new option for green suspensions using GO

Mixing of Graphene Oxyde and Oxydised Carbon Nanotubes in water

Advantages

- **Aqueous based supensions**
- **Very stable suspensions**
- **Low temperature process (120°C)**



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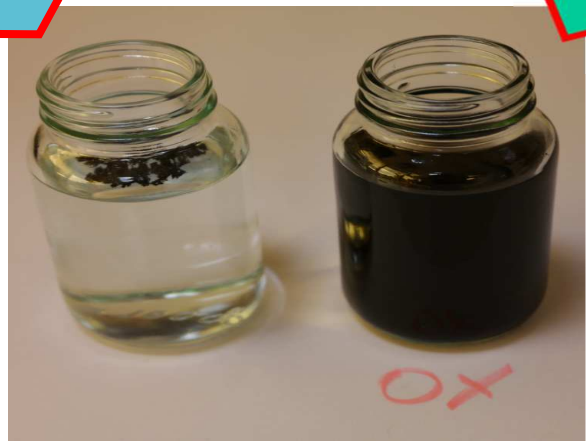
Oxidised CNTs can be put into water based suspensions very easily

Non-oxidised CNTs



Oxidised CNTs

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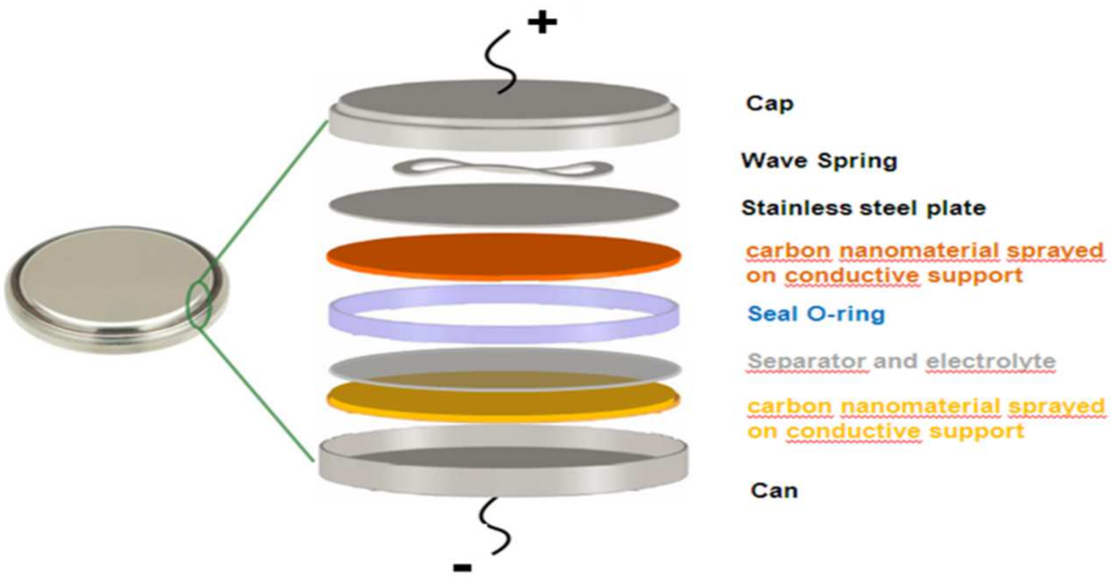
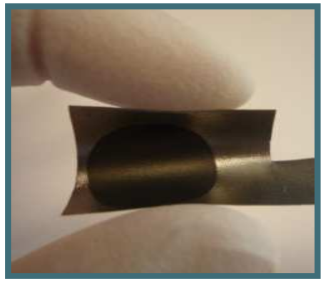
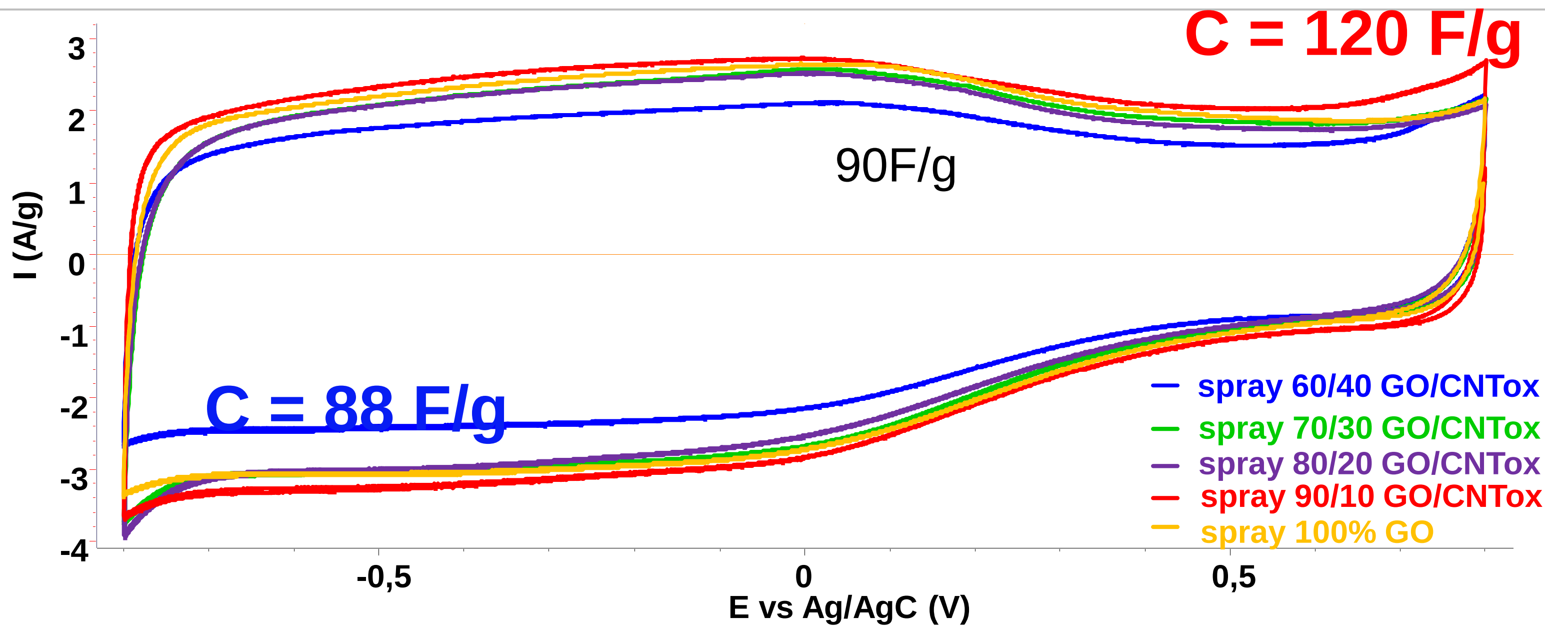
New packaged prototypes





THALES

Performances for different GO concentrations

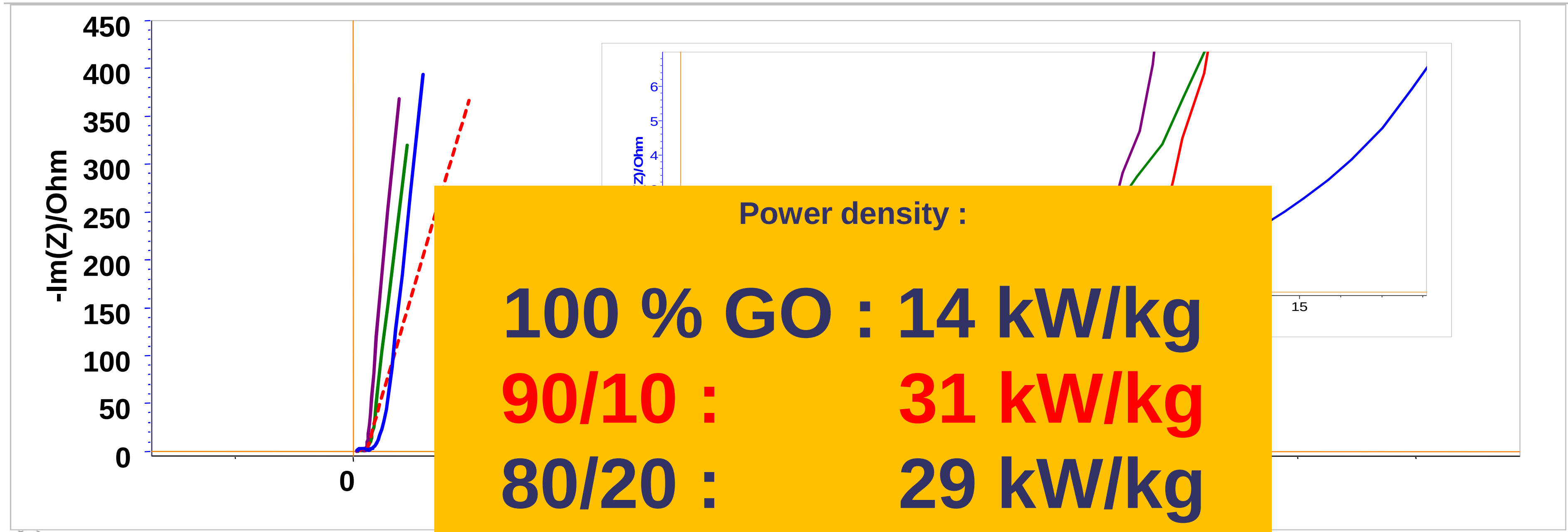


The surface allows to know the capacity of the materials to store energy

Graphene based electrodes for high performances supercapacitors, **P.Bondavalli,** G.Pognon, Procedding of IEEE NANO 2015, 17-20 July, Rome

THALES

Power density



Power density :

100 % GO : 14 kW/kg

90/10 : 31 kW/kg

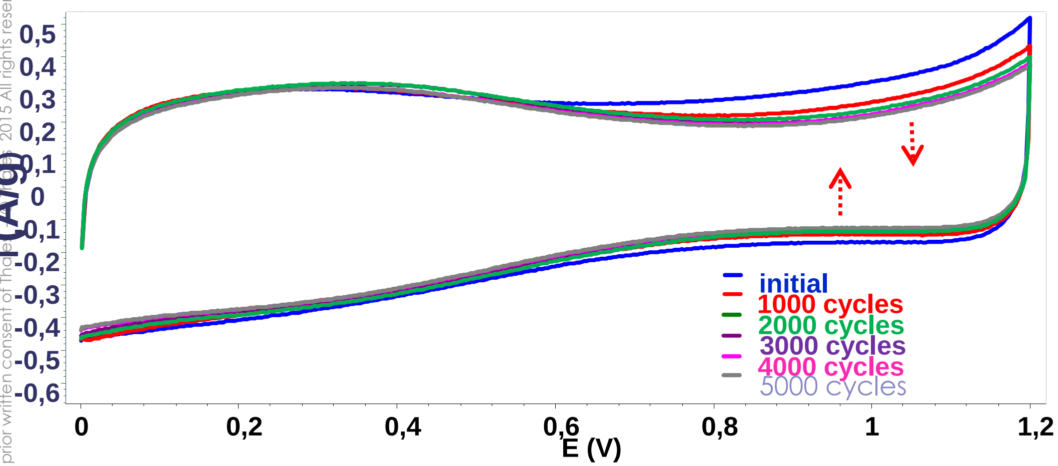
80/20 : 29 kW/kg

70/30 : 21 kW/kg

THALES

Graphene from IIT : Galvanostatic charge/discharge experiment

Very good stability (same capacitance that using GO)



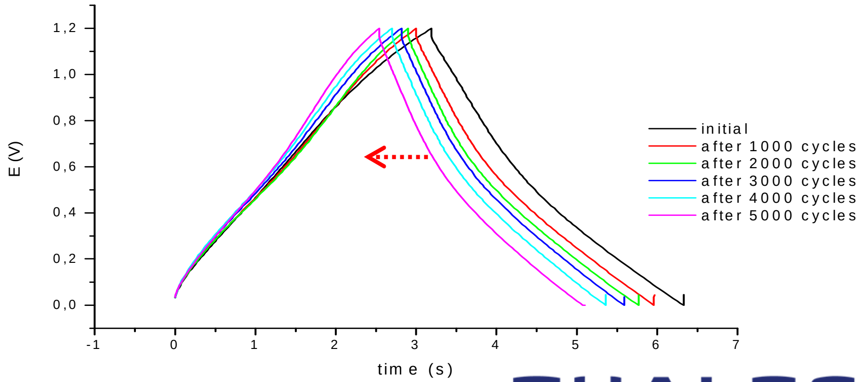
two-electrodes configuration

→ complete system

m = 1,15 mg (x2)

5000 cycles

I= 10mA

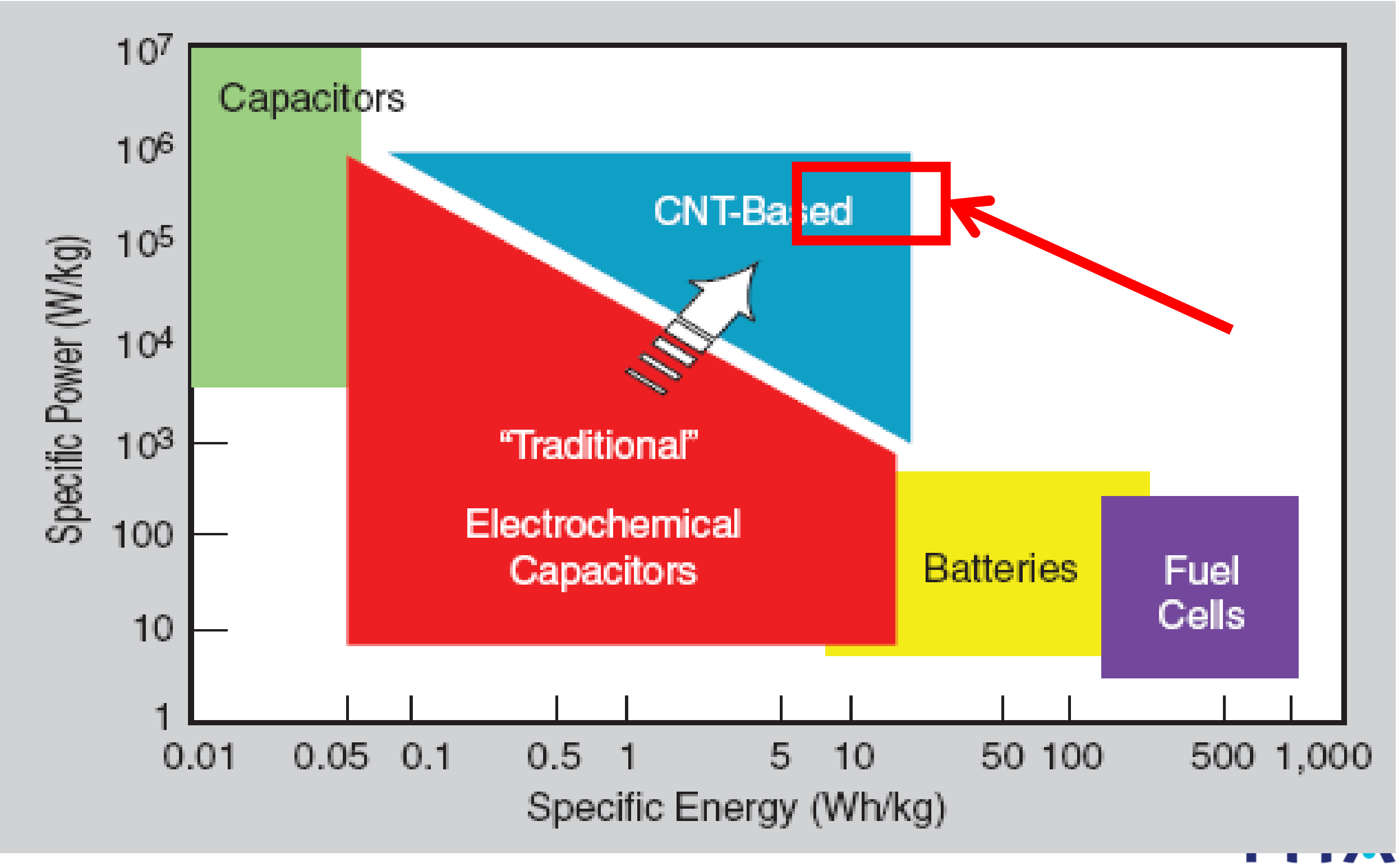


Graphene exfoliated by IIT :

- Loss essentially during the 1000 first cycles

-P= 92,3 kW/kg

THALES



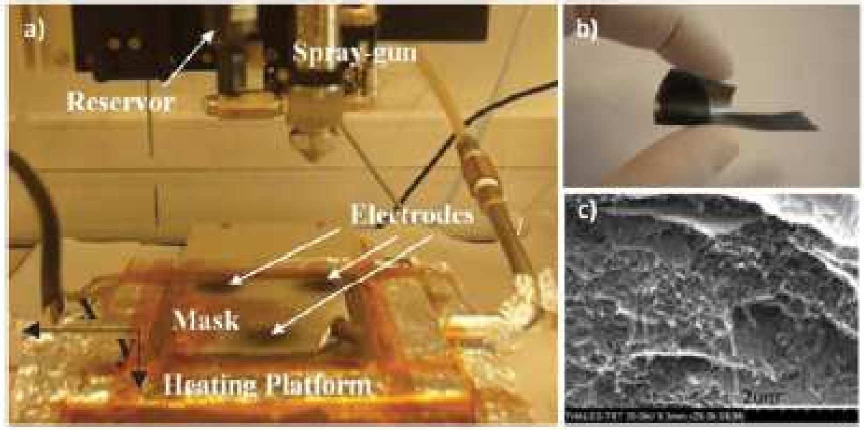
10^7
10^6
10^5
10^4
10^3
100
10
1
Specific Power (W/kg)
Capacitors
CNT-Based
"Traditional"
Electrochemical
Capacitors
Batteries
Fuel
Cells
0.01 0.05 0.1 0.5 1 5 10 50 100 500 1,000
Specific Energy (Wh/kg)

THALES

Contracts and Objectives

GRAPHENE FLAGSHIP

> **Results published in « 2D Materials » and presented at IEEE NANO 2015**



d)

Composite	Capacitance (F/g)	Power density (kW/kg)
Graphite/CNT	25	16
Graphene/CNT	120	92
GO/CNT_{ox}	120	25

Figure 11. (a) Dynamic spray-gun deposition set-up used for the deposition of mixtures of nanomaterial graphite collectors; (b) flexible electrode deposited on a graphite collector; (c) SEM cross section of a graphite collector covered with a mixture of sprayed graphene/graphite (50%)/CNTs (50%); (d) typical electrode performance versus composite material.

THALES

Contracts and Objectives

GRAPHENE FLAGSHIP

> Results published in « 2D Materials » and presented at IEEE NANO 2015 (link2)

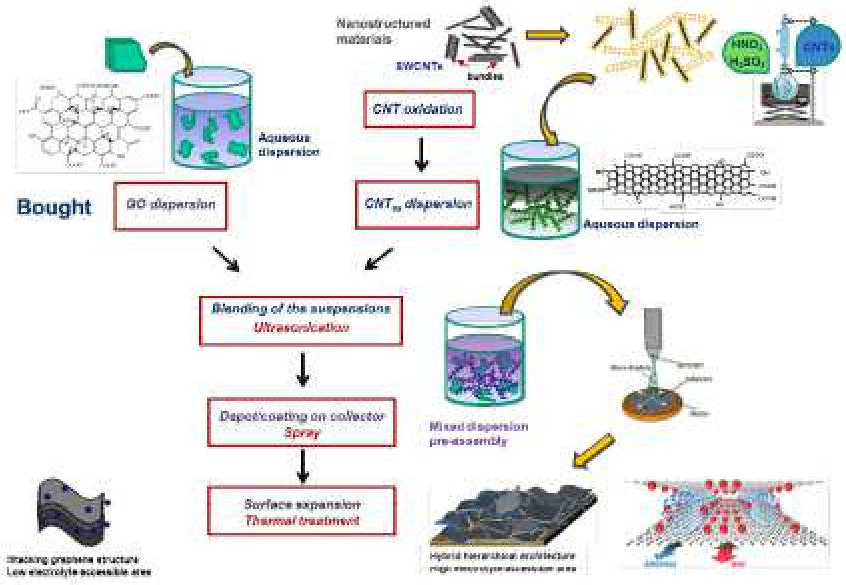


Fig3: Process to obtain stable suspensions of mixture of GO and oxidised Carbon Nanotubes (ox-CNTs).

THALES

Graphene Oxide based electrodes

Advantages

- Water based suspensions
- Low cost material
- Very stable suspensions (months, years?)
- Capacitance of 120F/g, Power density of 30kW/Kg

Drawbacks

- Power lower than for Graphene (factor three)

Graphene based electrodes

Advantages

- Same capacitance that GO but Larger power density demonstrated (~100kW/Kg)

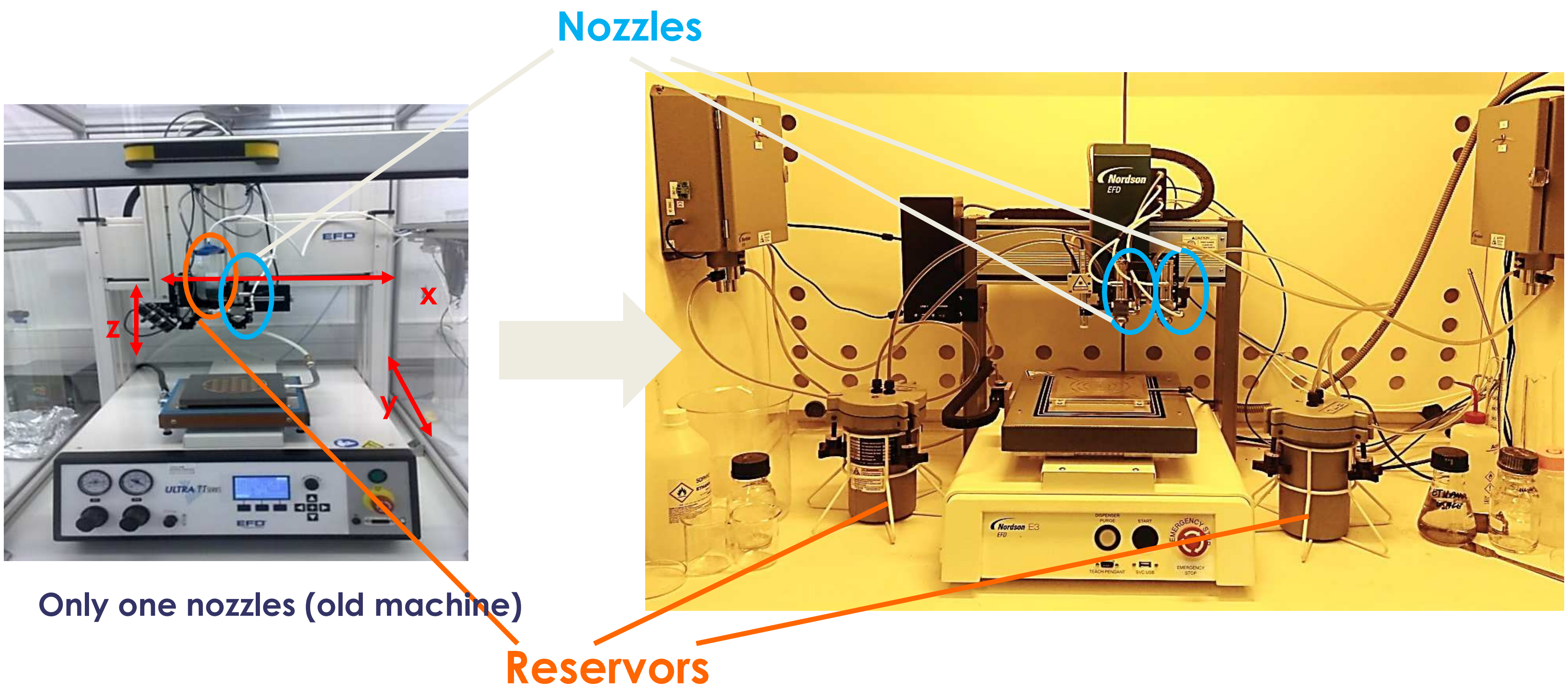
Drawbacks

- NMP based suspensions (toxic and higher boiling temperature than water)
- Stability of the suspensions (weeks?)

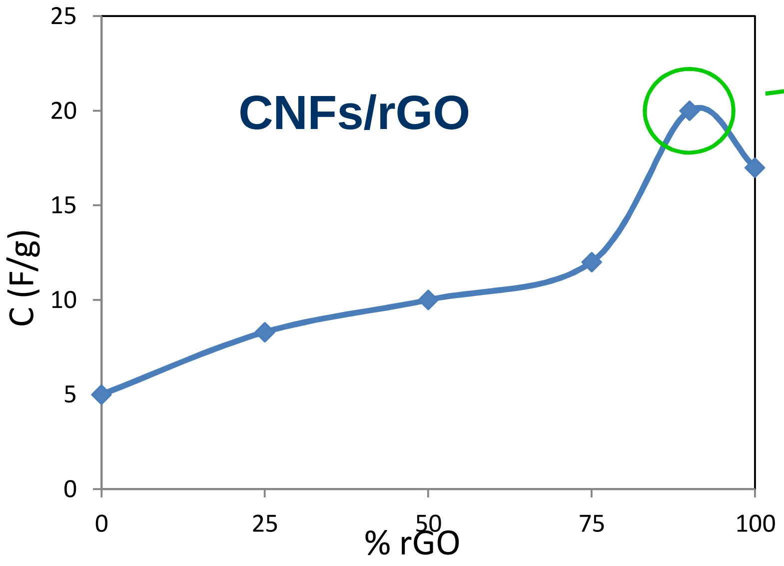
Video 1

- Last developments on nanostructuration

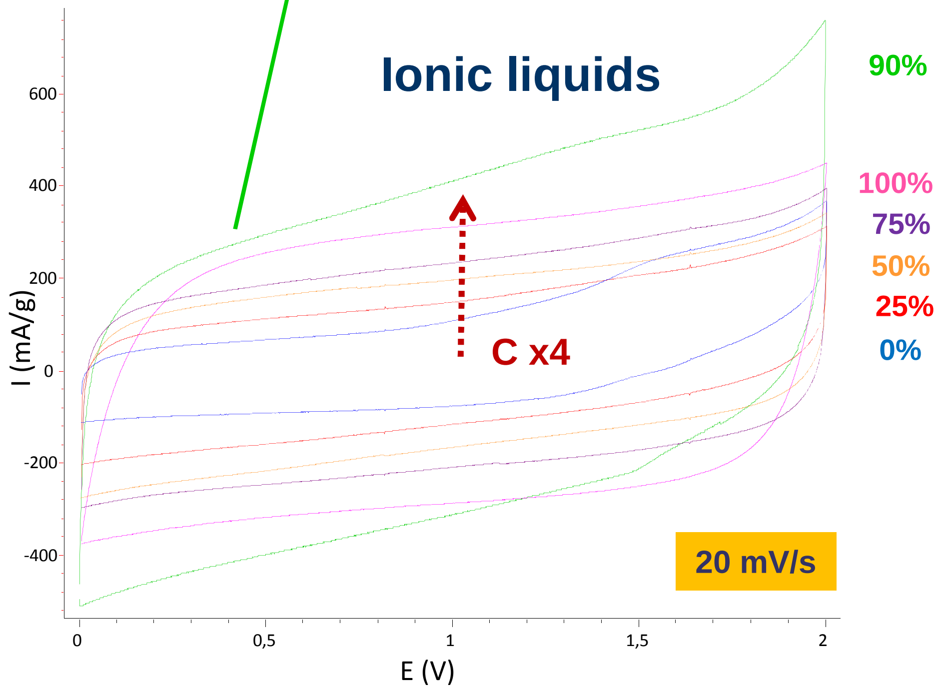
New mchine with two nozzles acquired by Thales (form January 2017)



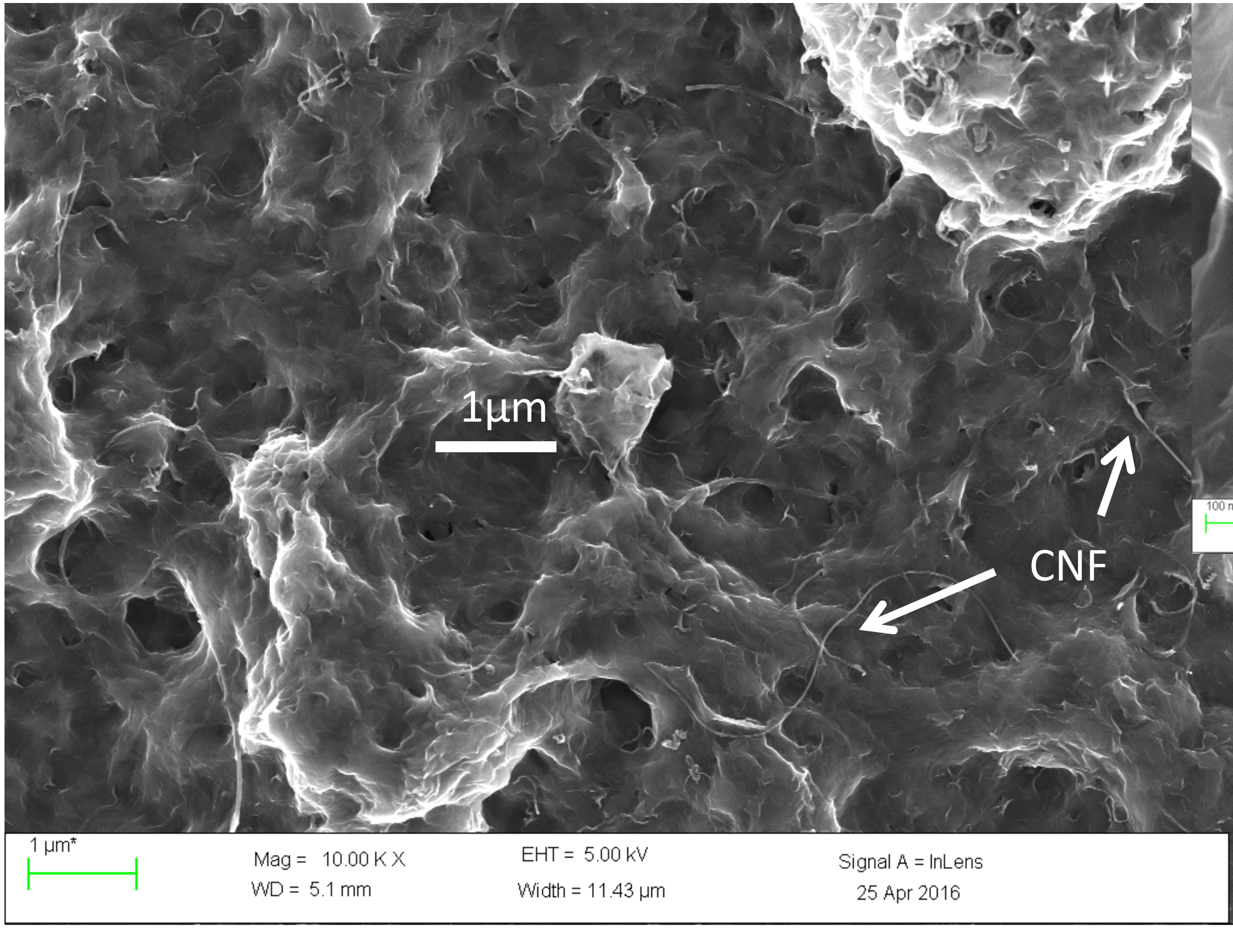
Utilisation of specific ionic liquids : large temperature interval for avionics (-55°C +105°C)

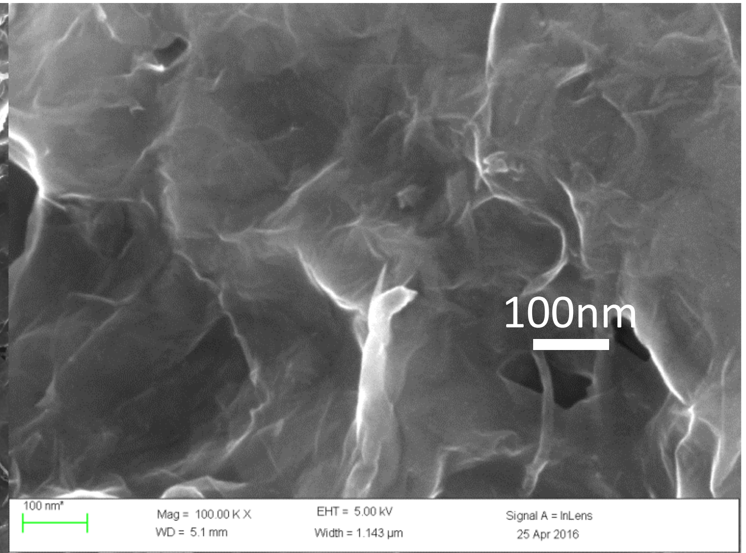


C= 30F/g (whole cell)
P = 40 kW/Kg



Deelopments using different solvents

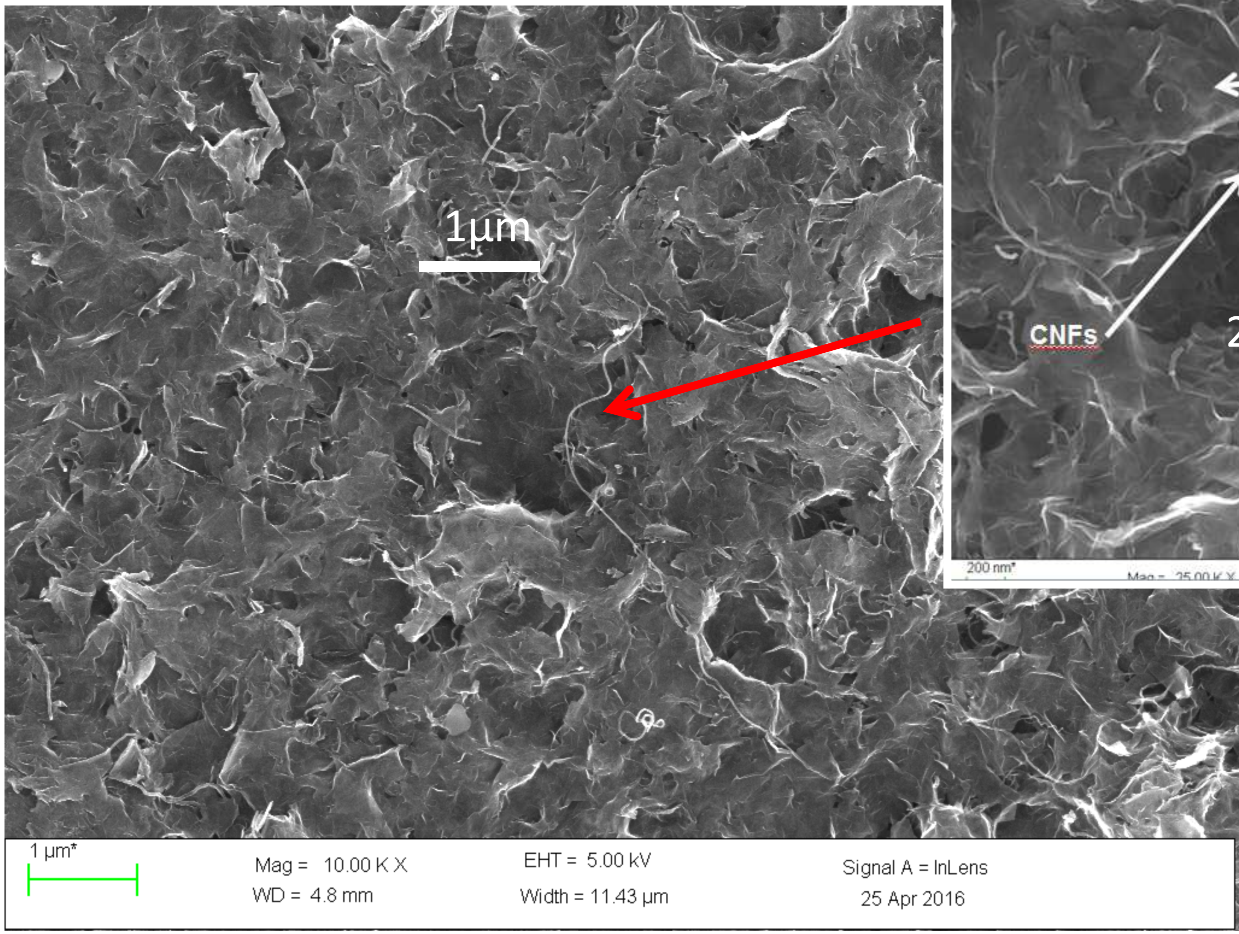


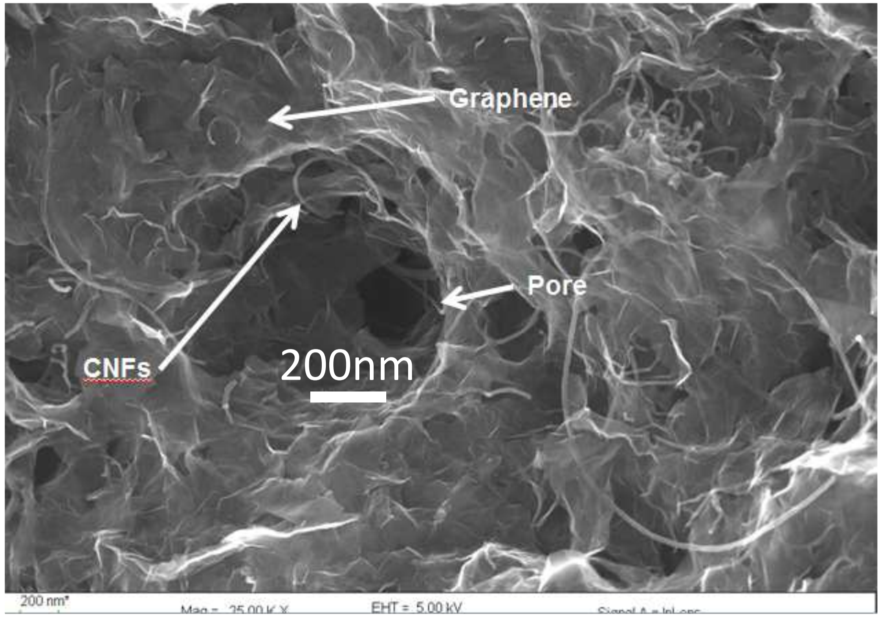


90/10 Cyclohexanone (rGO/CNFs)

The quality of Graphene is quite good considering thta it is transparent. The CNF

Deelopments using different solvents





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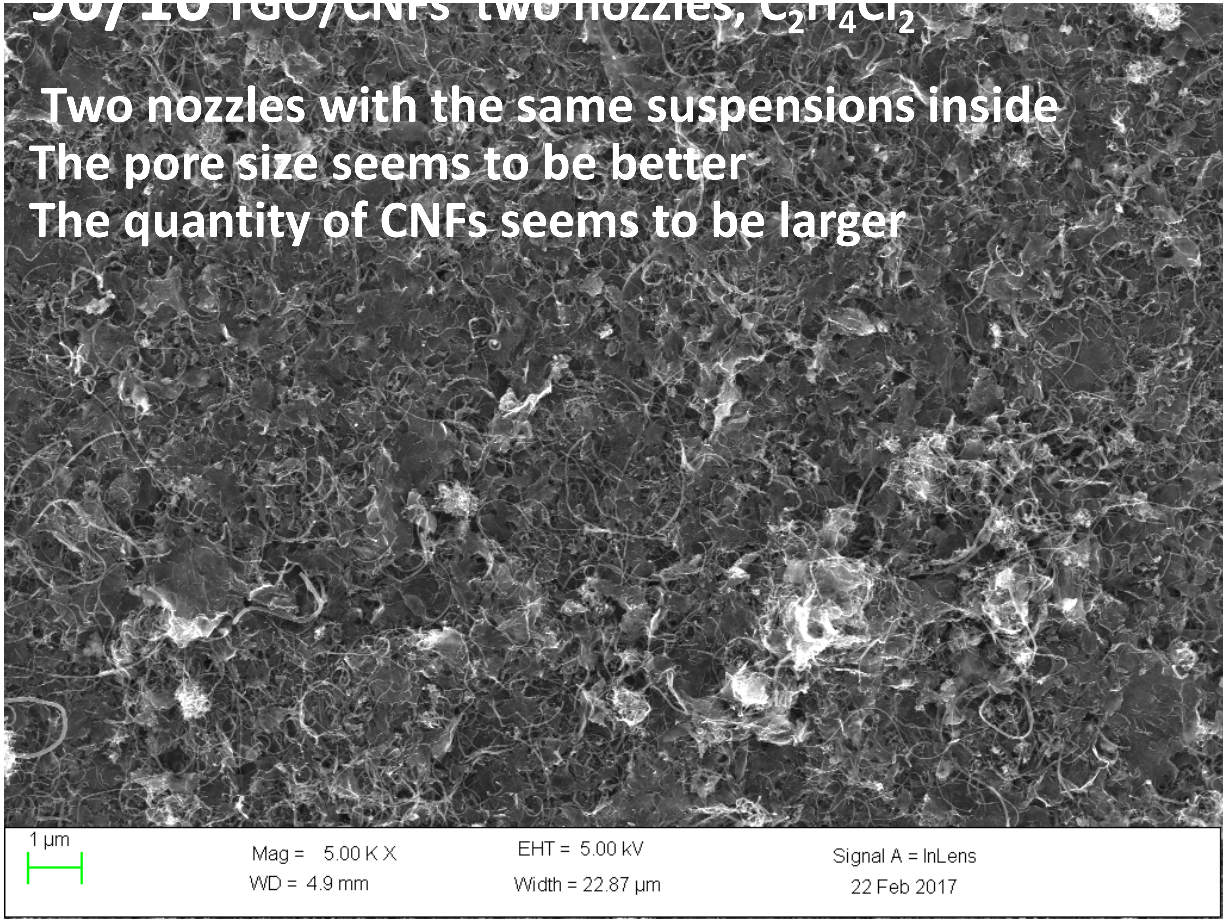
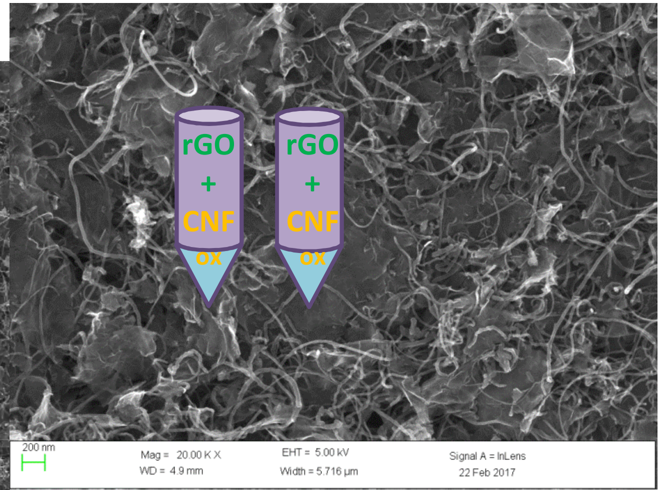
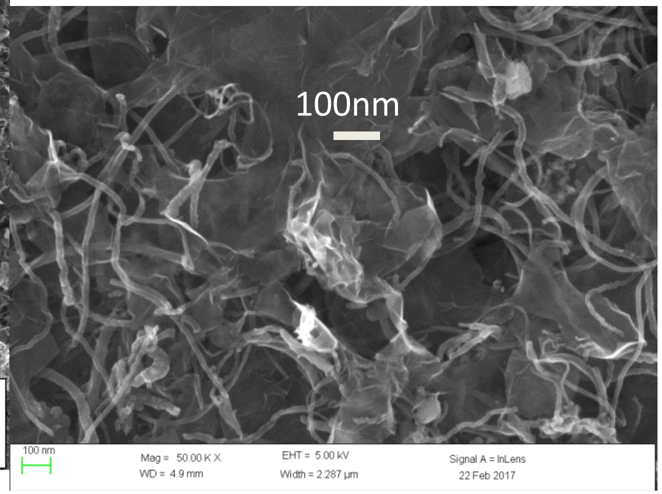
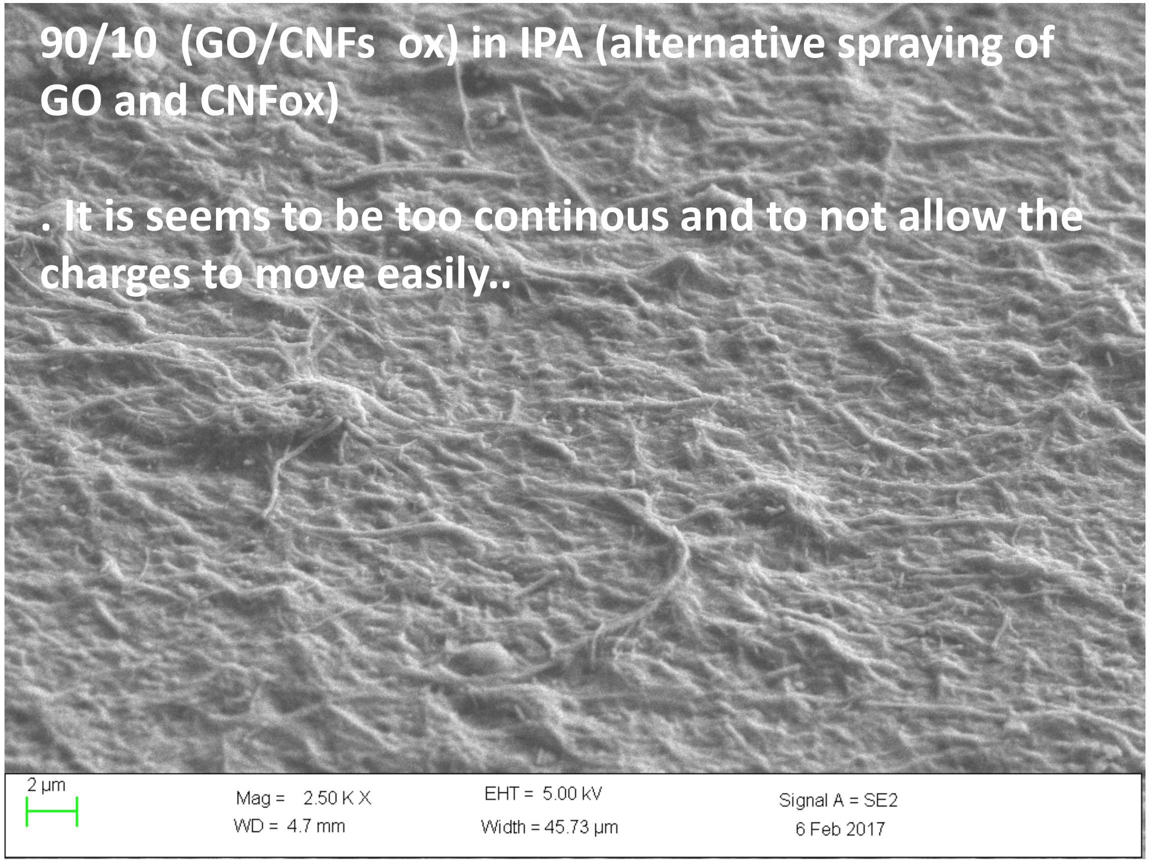
Deelopments using different solvents

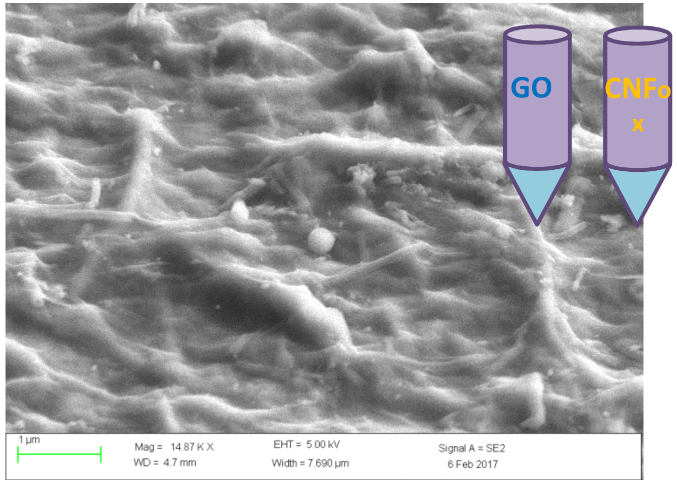


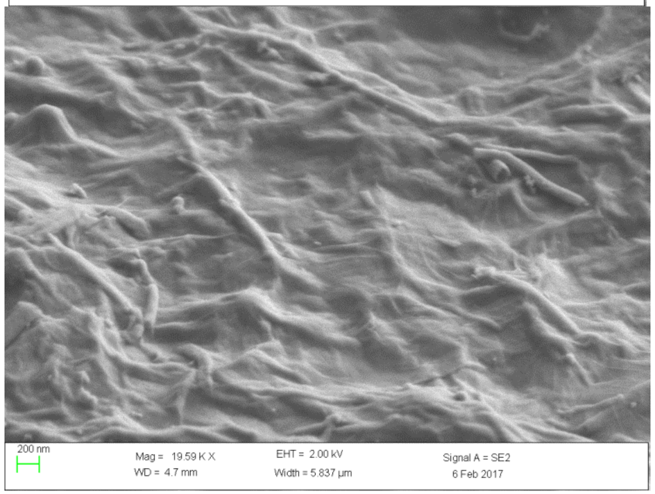




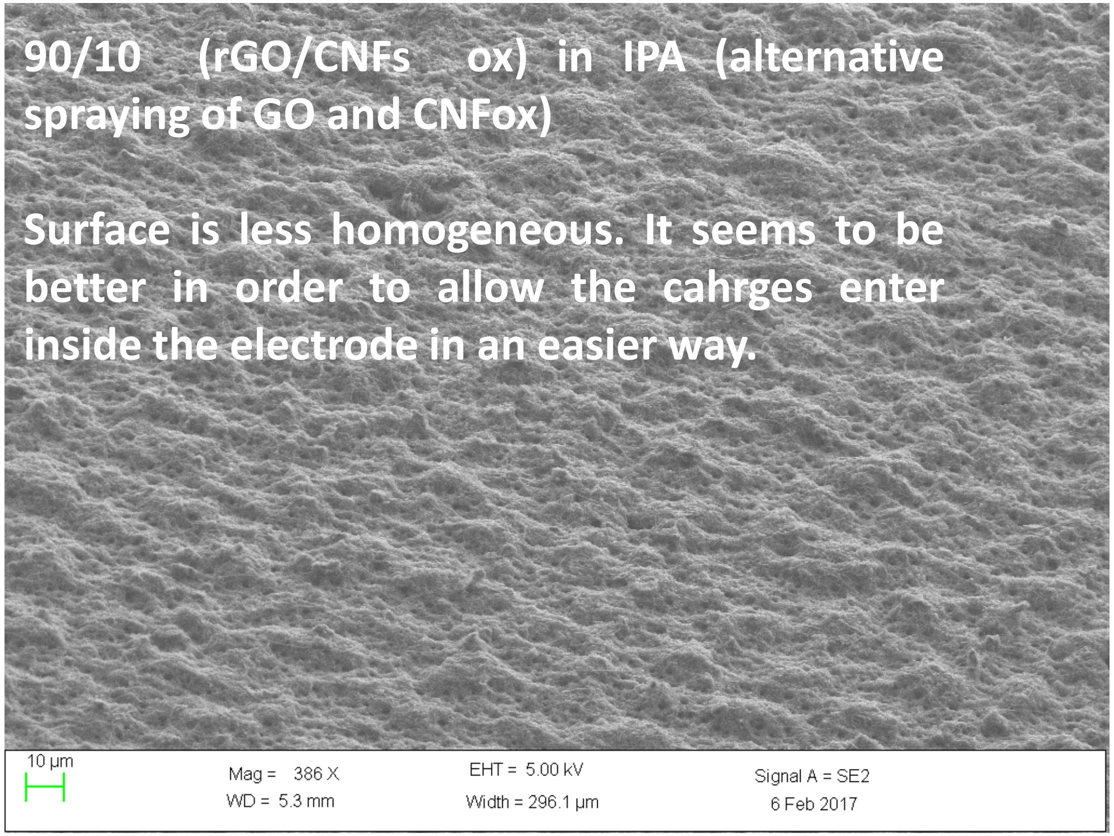
Developments using different solvents

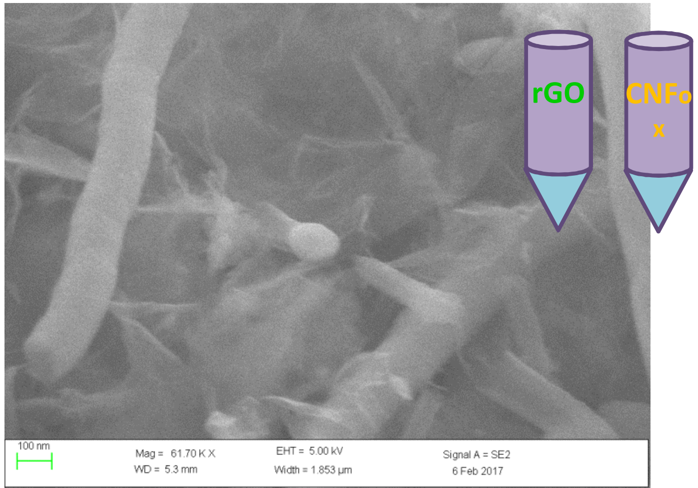


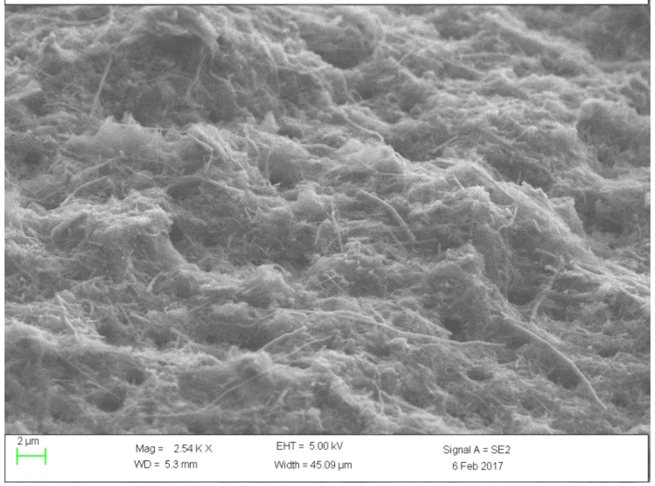




Developments using different solvents



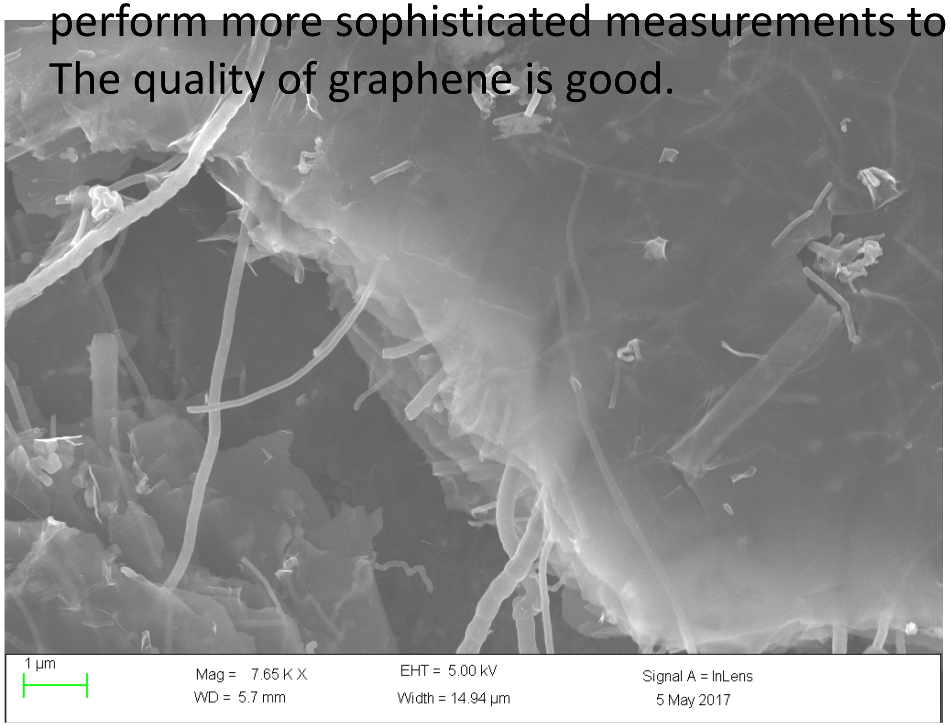


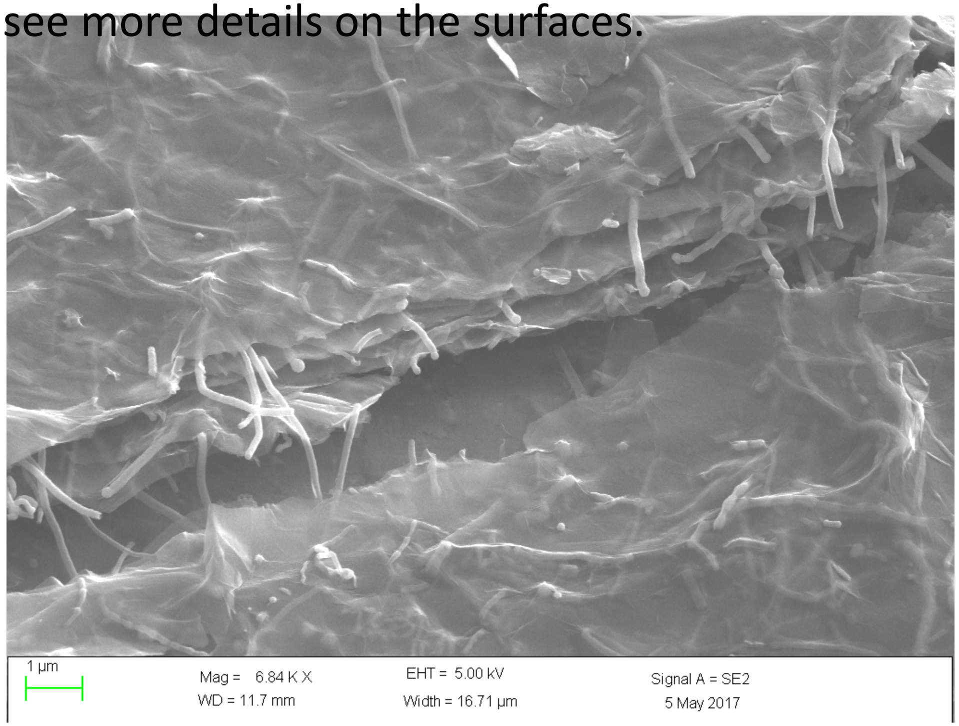


Developments using different solvents

75/25 Cyclohexanone (GO/CNFs ox) (alternative spraying of GO and CNFox)

The intercalation seems very good. The only issue is about the graphene layer. It is seems to be too continous and to not allow the charges to move easily. We have to perform more sophisticated measurements to see more details on the surfaces.
The quality of graphene is good.





Fundings

GRAPHENE FLAGSHIP

Work Package 9: Energy

Work Package Leader - Dr. Etienne Quesnel, CEA French Alternative Energies and Atomic Energy Commission, France
Work Package Leader - Dr. Vittorio Pellegrini, Italian Institute of Technology, IIT graphene labs, Italy

The FIBRALSPEC project is supported by the European Commission under the 7th Framework Programme and will run for 48 months from January 2014 to December 2017. Project coordinator: Costas A. Charitidis

SEVENTH FRAMEWORK PROGRAMME

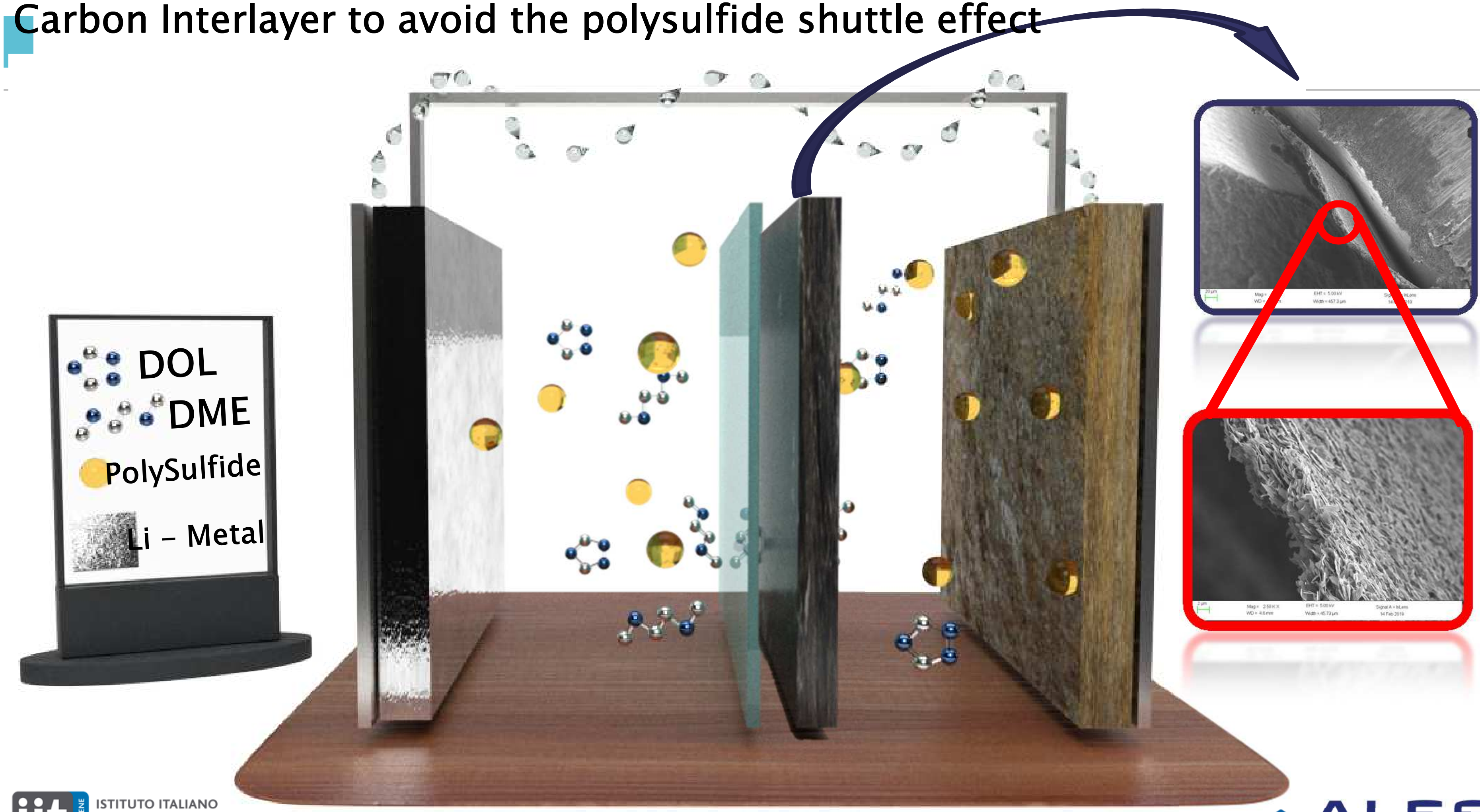
THALES

Outline

- Gas sensors application
- Supercapacitors based on mixtures of Graphene/CNTs (Energy)
- Graphene oxide based memories
- Electro Magnetic Shielding
- Li-S batteries

THALES

Carbon Interlayer to avoid the polysulfide shuttle effect



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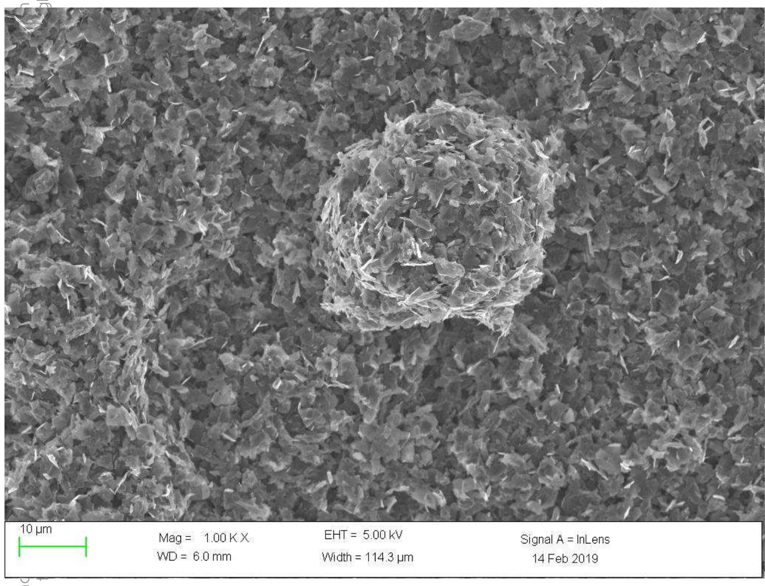
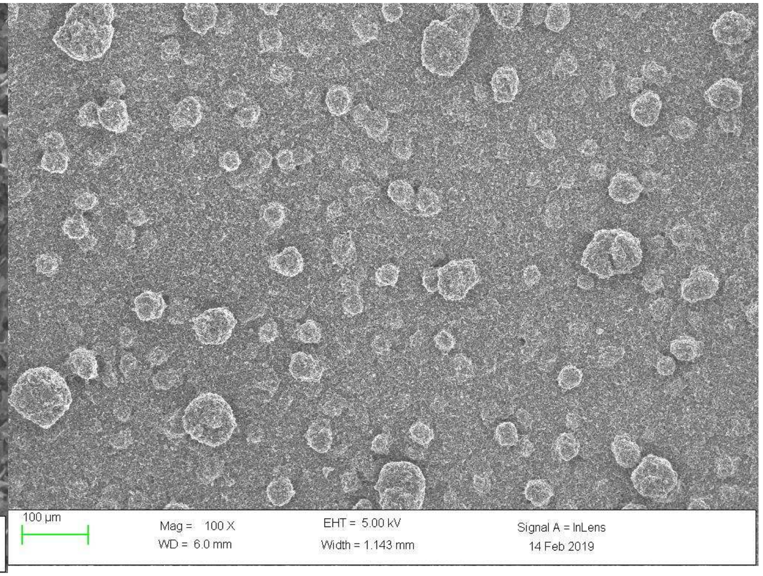
GRAPHENE FLAGSHIP

ALES

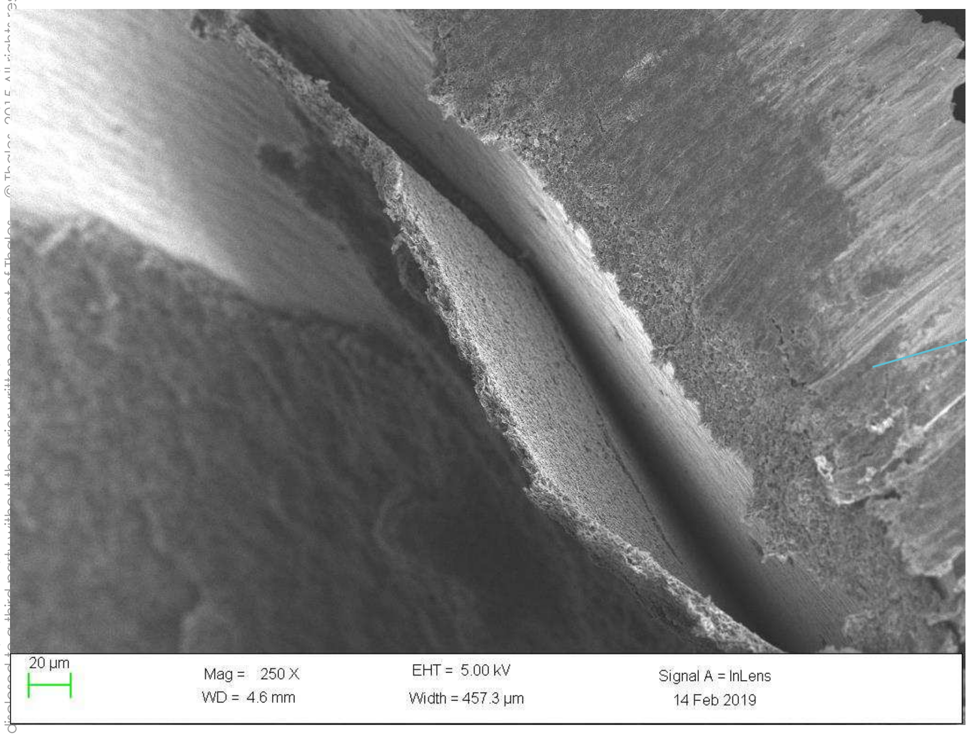
Li-Si batteries using spary-gun deposition in collaboration with IIT

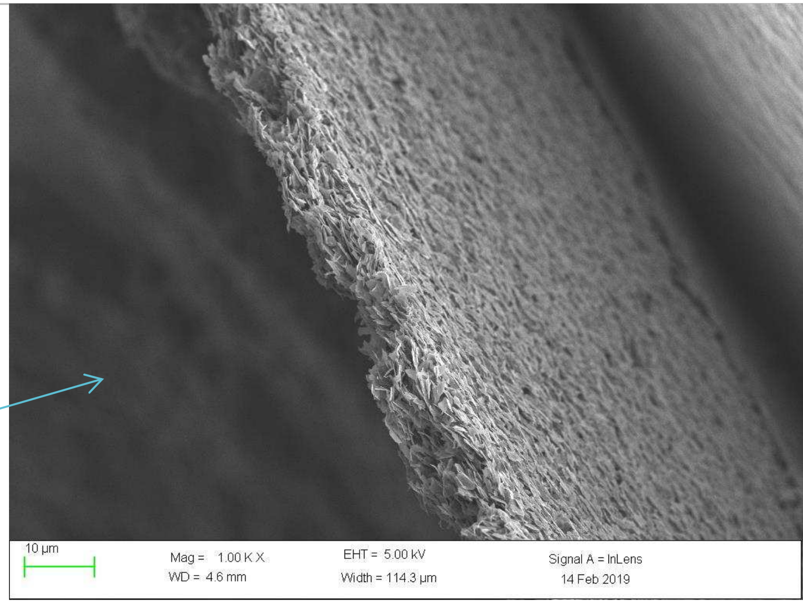




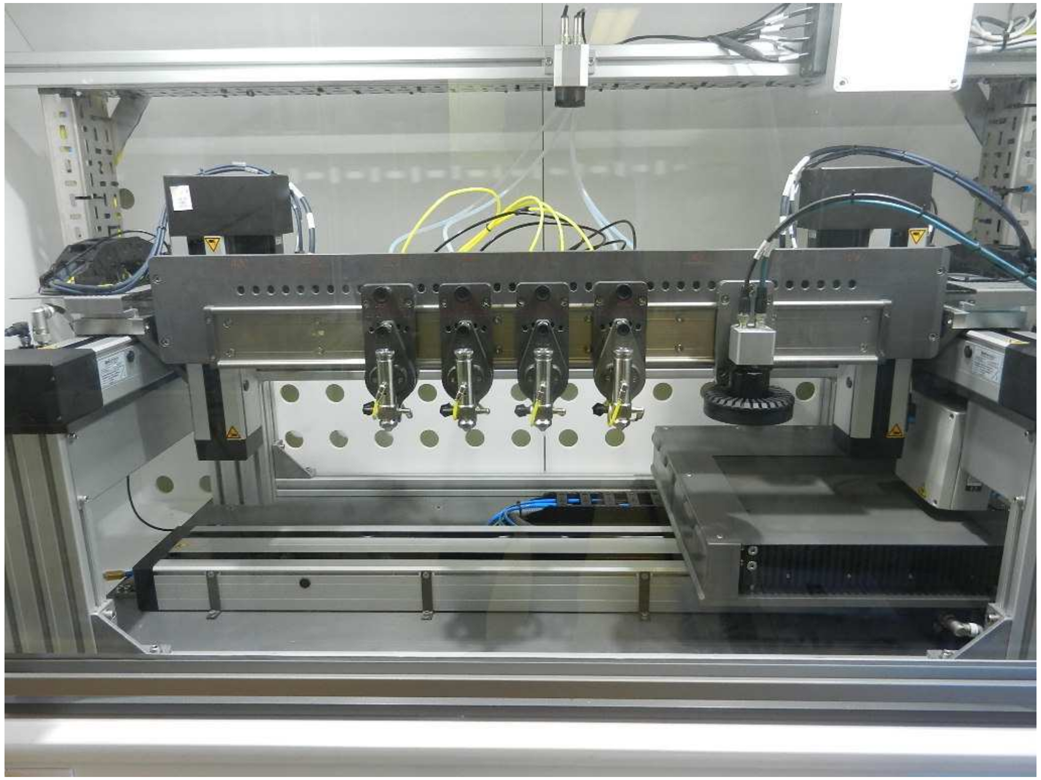
Lorenzo Carbone
IIT

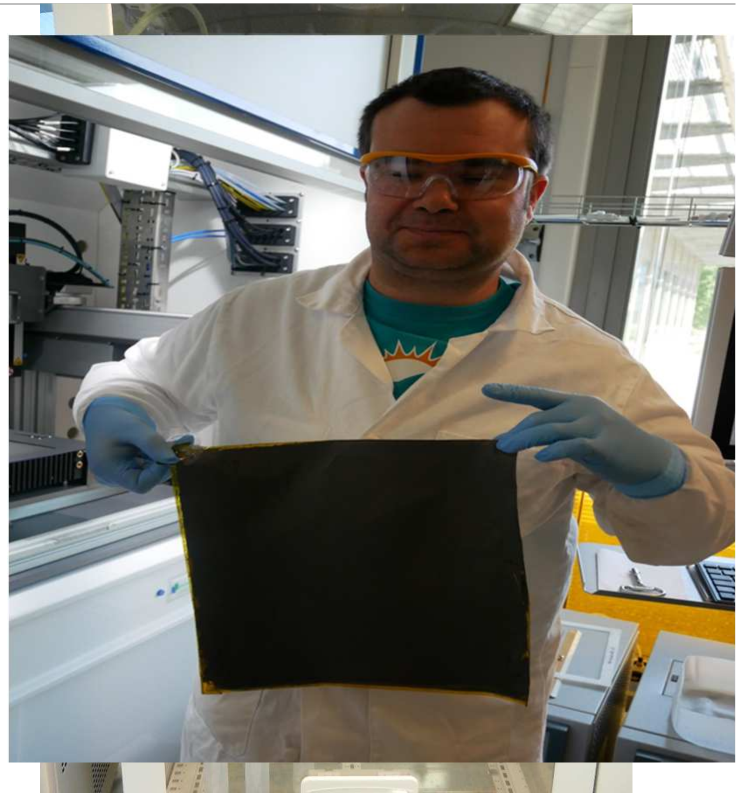
THALES


20 µm
Mag = 250 X
WD = 4.6 mm
EHT = 5.00 kV
Width = 457.3 µm
Signal A = InLens
14 Feb 2019


10 µm
Mag = 1.00 K X
WD = 4.6 mm
EHT = 5.00 kV
Width = 114.3 µm
Signal A = InLens
14 Feb 2019

New Machine funded by the Graphene Flagship





GRAPHENE FLAGSHIP

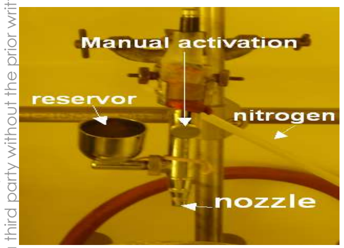
M-SOLV

Conclusions

THALES

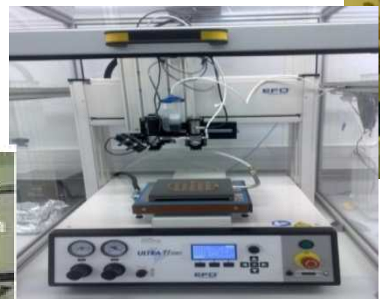
Spray-gun deposition method evolution

Deposition method evolution

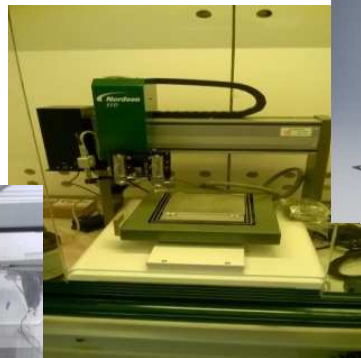


2006
Manual Air- brush

2007
Semi-automatic Air- brush



2008
Spray-gun robot with one nozzle



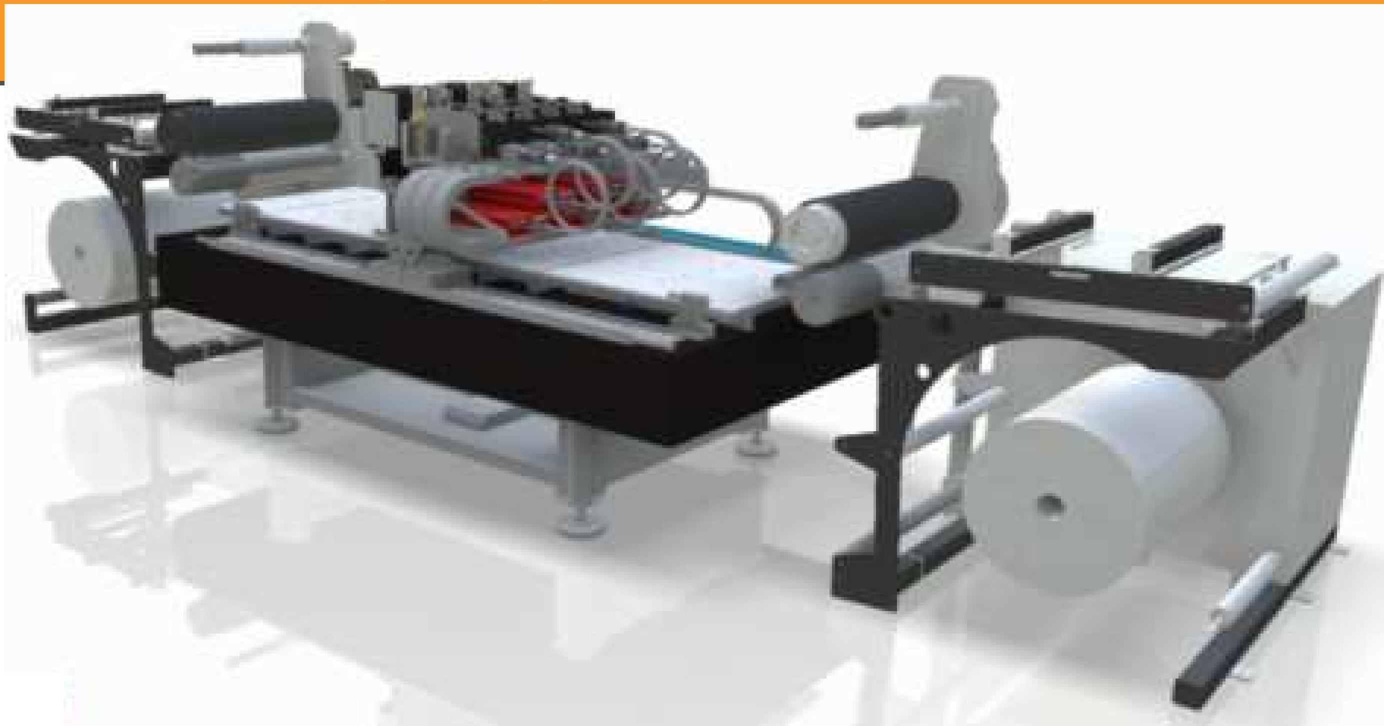
2016
Spray-gun robot with two nozzles

2017
Spray-gun robot with four nozzles pre-industrialized (M-Solv)

SMALL SURFACES
MANUAL SYSTEM

LARGE SURFACES
COMPLETELY AUTOMATISED SYSTEM
(pre-industrial prototype)

Roll to Roll Core 2 (2020) Pilot Production line using Spray



GRAPHENE FLAGSHIP

THALES

- **IIT for exfoliated Graphene**

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GRAPHENE FLAGSHIP

- **NTUA for carbon nanofibers**

National Technical University of Athens

www.Fibralspec.net

MODCOMP

SmartFAN

- **Graphenea for GO in water**

Graphenea

GRAPHENE FLAGSHIP

- **M-Solv for collaboration in developing the new machine**

M-SOLV Limited

GRAPHENE FLAGSHIP

THALES

Projet en cours qui concernent le spray

ICT FET Flagship Core 2 2018-2020

H2020 NMBP 2018-2022

H2020 NMBP 2015-2019

ANR France-Luxembourg 2019-2022

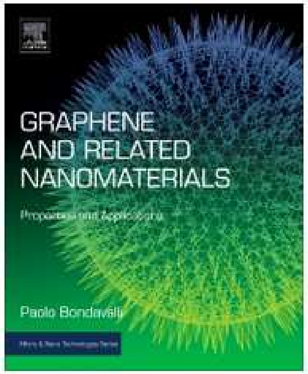
THALES


IDTechEx Show!
EMERGING TECHNOLOGIES UNLEASHED
11 - 12 April 2018 | Estrel Convention Center, Berlin, Germany
Connecting Emerging Technologies With Global Brands
GRAPHENE FLAGSHIP
GRAPHENE WEEK 2018
10-14 September, San Sebastian, Spain
2D MATERIALS FOR ENVIRONMENT AND ENERGY APPLICATIONS
GOTHENBURG (HINDÅS), SWEDEN, 1-6 JULY 2018
GRAPHENE STUDY
Graphene 2018
June 26 - 29
Dresden (Germany)
MOBILE WORLD CONGRESS
BARCELONA 26 FEB-1 MAR 2018
Creating a Better Future
#MWC18
IMAGINENANO 2018
SCIENCE • INDUSTRY • SOCIETY
Bringing together Nanoscience & Nanotechnology
2018 MRS FALL MEETING & EXHIBIT
November 25–30, 2018 | Boston, Massachusetts
PRINTED ELECTRONICS SUMMIT
BARCELONA, SPAIN
14–15 JUNE | 2018
2018 MRS SPRING MEETING & EXHIBIT
April 2–6, 2018 | Phoenix, Arizona

THALES

- **High-power graphene-carbon nanotube hybrid supercapacitors**. ChemNanoMat. Accepted Author Manuscript. Ansaldo, A., **Bondavalli**, P., Bellani, S., Del Rio Castillo, A. E., Prato, M., Pellegrini, V., Pognon, G. and Bonaccorso, F. (2017), doi:10.1002/cnma.201700093
- **Graphene-based technologies for energy applications, challenges and perspectives,** Etienne Quesnel, Frédéric Roux, **Paolo Bondavalli** et al., *2D Materials 08/2015; 2(3):030204*.
- **Supercapacitor electrode based on mixtures of graphite and carbon nanotubes deposited using a new dynamic air-brush deposition technique**, **P Bondavalli,** C.Delfaure, P.Legagneux, D.Pribat JECS 160 (4) A1-A6, **2013**
- **Non-faradic carbon nanotubes based supercapacitors : state of the art**, **P.Bondavalli,** D.Pribat, C.Delfaure, P.Legagneux, L.Baraton, L.Gorintin, J-P. Schnell, Eur. Phys. J. Appl. Phys. 60,10401, **2012**
- **Graphene based supercapacitors fabricated using a new dynamic spray-gun deposition technique**, **P. Bondavalli** and G. Pognon, 2015 IEEE 15th International Conference on Nanotechnology (IEEE-NANO), Rome, 2015, pp. 564-567.

- Graphene and Related Nanomaterials 1st Edition, Properties and Applications, **Paolo Bondavalli,** Hardcover ISBN: 9780323481014, Elsevier, Published Date: 20th October September 2017



Graphene and Related Nanomaterials

1st Edition

Properties and Applications

Authors: Paolo Bondavalli

- WO/2016/124756 (A1) - **Method of depositing oxidized carbon-based microparticles and nanoparticles,** Pognon Grégory [fr]; Bondavalli Paolo [fr]; Galindo Christophe [fr]

- WO/2014/166952 (A1) **- Electrode-gel electrolyte assembly comprising a porous carbon material and obtained by radical polymerisation,** Le Barny, Pierre; (FR). Divay, Laurent; (FR).Galindo, Christophe; (FR)

- FR2988900 (A1) **- Electrode pour supercondensateur**, Christophe Galindo, Laurent Divay, Le Barny Pierre

- FR2989215 (A1) - **Electrode hybride pour supercondensateur**, , Le Barny Pierre , Laurent Divay, Christophe Galindo

- FR2989821 (A1) - Electrode hybride nanotubes de carbone-carbonne mesoporeux pour supercondensateur, Le Barny Pierre , Laurent Divay, Christophe Galindo
- FR3011671 (A1) - **Collecteur de courant pour supercapacite**, Bondavalli Paolo [fr]; Legagneux Pierre [fr]; Galindo Christophe

- WO2012049428 (A2) - **Method for depositing nanoparticles on a surface and corresponding nanoparticle depositing appliance**, Bondavalli Paolo [fr]; Gorintin Louis [fr]; Legagneux Pierre [fr]; Ponard Pascal [fr]

- FR2976118 (A1) - **Method for manufacturing collector-electrode assembly that is utilized in supercapacitor, involves forming collector and electrode by spraying suspension comprising nano/microparticles suspended in liquid in substrate,** Bondavalli Paolo [fr]; Schnell Jean Philippe [fr]; Legagneux Pierre [fr]; Gorintin Louis [fr]

- EP2769395 (A1) - **Collector-electrode assembly which can be integrated into an electrical energy storage device,** Legagneux Pierre [fr]; Bergonzo Philippe [fr]; Bondavalli Paolo [fr]; Mazellier Jean-Paul [fr]; Scorsone Emmanuel [fr]

Thank you for your attention!!!