# From Carbon nanotubes to Graphene

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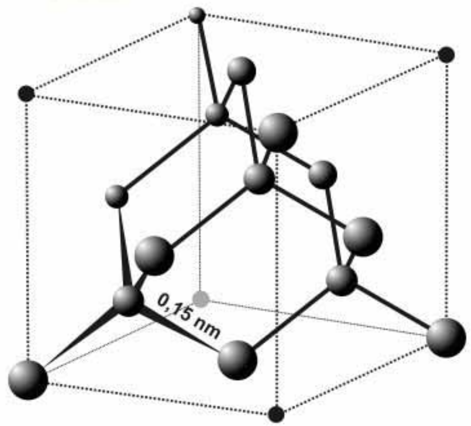
*Istituto Italiano di Tecnologia, Graphene Labs, Genova, Italy*

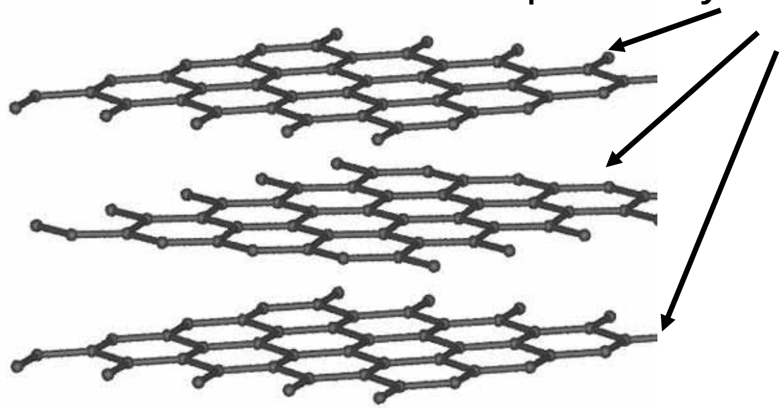
*Bedimensional spa, Genova, Italy*

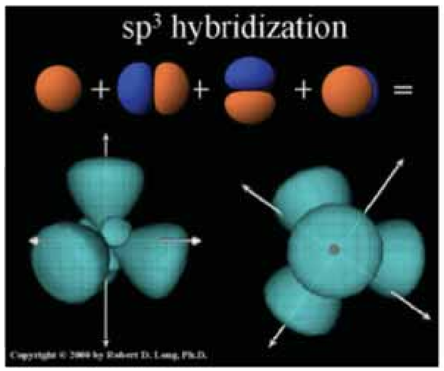
# Common Carbon-based materials

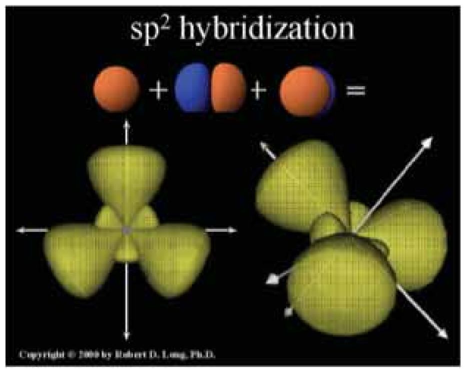
Diamond



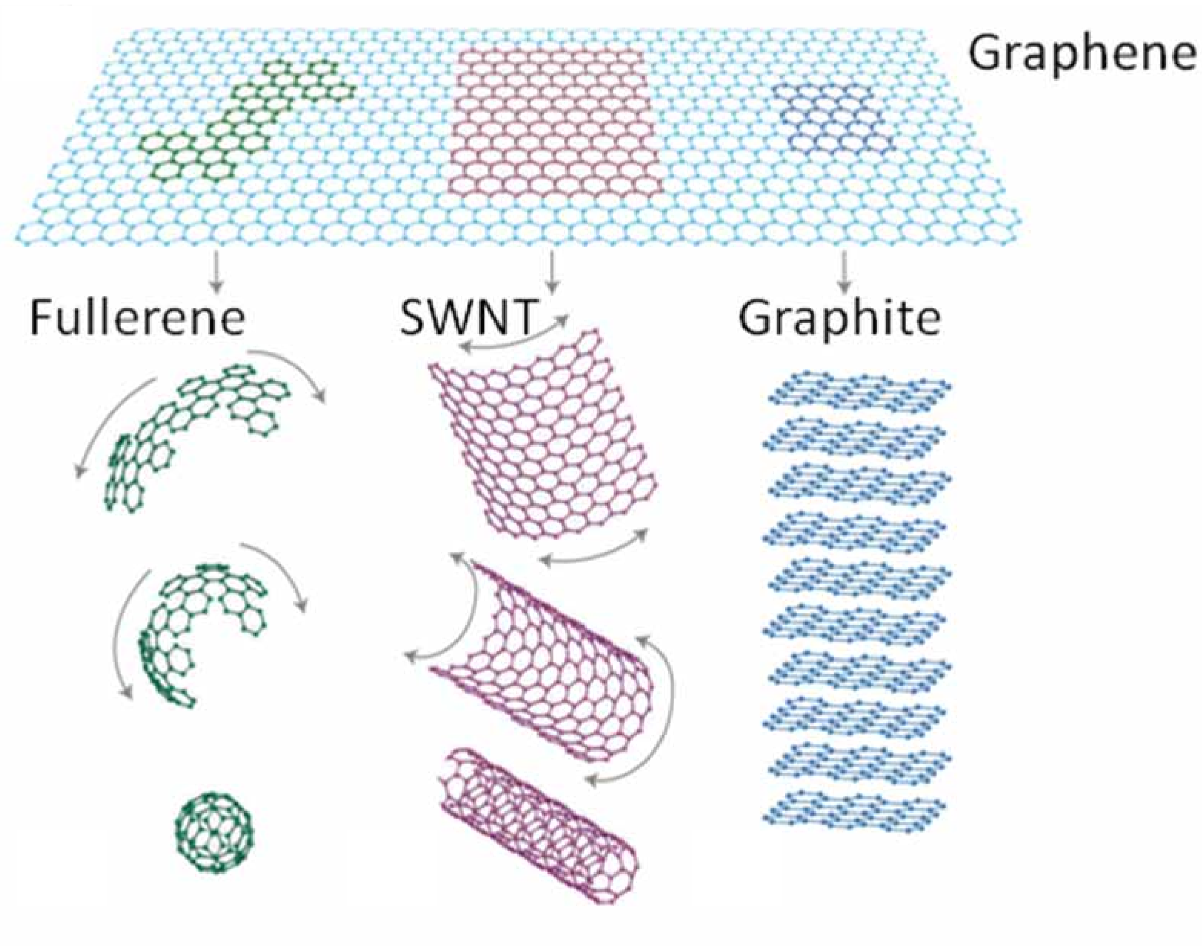

Graphene layers





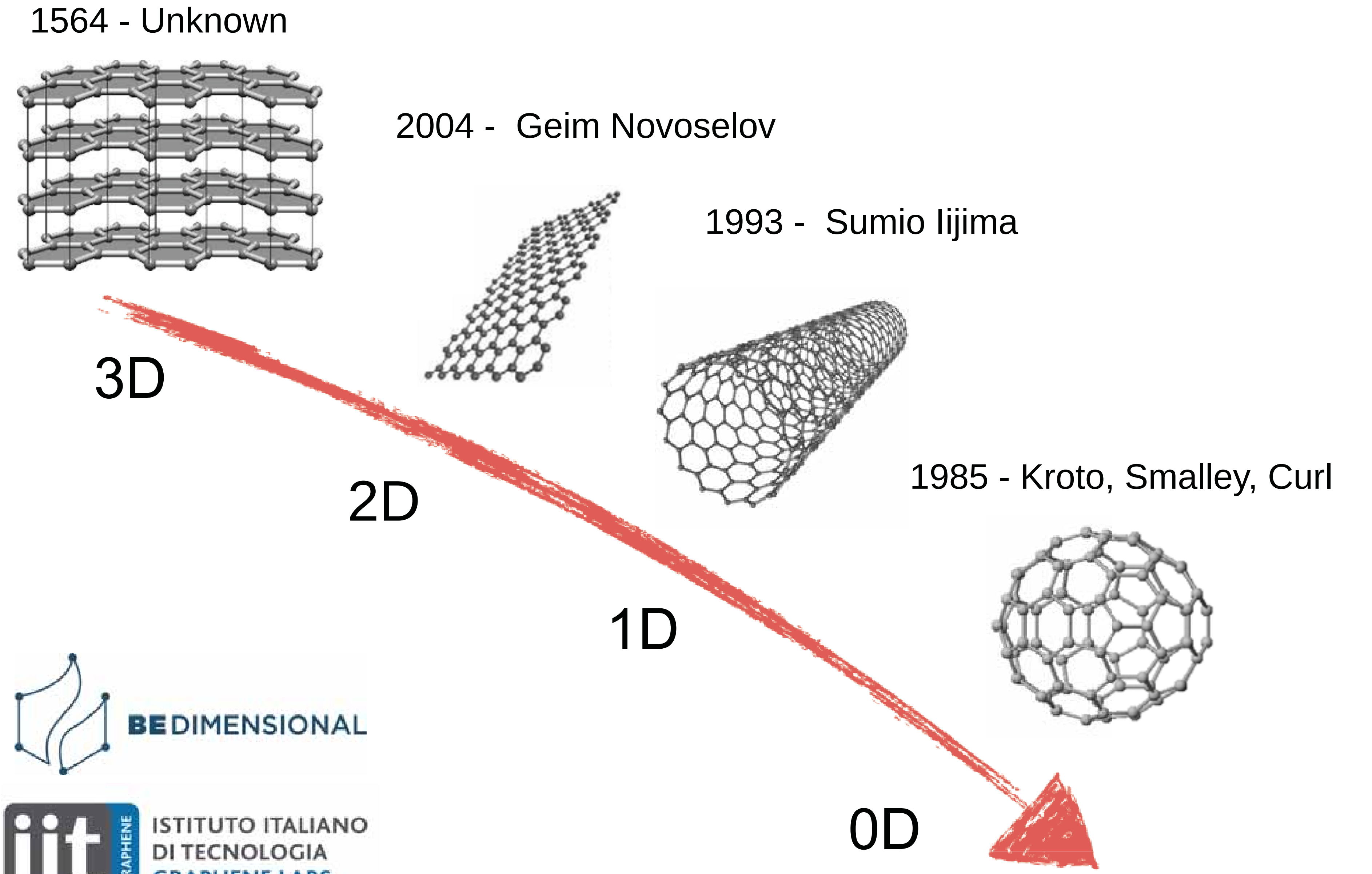



# Carbon nanostructures




Formation of 0-dimensional fullerenes, 1-dimensional CNTs and 3-dimensional graphite from 2-dimensional graphene sheets

# Carbon: a multi-dimensional story

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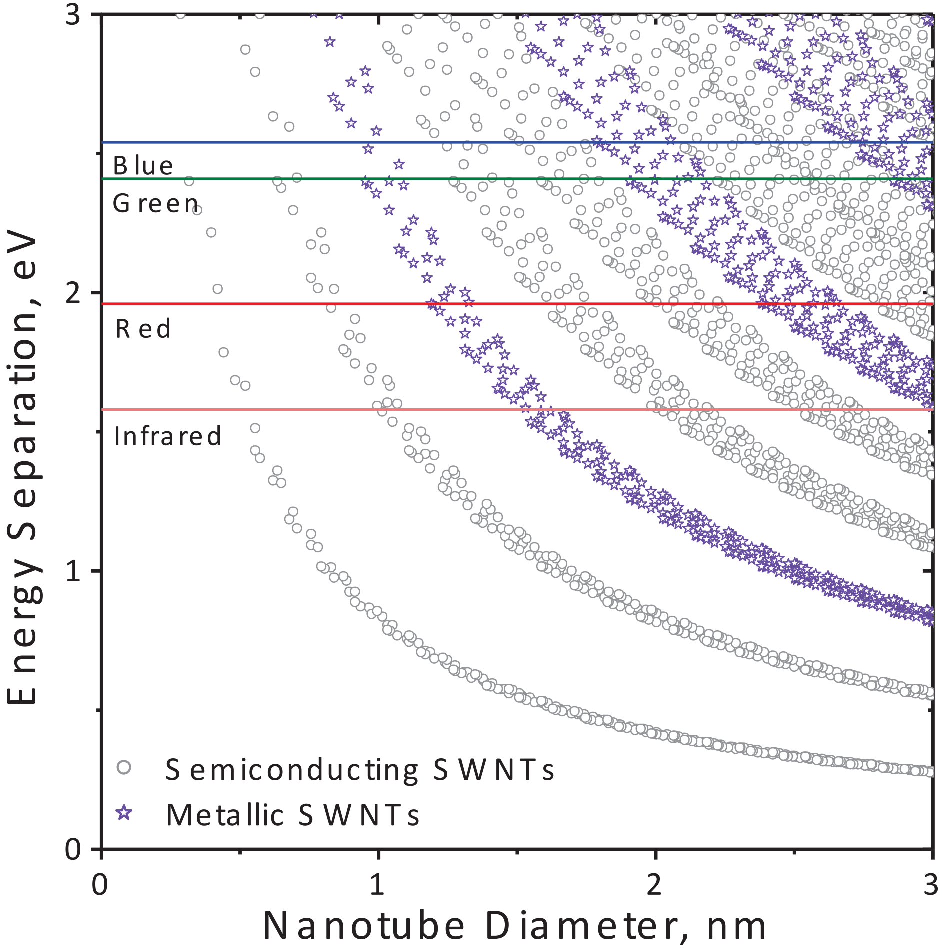
# Carbon nanotubes

The original ‘Kataura plot’ of SWNTs showing relationships between the transition energies and diameters of different SWNTs

Kataura, *Synth. Met. 1999, 103, 2555.*

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# 1947: graphene is conceived

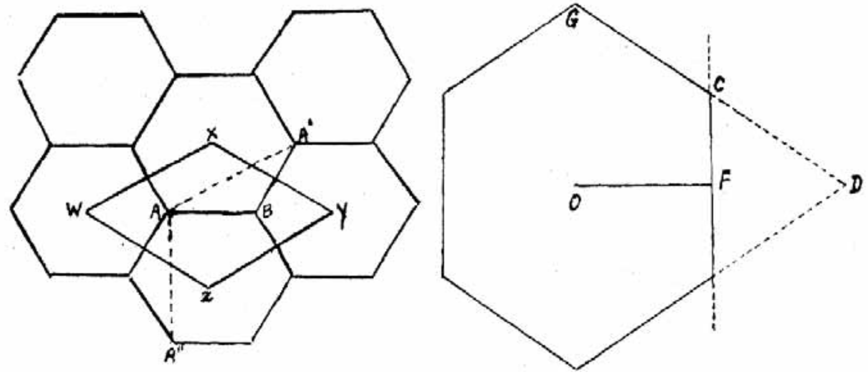
PHYSICAL REVIEW VOLUME 71, NUMBER 9 MAY 1, 1947

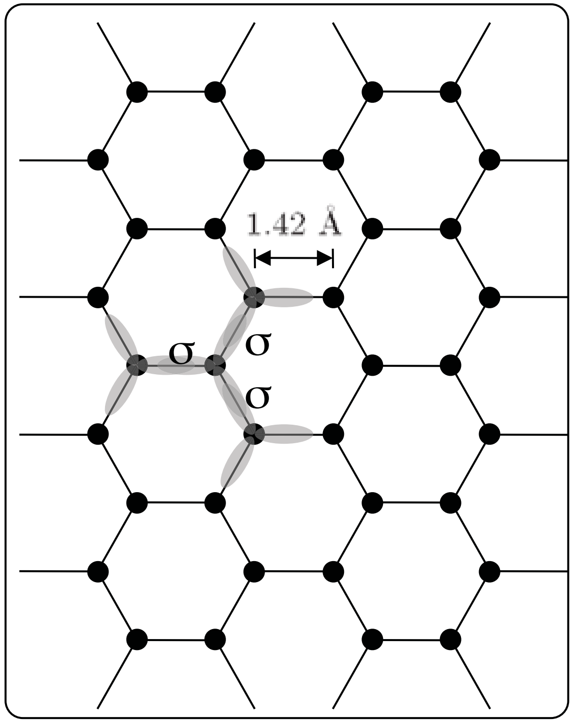
## The Band Theory of Graphite

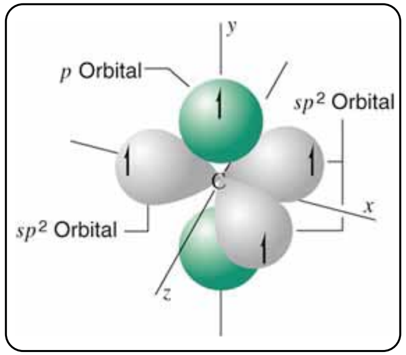
P. R. WALLACE*
*National Research Council of Canada, Chalk River Laboratory, Chalk River, Ontario*

(Received December 19, 1946)

The structure of the electronic energy bands and Brillouin zones for graphite is developed using the "tight binding" approximation. Graphite is found to be a semi-conductor with zero activation energy, i.e., there are no free electrons at zero temperature, but they are created at higher temperatures by excitation to a band contiguous to the highest one which is normally filled. The electrical conductivity is treated with assumptions about the mean free path. It is found to be about 100 times as great parallel to as across crystal planes. A large and anisotropic diamagnetic susceptibility is predicted for the conduction electrons; this is greatest for fields across the layers. The volume optical absorption is accounted for.

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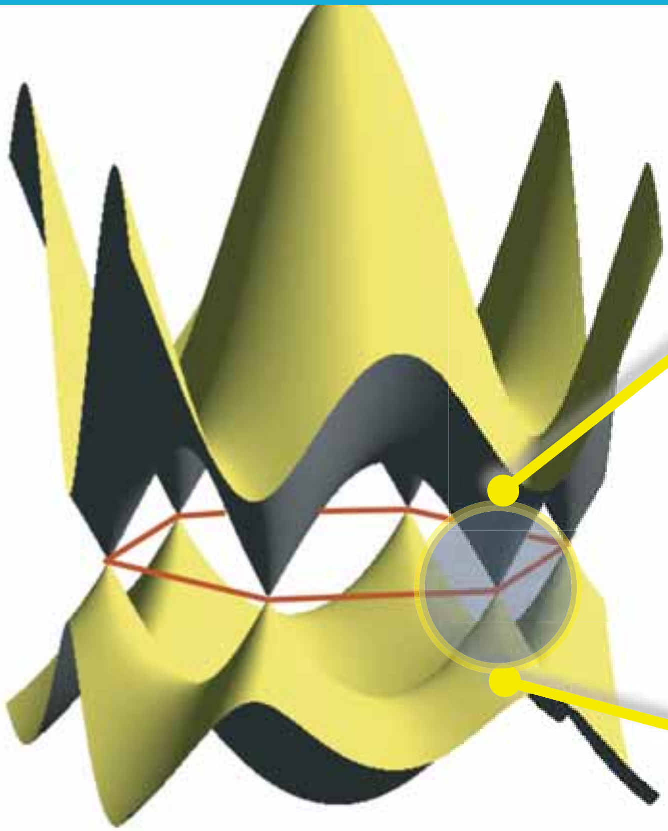
# Massless Dirac Fermions



Dirac cones....

$$E = v_F \sigma \cdot \mathbf{p}$$

$$v_F = 3ta/2 \approx 10^6 m/sec$$

massless Dirac fermions!

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# 2004: graphene is born

The first graphene

One million of a meter

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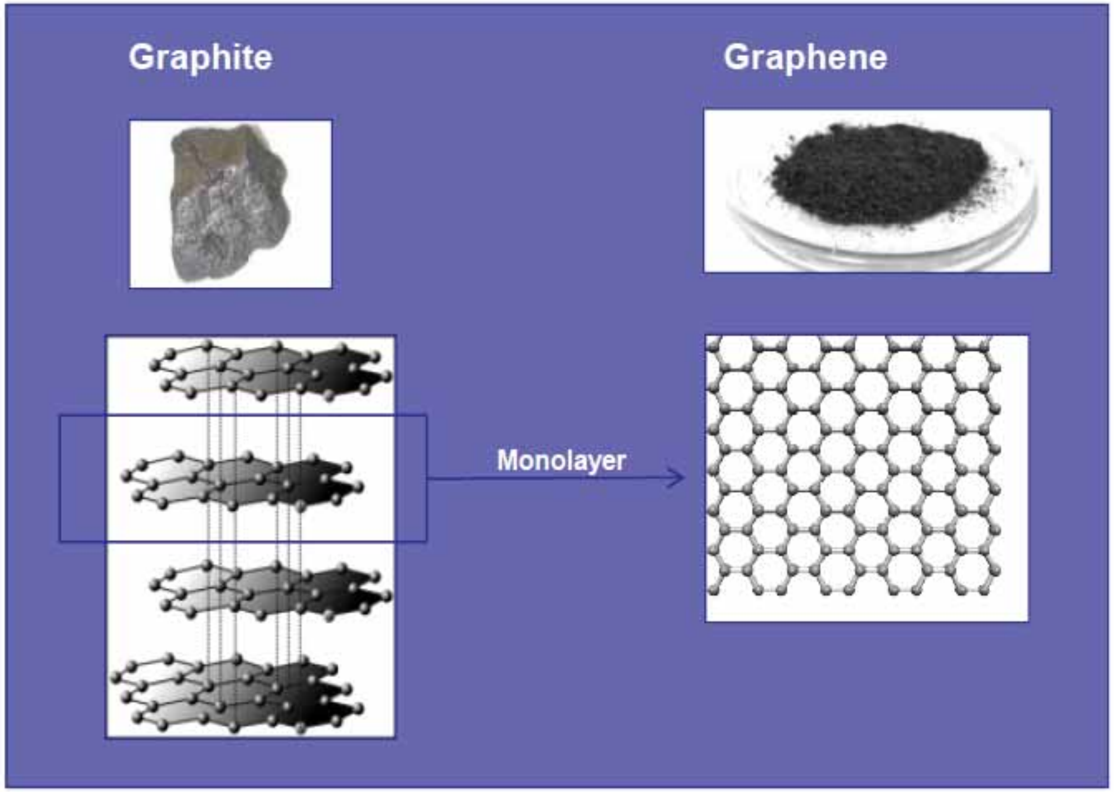
# Graphene superlatives

2010 Nobel Prize in Physics




- **Strongest Material**
  - 100 times tensile strength of steel
    - -Young's modulus = 1 TPa
    - -Tensile strength = 80 GPa
- **Record Electronic Properties**
  - 60% higher conductivity than silver and copper; 1 million times the current density of copper
    - $e^-$ mobility 200 000 $cm^2/V\ s$
    - Bulk resistivity = $10^{-6}\ \Omega cm^{-1}$
- **Highest thermal conductivity**
  - 5 times that of copper, better than diamond
    - Thermal conductivity = 5000 W/mK
- **Highest Surface Area**
  - Superior to active carbon
    - 2630 $m^2g^{-1}$
- **Optically Transparent**
- **Completely Impermeable**

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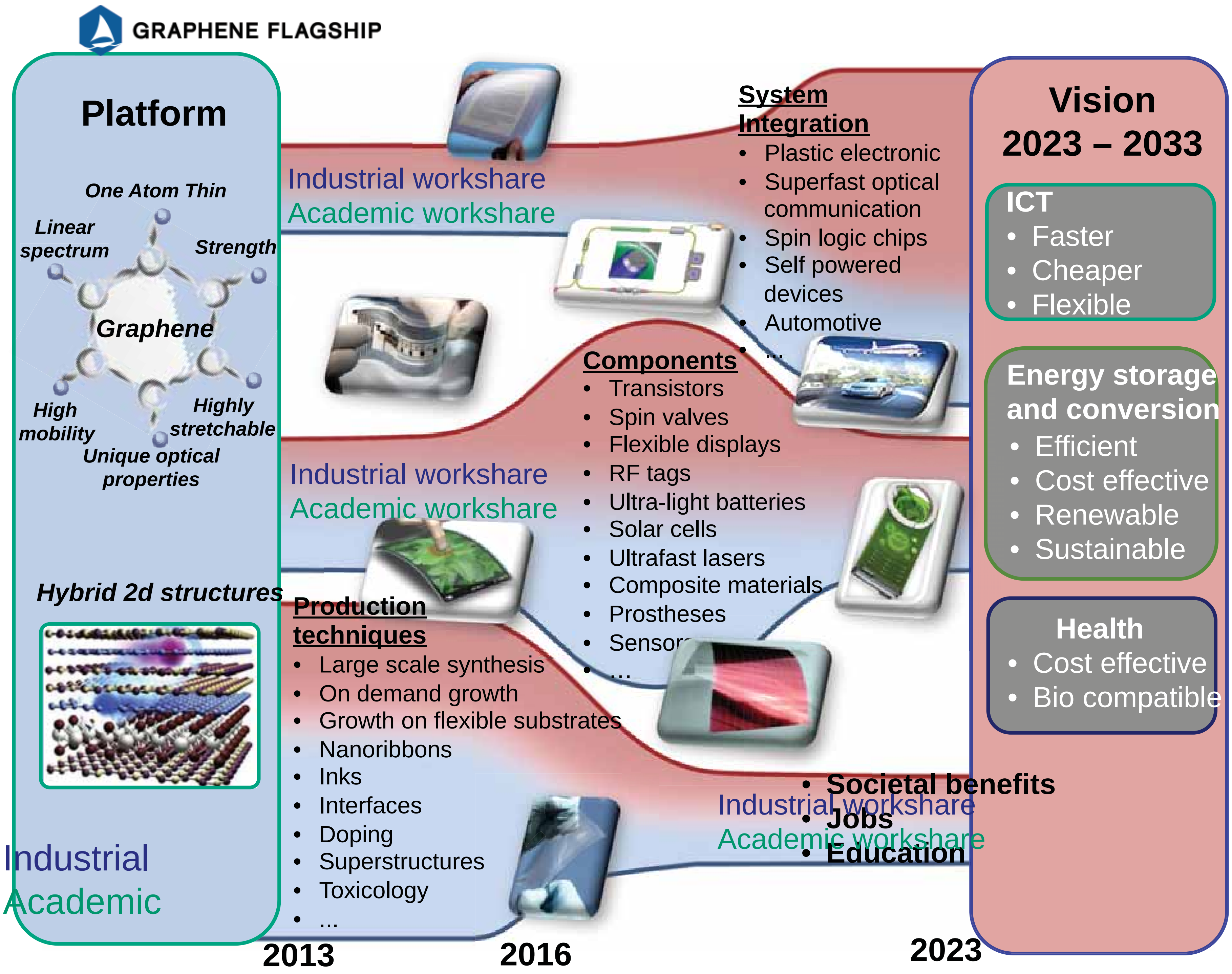
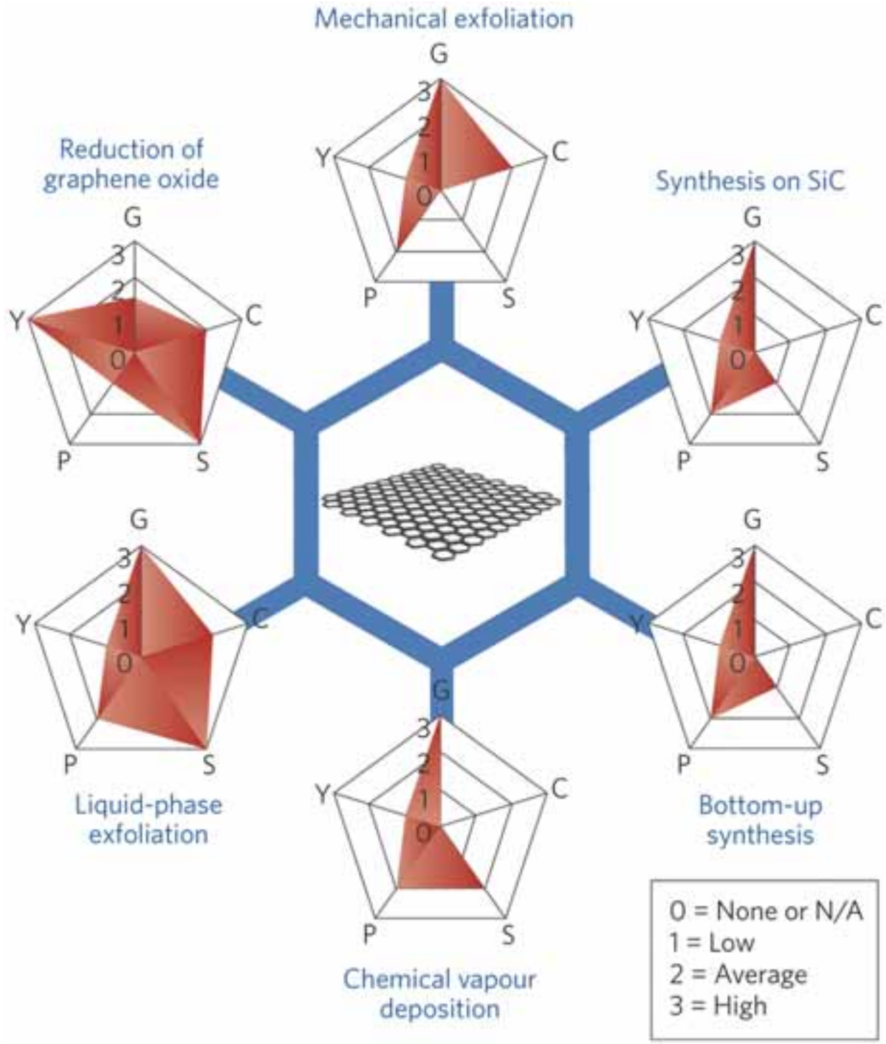
Science and technology roadmap for graphene, related two-dimensional crystals, and hybrid systems *Nanoscale*, **7** 4598 (2015)


GRAPHENE FLAGSHIP
Platform
One Atom Thin
Linear spectrum
Strength
Graphene
High mobility
Highly stretchable
Unique optical properties
Hybrid 2d structures
Industrial
Academic
Industrial workshare
Academic workshare
Industrial workshare
Academic workshare
Production techniques
• Large scale synthesis
• On demand growth
• Growth on flexible substrates
• Nanoribbons
• Inks
• Interfaces
• Doping
• Superstructures
• Toxicology
• ...
Components
• Transistors
• Spin valves
• Flexible displays
• RF tags
• Ultra-light batteries
• Solar cells
• Ultrafast lasers
• Composite materials
• Prostheses
• Sensors
• ...
System Integration
• Plastic electronic
• Superfast optical communication
• Spin logic chips
• Self powered devices
• Automotive
• ...
Industrial workshare
Academic workshare
• Societal benefits
• Jobs
• Education
Vision 2023 – 2033
ICT
• Faster
• Cheaper
• Flexible
Energy storage and conversion
• Efficient
• Cost effective
• Renewable
• Sustainable
Health
• Cost effective
• Bio compatible
2013
2016
2023

# Graphene production




G Graphene quality
C cost aspect
S scalability
P purity
Y yield

R. Raccichini et al., Nature Mat. **14**, 271–279 (2015)

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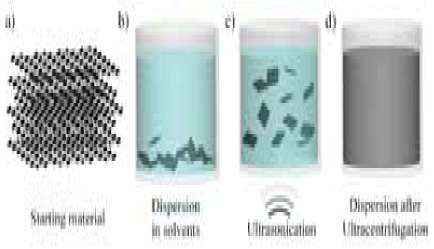
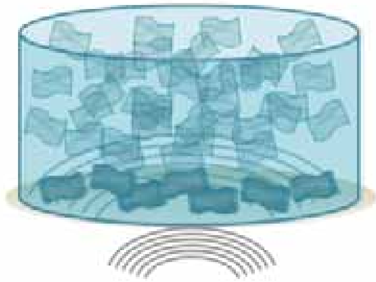
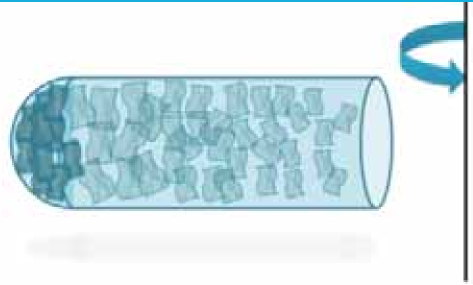
# Liquid phase exfoliation of 2d crystals





**2D bulk materials** **Ultrasonication** **Ultracentrifugation**

Graphene $Bi_2Te_3$ $WSe_2$ $WS_2$ $MoS_2$ BN

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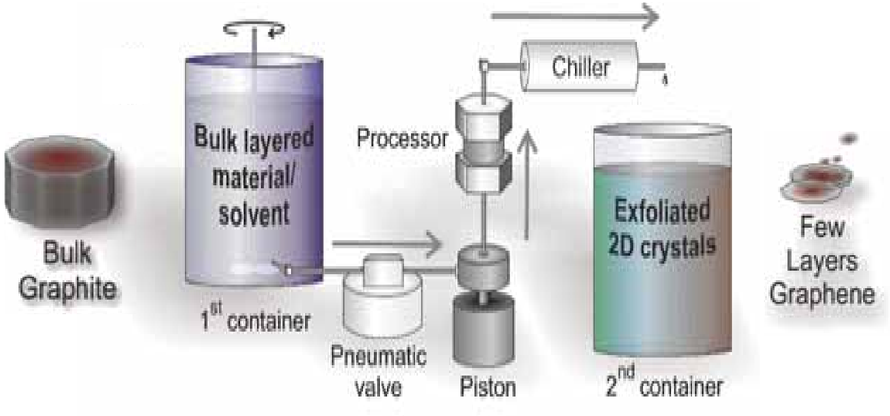
# Graphene production




Surface tension (γ) of the solvent must be similar to the surface free energy of the material

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# Graphene production

Materials Horizons

ROYAL SOCIETY OF CHEMISTRY

COMMUNICATION

View Article Online
View Journal

Check for updates

Cite this: DOI: 10.1039/c8mh00487k

Received 25th April 2018,
Accepted 6th July 2018

DOI: 10.1039/c8mh00487k

rsc.li/materials-horizons

## High-yield production of 2D crystals by wet-jet milling†

A. E. Del Rio Castillo, *[a] V. Pellegrini,[ab] A. Ansaldo, [a] F. Ricciardella,[a] H. Sun,[a] L. Marasco,[a] J. Buha,[c] Z. Dang,[c] L. Gagliani,[a] E. Lago,[a] N. Curreli,[ad] S. Gentiluomo,[a] F. Palazon, [c] M. Prato, [e] R. Oropesa-Nuñez,[b] P. S. Toth,[a] E. Mantero,[a] M. Crugliano,[a] A. Gamucci,[a] A. Tomadin,[a] M. Polini[a] and F. Bonaccorso *[ab]

Patent
WO2017089987A1

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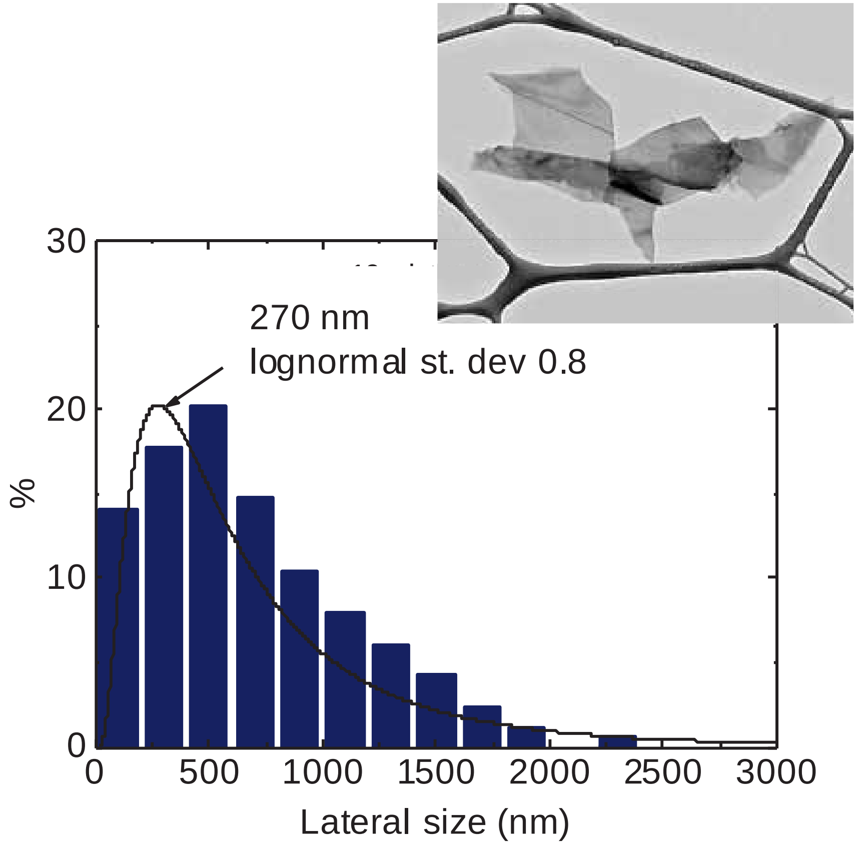
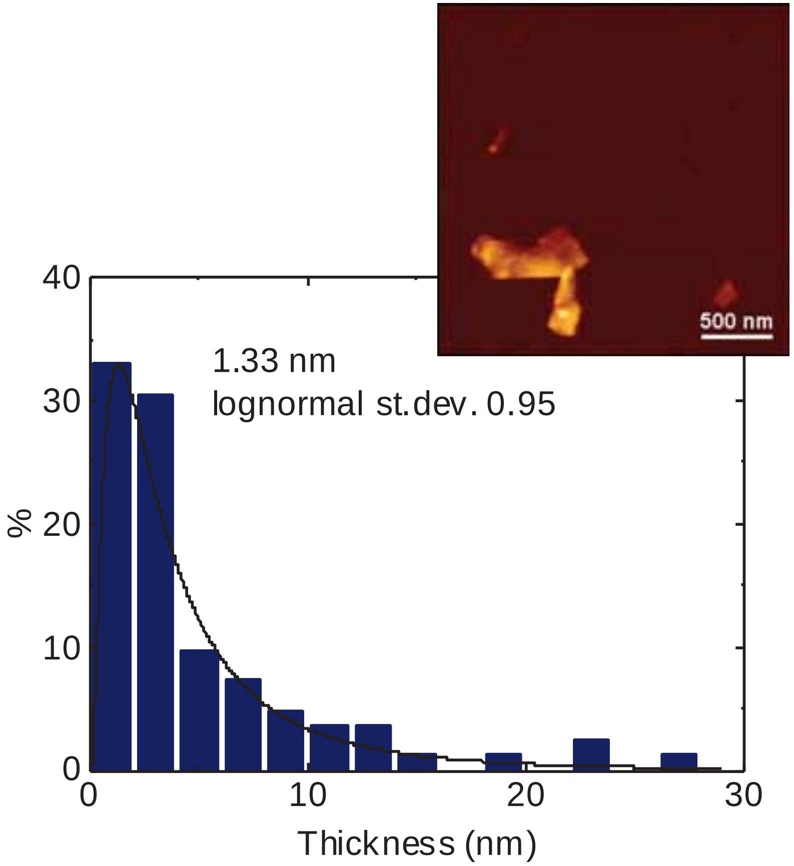
# Graphene production




FLG flakes have tunable lateral size 1μm-300 nm and down ~1.3 nm in thickness.

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CURIOSITY AND IMAGINATION
WILL TAKE YOU FURTHER
We offer unique and customized solutions to improve
and innovate the performance of our customers products,
shaping the material through the use of two-dimensional
crystals and the application of our know-how with scientific method.


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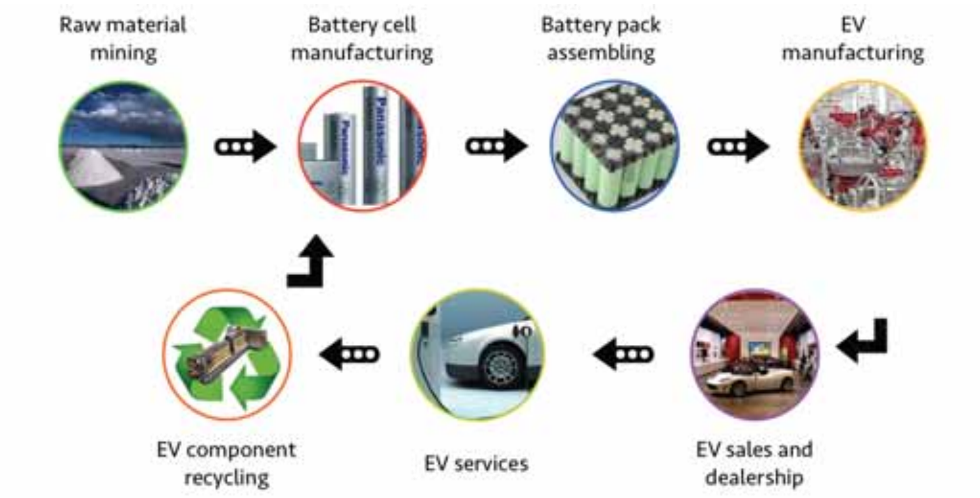



Supply chain

**DEVELOPMENT OF INNOVATIVE SOLUTIONS**

2D materials included compounds with high performances and multifunctionalities

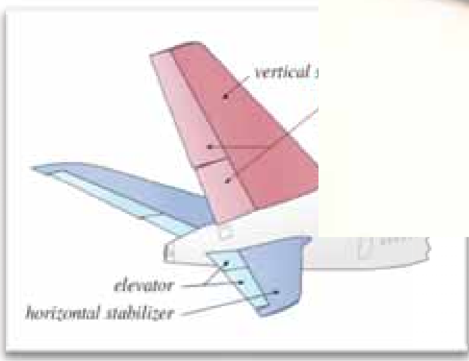
High quality 2D materials

**PRODUCTION**

Specific products


HEAD
super JOY


tennis
HEAD
GRAPHENE


vertical
elevator
horizontal stabilizer


-GRAPHENE ON PMMA

# What is the killer application?

# Alluminum's legacy

**Humphry Davy** **1808**
Aluminium can be produced from electrolytic reduction from alumina

**Hans Christian Ørsted** **1825**
First isolation of Aluminium

**Friedrich Woehler** **1827**
First Aluminium
30g production

**Henri Sainte-Claire Deville** **1856**
First industrial production
Few Tons per year

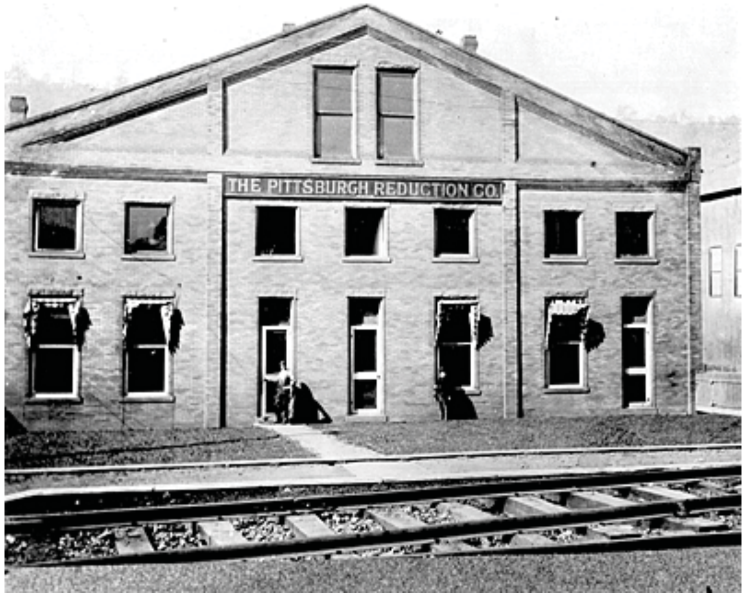
First applications:
ornaments and luxury items

*There is nothing harder than to make people use a new metal. Luxury items and ornaments cannot be the only sphere of its application. I hope the time will come when aluminium will serve to satisfy the daily needs.*

**Sainte-Claire Deville**


**Paul Héroult & Charles Hall** **1886**

New method of production.
Scale-up in Swiss and USA
up to hundreds of tons per year

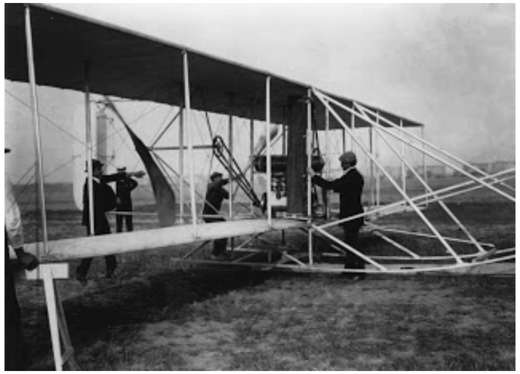



**1889**

Karl Benz
Sport car

**1903**

Wright airplane
with Aluminium
engine



# graphene

**2004 isolation**
**2019 production available**

**50 years compressed into 15 years…**

# Battery: basic facts

A battery is a device that converts chemical energy into electrical energy and vice versa


**Alessandro Volta 1799**

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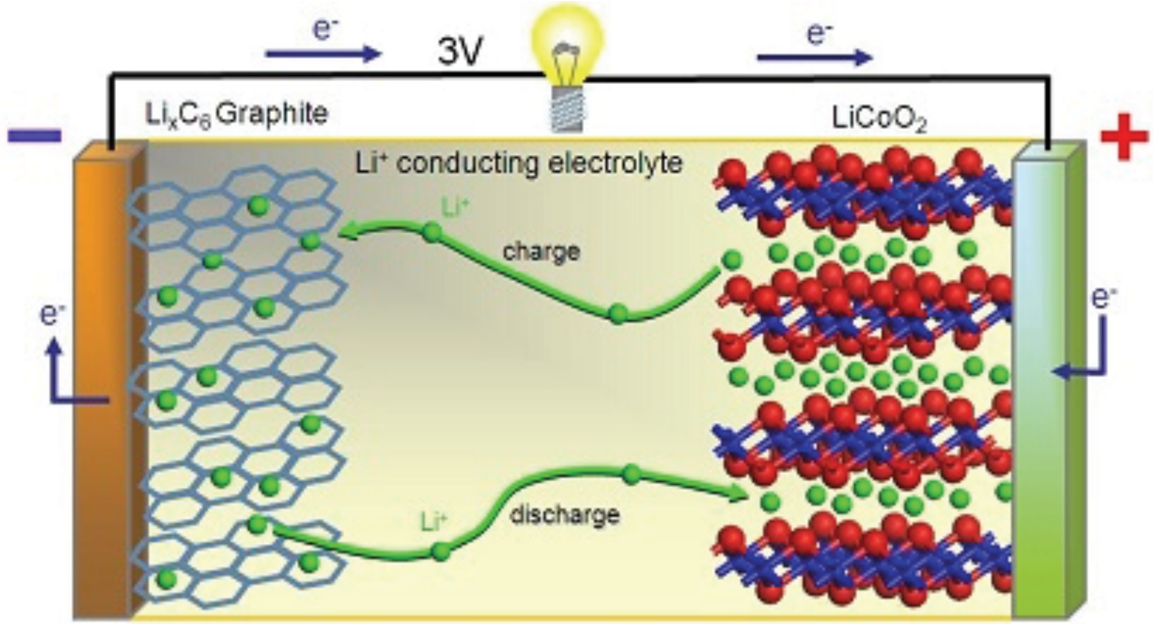
# Battery: basic facts

The primary components of Li-ion batteries are the anode, cathode, electrolyte ($LiPF_6$ in EC/DMC) and separator




Energy density

**150-250Wh/kg**

(cathode) reduction reaction : $CoO_2 + Li_+ + e^- \rightarrow LiCoO_2$ **160 mAh/g**

(anode) oxidation reaction : $LiC_6 \rightarrow Li^+ + C_6 + e^-$ **372 mAh/g**

**One Li ion for six Carbon atoms**

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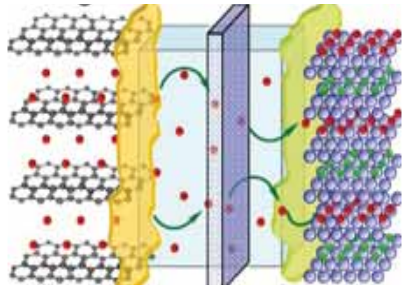
“Prices on electric cars will continue to drop until they’re within the reach of the average family”

**Washington Post 1915**

EV adoption is limited primarily due to the cost and inedeguate storage capacity of today’s batteries

# Need for high-energy density batteries

**1 liter of gasoline**
6000Wh/Kg



**1kg of Li-ion**
150-250 Wh/Kg

A 20-30 time gap!

Tesla model S→ 100Wh → >700-1000kg

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# The future of Lithium batteries

Lithium Ion Battery,
Cathode - $LiCoO_2$
150 mAh $g^{-1}$

Lithium Ion Battery,
Cathode – $LiFePO_4$
170 mAh $g^{-1}$

Lithium Ion Battery,
Anode – Si / carbon
1000 mAh $g^{-1}$

Lithium Ion Battery,
Cathode – NMC
200 mAh $g^{-1}$

Lithium Sulfur Battery,
Cathode – Sulfur Carbon
1675 mAh $g^{-1}$

Lithium Oxygen Battery,
Cathode – Oxygen Carbon
3500 mAh $g^{-1}$

**400 Wh $kg^{-1}$ → 500 Wh $Kg^{-1}$**

**1000 Wh $kg^{-1}$ → 3000 Wh $Kg^{-1}$**

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# The future of Lithium batteries

- New functionalities
- New materials
- New chemistries
- Industrially-scalable and cost effective approaches

<100$/kWh on cell level
<120$/KWh on module level
30$/Kg on active material

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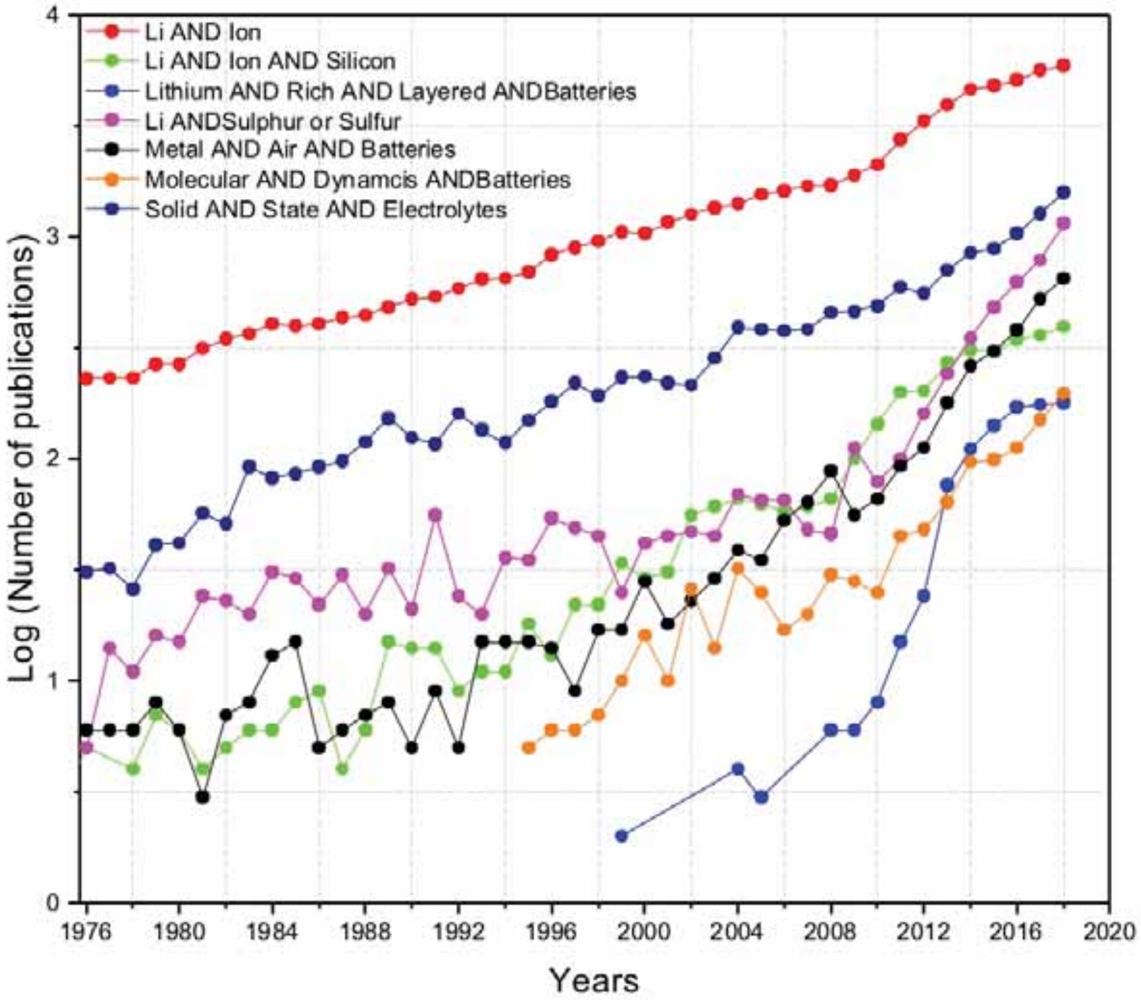
# The future of Lithium batteries




V. Pellegrini, S. Bodoardo, D. Brandell, K. Edstrom Solid State Communications (2019)

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## Is the killer application of graphene in energy storage ?

- Mechanical flexibility
- Electrical Conductivity
- Tunable morphological properties
- Functionalization
- Price compatible with market requirements (?)

*RESEARCH*

**REVIEW**

**2D MATERIALS**

# Graphene, related two-dimensional crystals, and hybrid systems for energy conversion and storage

Francesco Bonaccorso,[1,2*] Luigi Colombo,[3] Guihua Yu,[4] Meryl Stoller,[5] Valentina Tozzini,[6] Andrea C. Ferrari,[2] Rodney S. Ruoff,[7] Vittorio Pellegrini[1,6]

Graphene and related two-dimensional crystals and hybrid systems showcase several key properties that can address emerging energy needs, in particular for the ever growing market of portable and wearable energy conversion and storage devices. Graphene's flexibility, large surface area, and chemical stability, combined with its excellent electrical and thermal conductivity, make it promising as a catalyst in fuel and dye-sensitized solar cells. Chemically functionalized graphene can also improve storage and diffusion of ionic species and electric charge in batteries and supercapacitors. Two-dimensional crystals provide optoelectronic and photocatalytic properties complementing those of graphene, enabling the realization of ultrathin-film photovoltaic devices or systems for hydrogen production. Here, we review the use of graphene and related materials for energy conversion and storage, outlining the roadmap for future applications.

Science **347**,1246501 (2015)

F. Bonaccorso, V. Pellegrini
Material Matters, Sigma-Aldrich Merk
2016

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**2D** Materials

EDITORIAL

# Graphene-based technologies for energy applications, challenges and perspectives

Etienne Quesnel[1], Frédéric Roux[1], Fabrice Emieux[1], Pascal Faucherand[1], Emmanuel Kymakis[2], George Volonakis[3], Feliciano Giustino[3], Beatriz Martín-García[4,5], Iwan Moreels[4,5], Selmiye Alkan Gürsel[6], Ayşe Bayrakçeken Yurtcan[7], Vito Di Noto[8], Alexandr Talyzin[9], Igor Baburin[10], Diana Tranca[10], Gotthard Seifert[10], Luigi Crema[11], Giorgio Speranza[11], Valentina Tozzini[12], Paolo Bondavalli[13], Grégory Pognon[13], Cristina Botas[14], Daniel Carriazo[14,15], Gurpreet Singh[14], Teófilo Rojo[14], Gunwoo Kim[16,17], Wanjing Yu[16,17], Clare P Grey[16,17] and Vittorio Pellegrini[4]

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15 IKERBASQUE, Basque Foundation for Science, Bilbao, Spain
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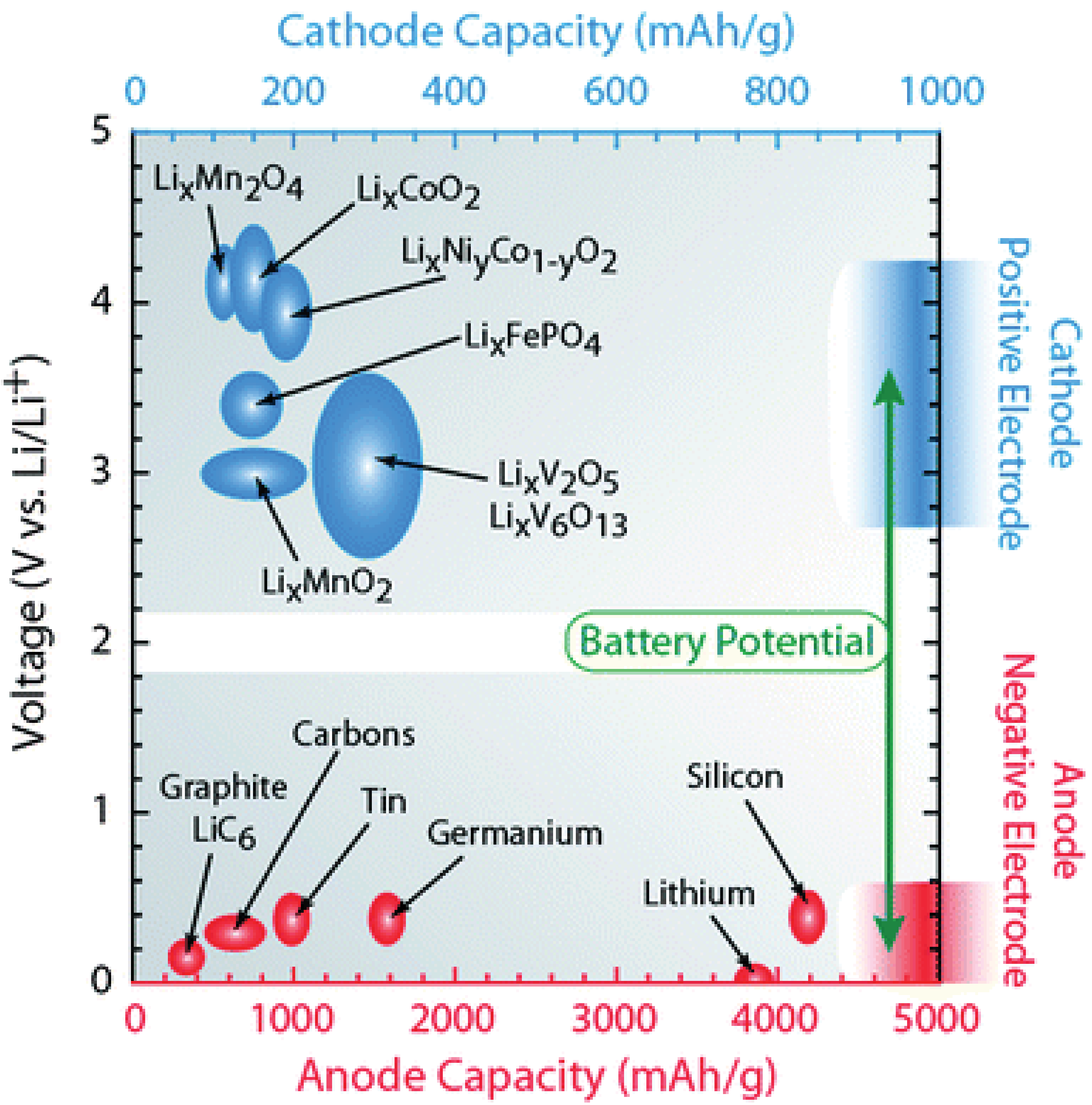
**Keywords:** graphene, energy application, hydrogen storage, battery, supercapacitor, photovoltaics, fuel cell

**Abstract**

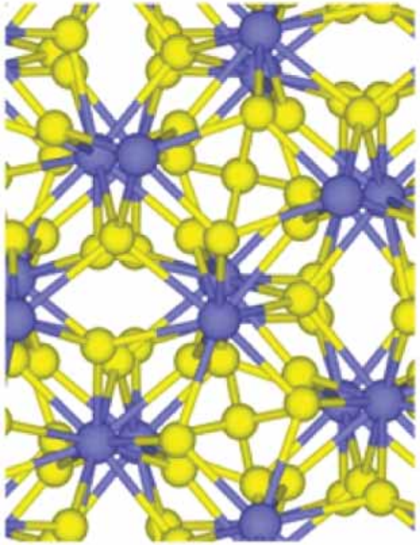
Here we report on technology developments implemented into the Graphene Flagship European project for the integration of graphene and graphene-related materials (GRMs) into energy application devices. Many of the technologies investigated so far aim at producing composite materials associating graphene or GRMs with either metal or semiconducting nanocrystals or other carbon nanostructures (e.g., CNT, graphite). These composites can be used favourably as hydrogen storage materials or solar cell absorbers. They can also provide better performing electrodes for fuel cells, batteries, or supercapacitors. For photovoltaic (PV) electrodes, where thin layers and interface engineering are required, surface technologies are preferred. We are using conventional vacuum processes to integrate graphene as well as radically new approaches based on laser irradiation strategies. For each application, the potential of implemented technologies is then presented on the basis of selected experimental and modelling results. It is shown in particular how some of these technologies can maximize the benefit taken from GRM integration. The technical challenges still to be addressed are highlighted and perspectives derived from the running works emphasized.



Cathode Capacity (mAh/g)
0
200
400
600
800
1000
5
4
3
2
1
0
Voltage (V vs. Li/Li+)
$Li_xMn_2O_4$
$Li_xCoO_2$
$Li_xNi_yCo_{1-y}O_2$
$Li_xFePO_4$
$Li_xV_2O_5$
$Li_xV_6O_{13}$
$Li_xMnO_2$
Battery Potential
Cathode
Positive Electrode
Anode
Negative Electrode
Carbons
Graphite
$LiC_6$
Tin
Germanium
Silicon
Lithium
0
1000
2000
3000
4000
5000
Anode Capacity (mAh/g)

# Silicon

## Silicon-based anodes for Li-ion batteries



### PLUS

Silicon expected capacity at room temperature:

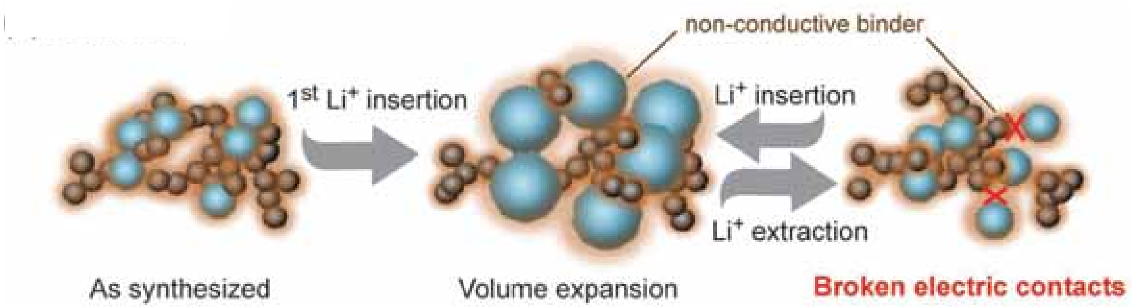
~ 3597 mAh/g ($Li_{15}Si_4$)

### MINUS

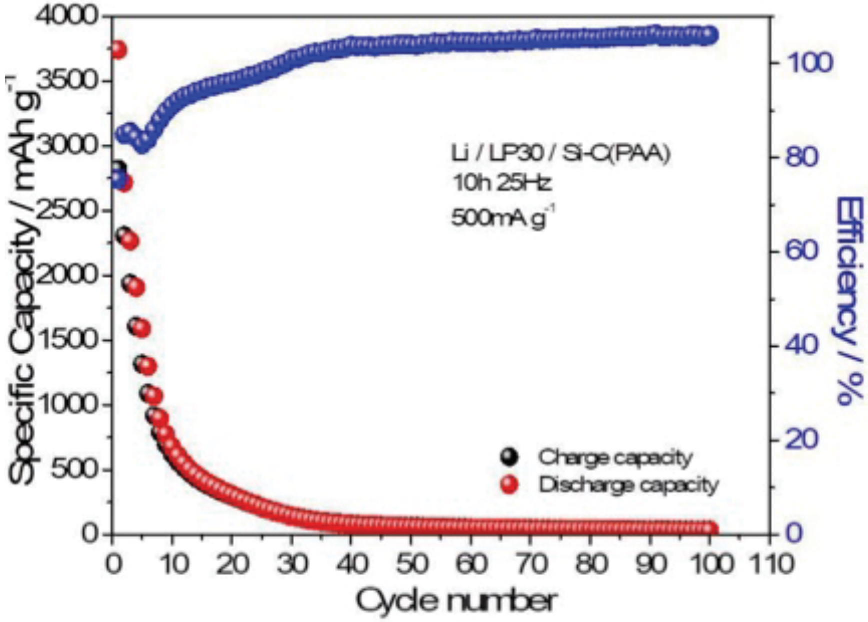
Large volume expansion (300%) → large irreversible capacity

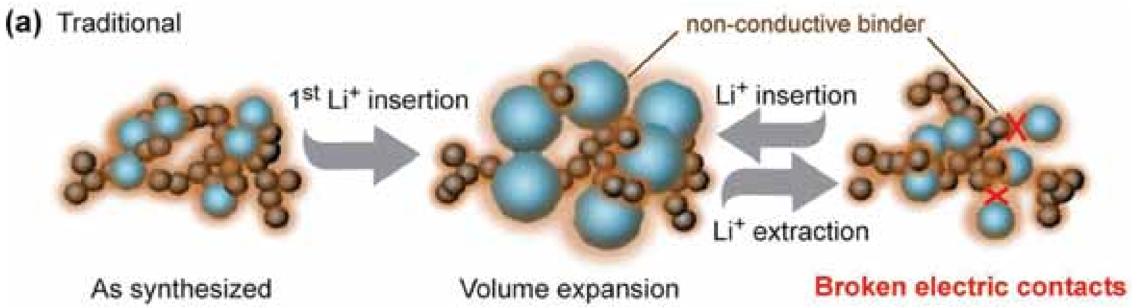
(cracks and pulverization)

Poor electrical conductivity



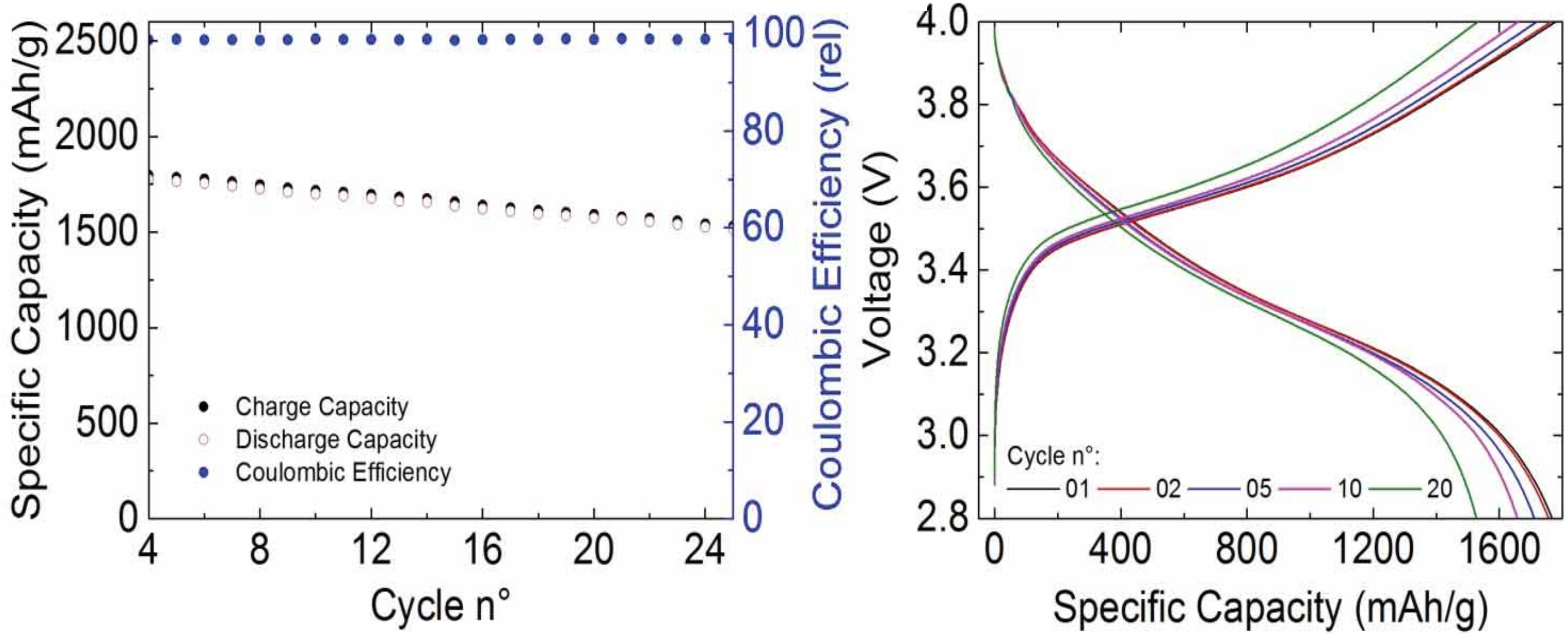
# Silicon capacity fading



# Si-Graphene–NMC full cell

## Graphene as additive outperforms both SuperP and r-GO




- NMC with a nominal capacity of 2.25mAh/cm$^2$.
- Anode and cathode masses balanced → anode reversible capacity (2300 mAh/g) → cathode reversible capacity (137mAh/g).
- 350-400 Wh/kg

S. Palumbo, L. Silvestri et al. ACS Applied Energy Materials (2019)

E. Greco et al, J. Material Chemistry A **5**, 19306 (2017)

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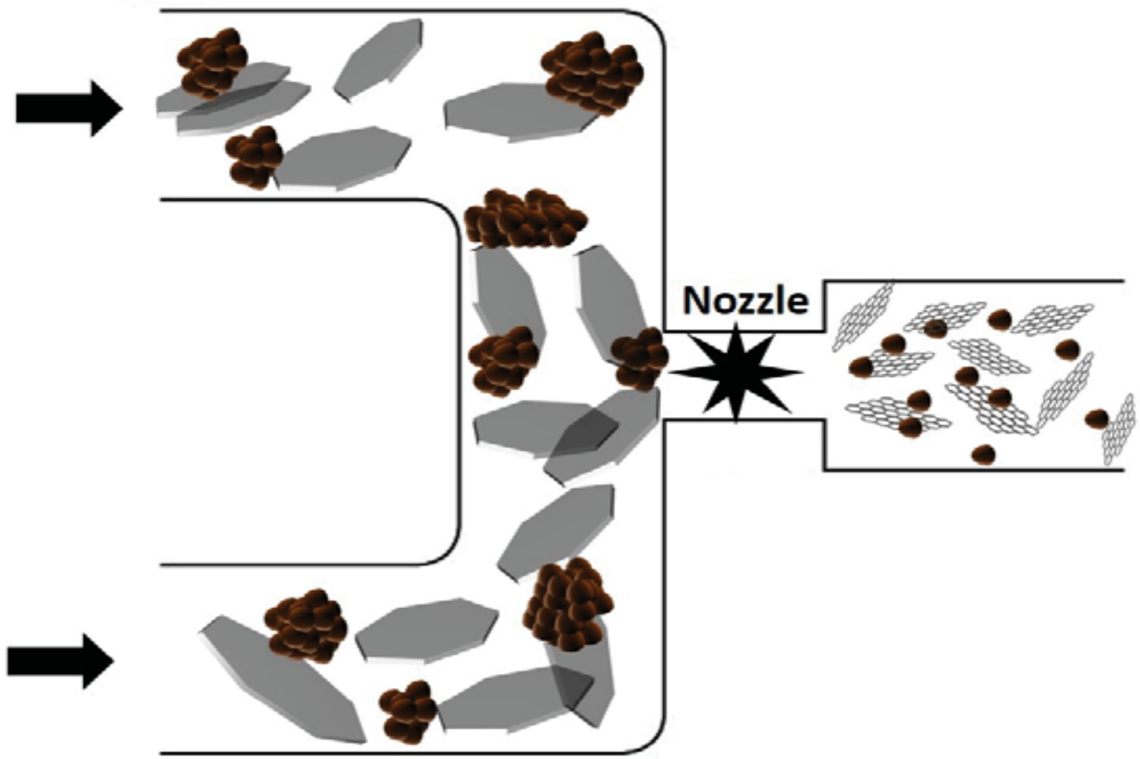
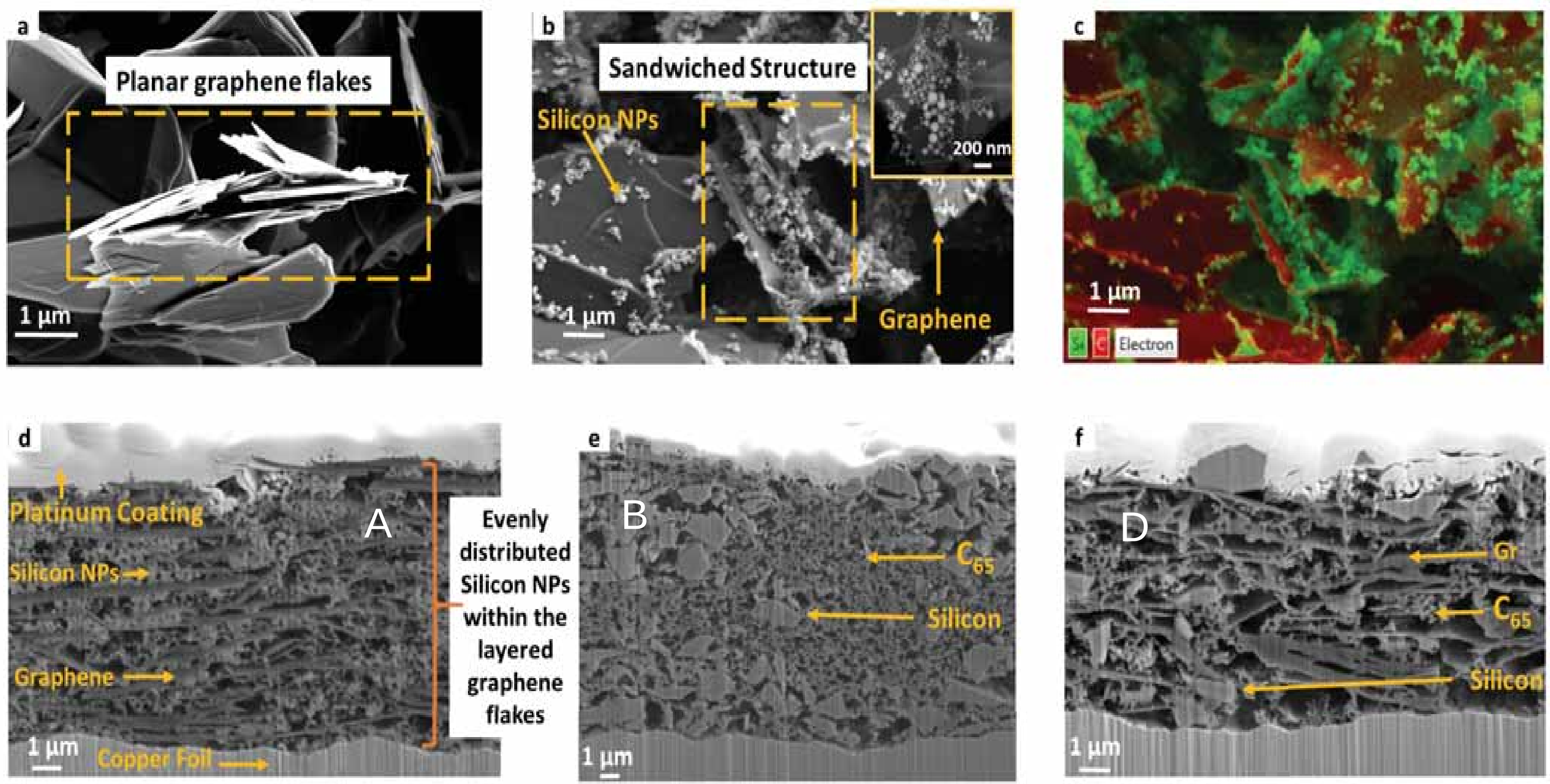
# A new method of production



# Laminated structure




R. Maik, M. Loveridge, V. Pellegrini et al. submitted

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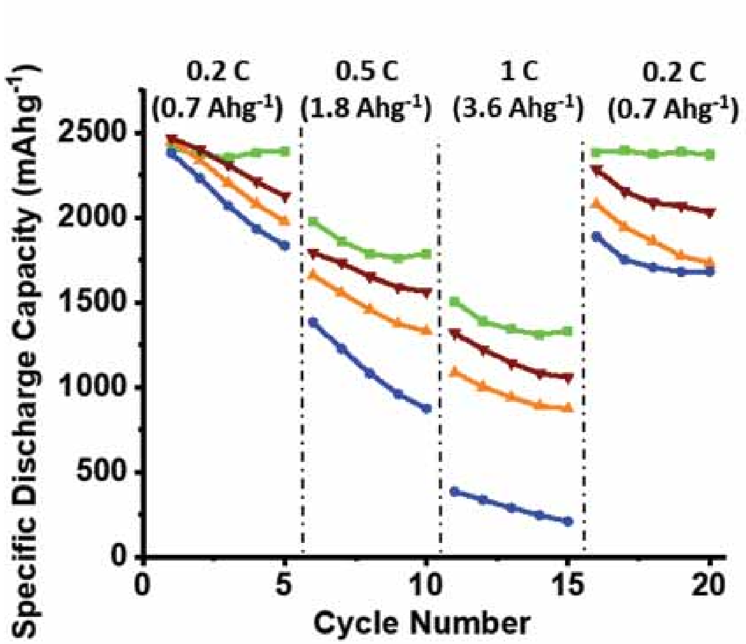
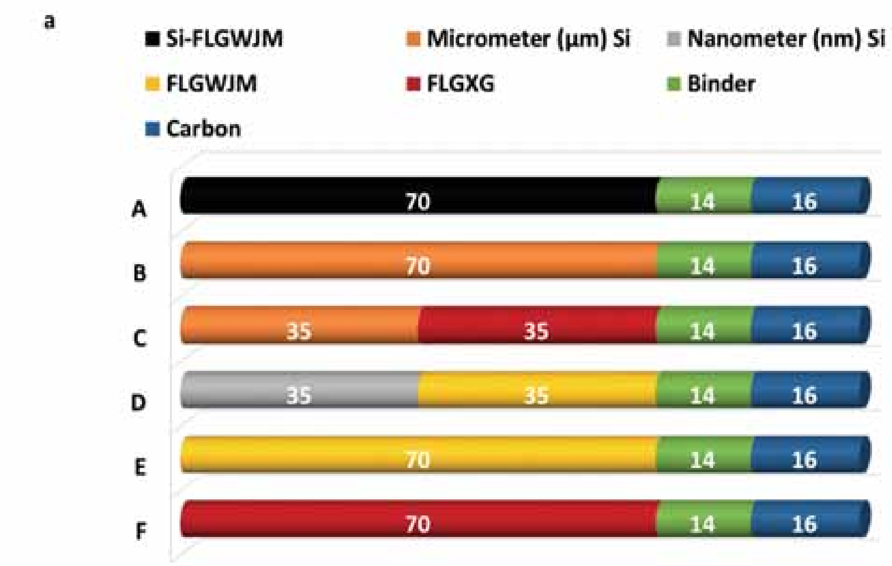
# Exceptional electrochemical behavior




**450 cycles: capacity retention of 98 %; columbic efficiency of 99.85% (under a current density of 358 mA $g^{-1}$).**

R. Maik, M. Loverdige, V. Pellegrini et al. submitted

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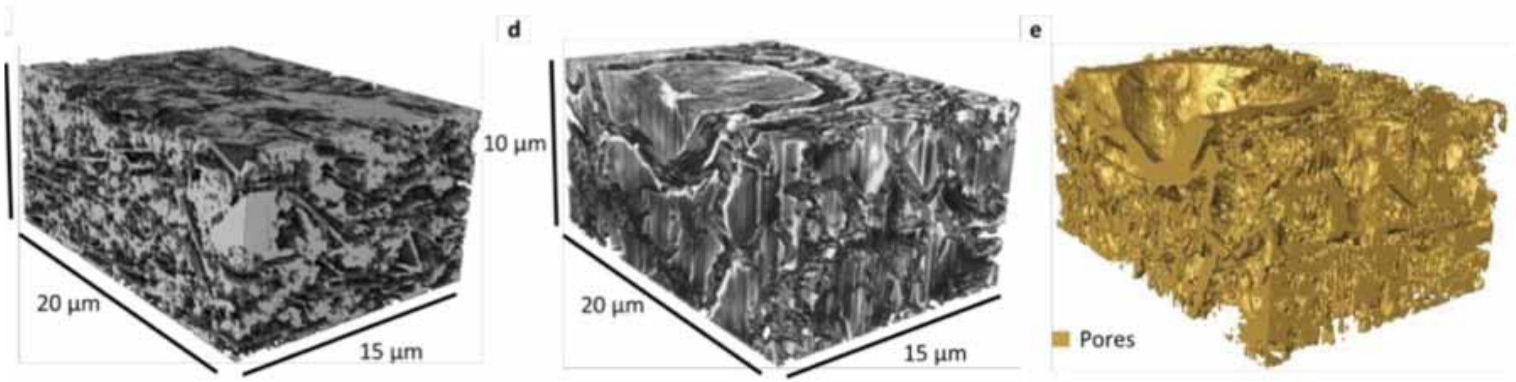
# Impact of graphene in silicon electrodes

**Stabilization of the electrode's structure**

→ facilitate movement of Li+ ions throughout the bulk of the electrode material during cycling




Pristine IIT Si-Gr electrode
Porosity: 34.40%

Cycled IIT Si-Gr electrode
Porosity: 29.21%

Pore volume reconstruction of cycled IIT Si-Gr electrode

**Decrease in porosity**:

- **<u>Silicon 75%</u>** after 200 cycles
- **<u>Silicon/Graphene 15%</u>** after 200 cycles

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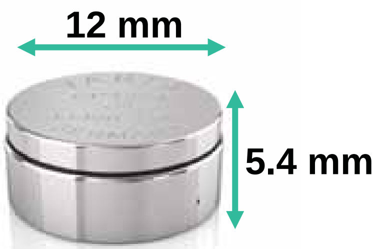
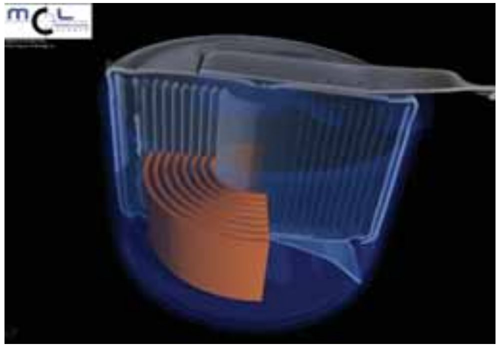
# Silicon/Graphene - Prototyping



In-Ear Headset

Fitness Tracker

Smart Key

Insulin Patch

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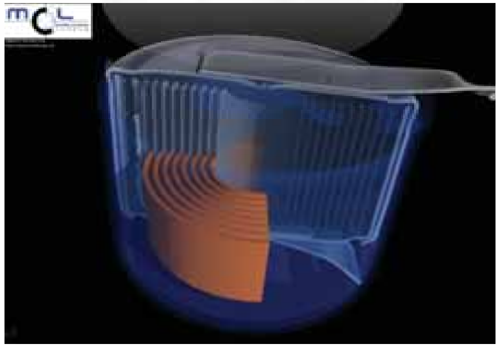
VARTA micro innovation

# Silicon/Graphene - Prototyping

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First prototypes from industrial production line
+25% of energy density vs market products

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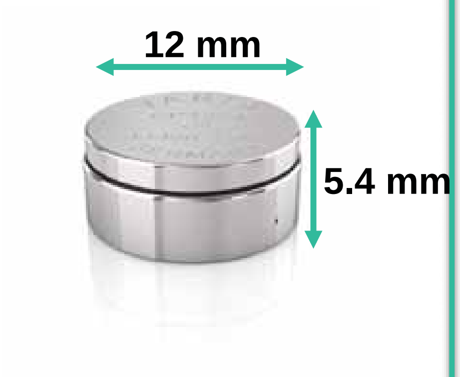
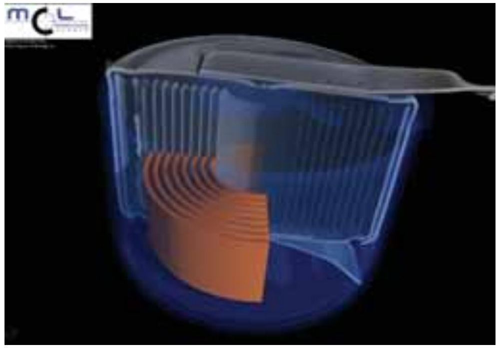
# Silicon/Graphene - Prototyping

## Format



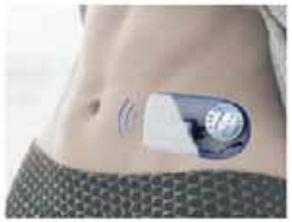



## Application

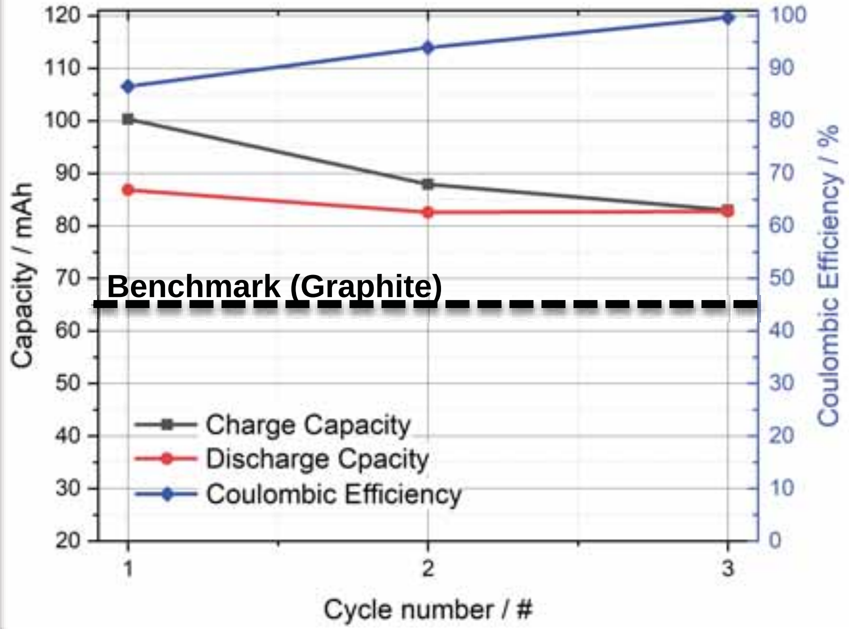

In-Ear Headset


Fitness Tracker


Smart Key


Insulin Patch

## Current results




| Parameter | Value |
|---|---|
| Total Capacity* | 88 mAh (+35%**) |
| Total Energy* | 300 mWh (+25%**) |
| Cyclability | tbd |

*0.1C Discharge
** compared to benchmark

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VARTA micro innovation


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www.graphene-flagship.eu

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Elkem
A Bluestar Company

enel
Green Power

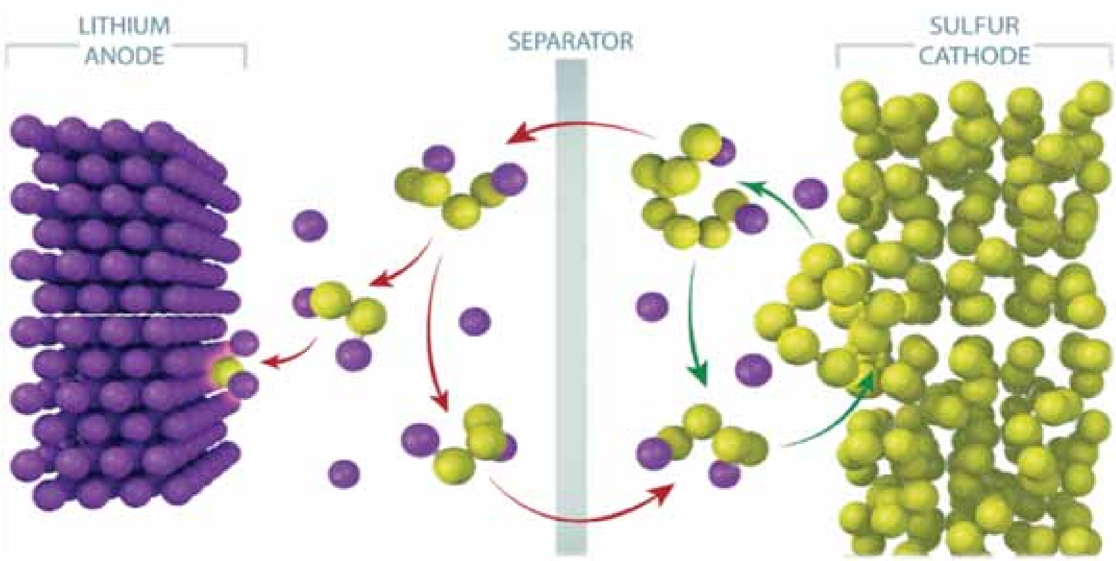
# Sulfur rich graphene-based cathode

- Graphene in cathodes in Li-S

$S_8 + 16Li^+ = 8Li_2S$

1675 $mAhg^{-1}$ → 3500 $Whkg^{-1}$

# Li-S : Drawbacks

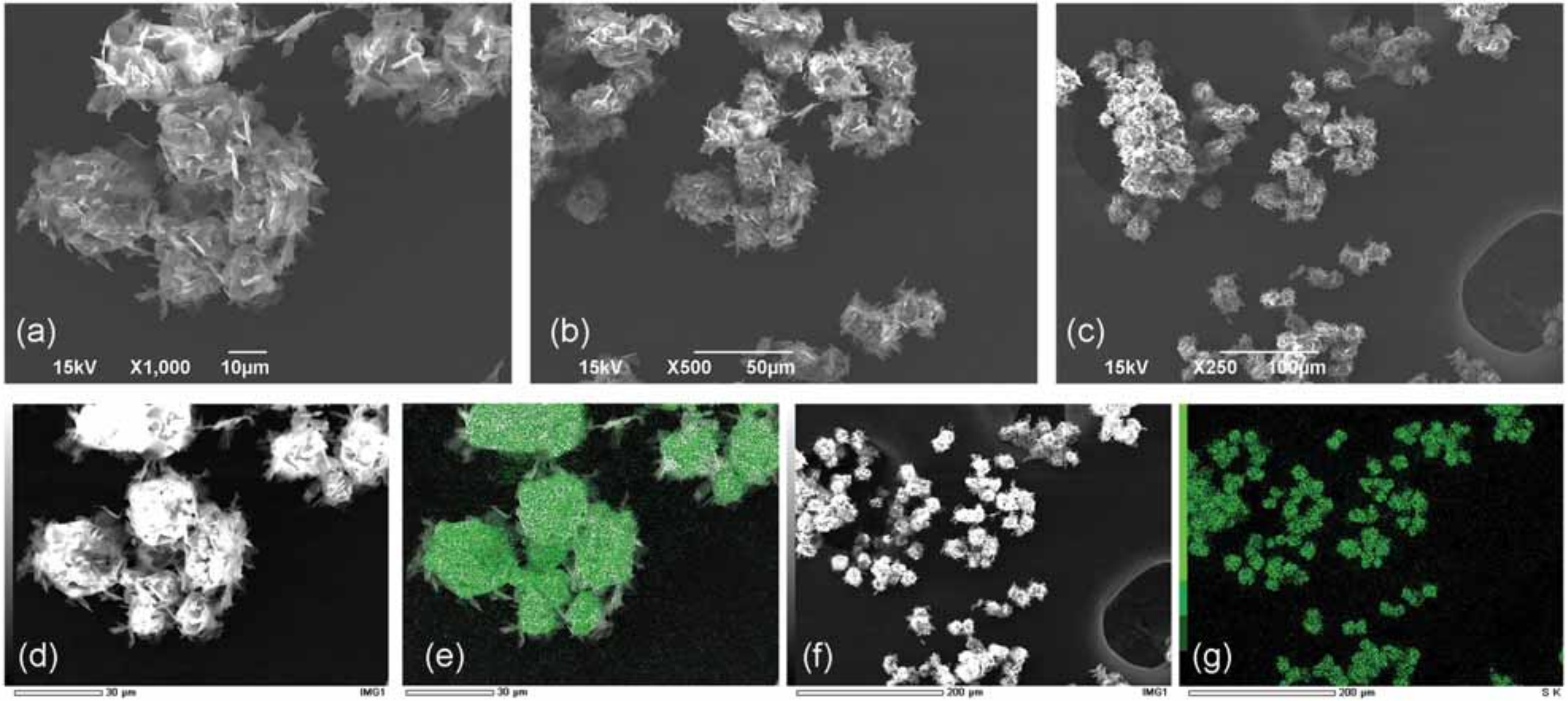



$Li_2S_8$ , $Li_2S_6$ , $Li_2S_4$ , $Li_2S_2$

$Li_2S$

- Both sulfur and lithium sulfides are intrinsically insulated
- The intermediate discharge products lithium polysulfides are soluble in the organic electrolyte → loss of active materials
- The soluble polysulfide during the charge processes may migrate from the cathode to the anode, reacting on the anode surface producing an electrochemical short circuit well known as polysulfide shuttle effect

# Sulfur rich graphene-based cathode




homogenous agglomerate of sulfur particles covered by grapher

Active material: 89% Sulfur 10% graphene

Sulfur mass loading of 1.5mg$_{(S)}$ cm$^{-2}$

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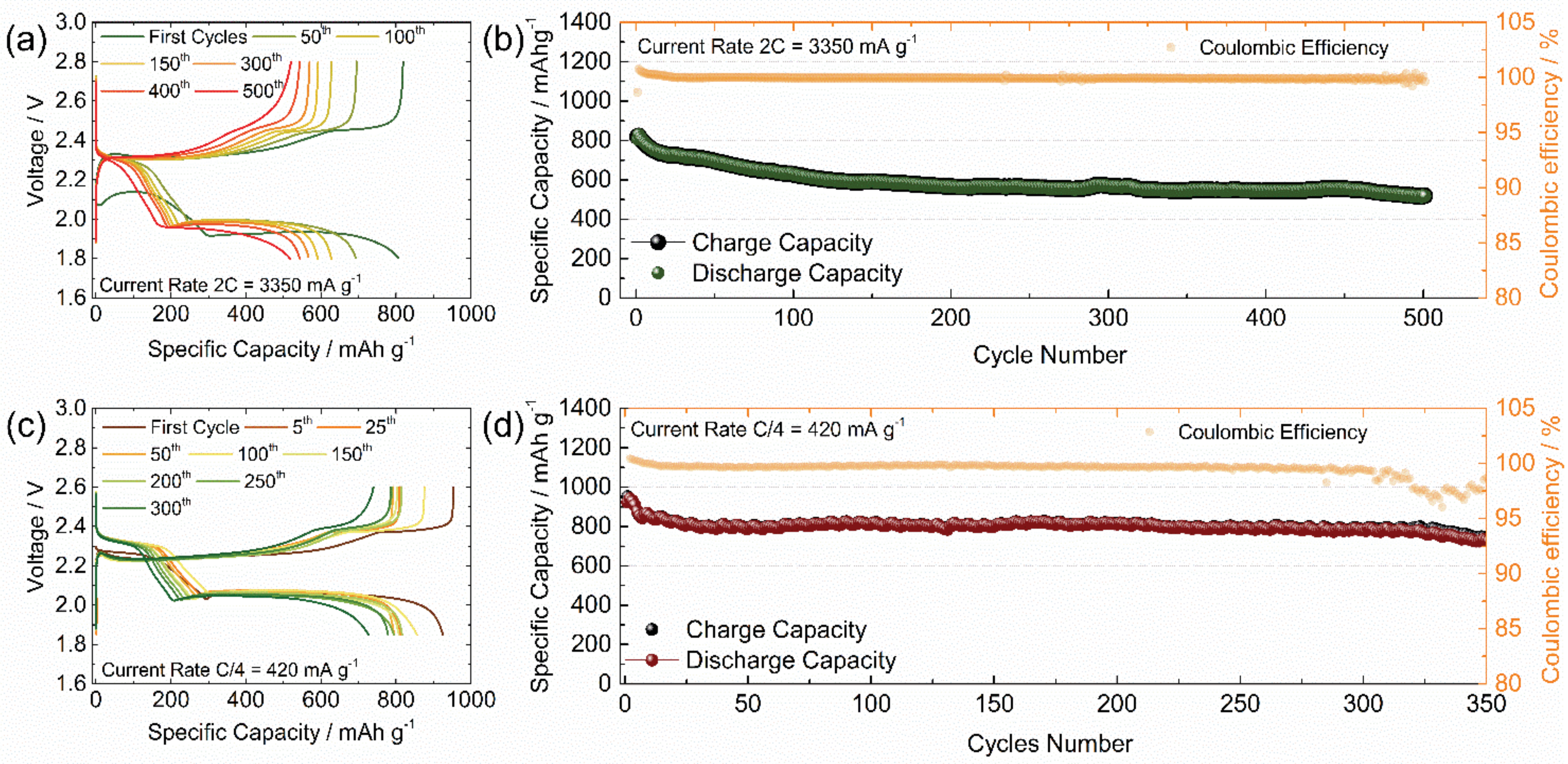
# S-Graphene cathode




Patent submitted
L. Carbone et al. ChemSusChem (accepted)

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# Graphene

- Mechanical flexibility
- Electrical Conductivity
- Tunable morphological properties
- Functionalization
- Price compatible with market requirements (?)

Li-ion anodes

Li-rich layered oxides

Li-S cathodes

Aluminum-ion cathodes

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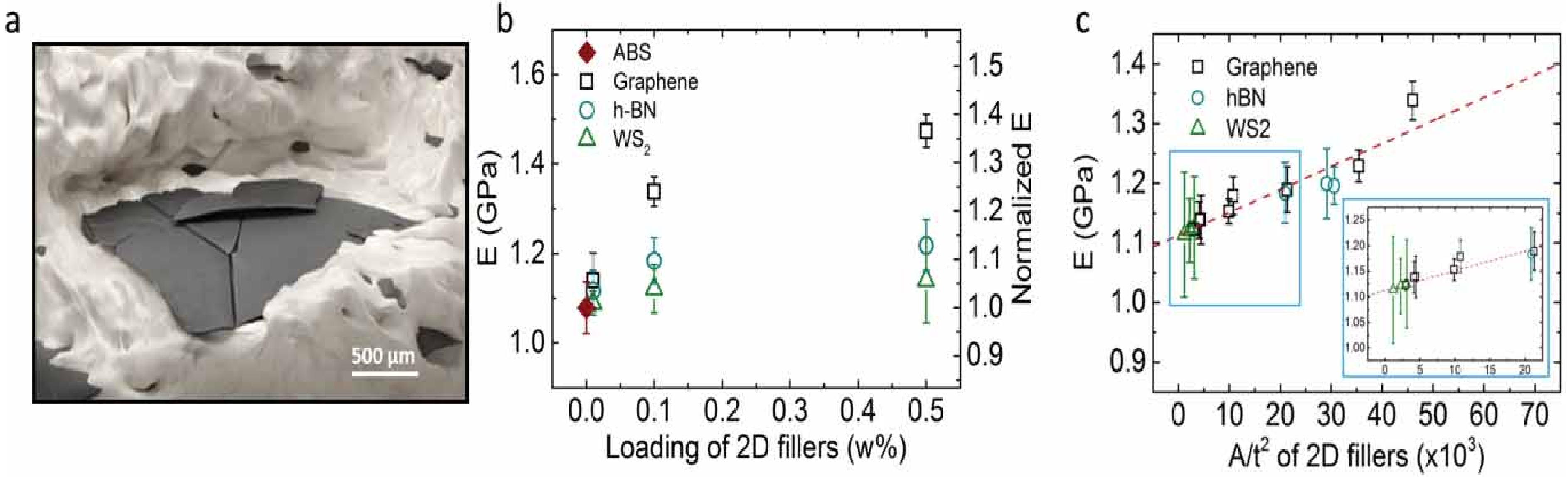
# ABS-graphene: role of quality




E. Lago et al., submitted

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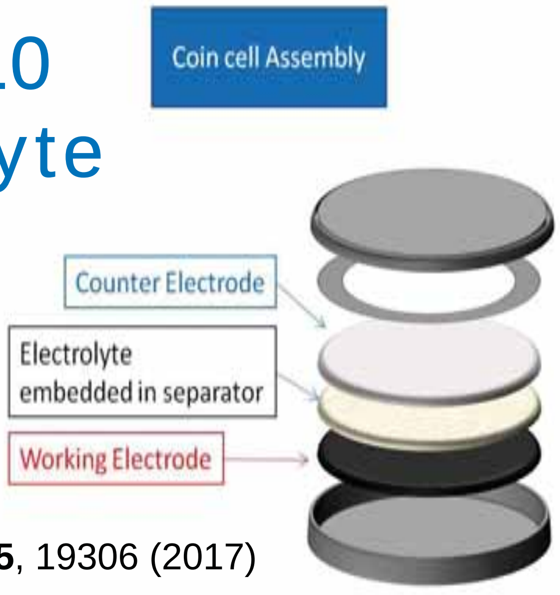
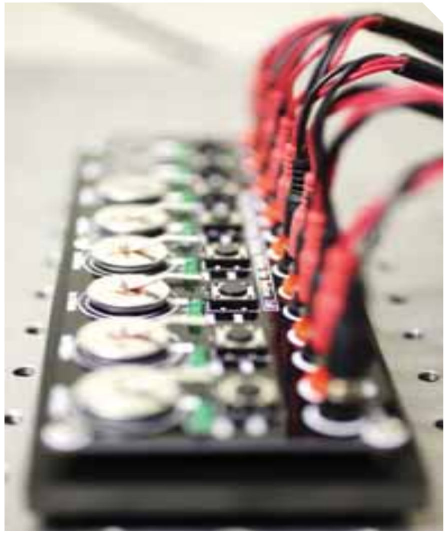
# Graphene Silicon composite for anodes

Different approaches:

- Dispersion/sonication/annealing @ 700˚

Si/G:SP:PAA= 80:10:10
LP30+FEC as electrolyte



E. Greco et al, J. Material Chemistry A **5**, 19306 (2017)

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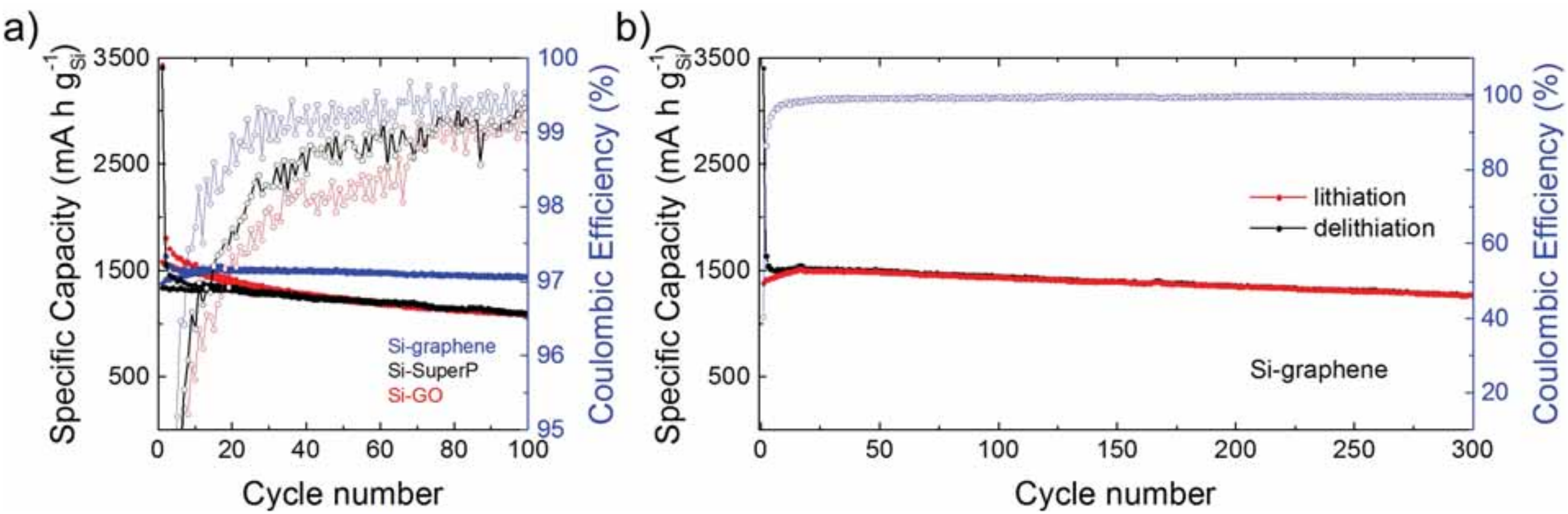
# Si-Graphene anode: stability

Silicon/Graphene:PAA:SP (80:10:10)
LP30 + 10% FEC , 400mA/g , Coin cell, Annealed at 750 ºC




**Graphene as additive outperforms both SuperP and r-GO**

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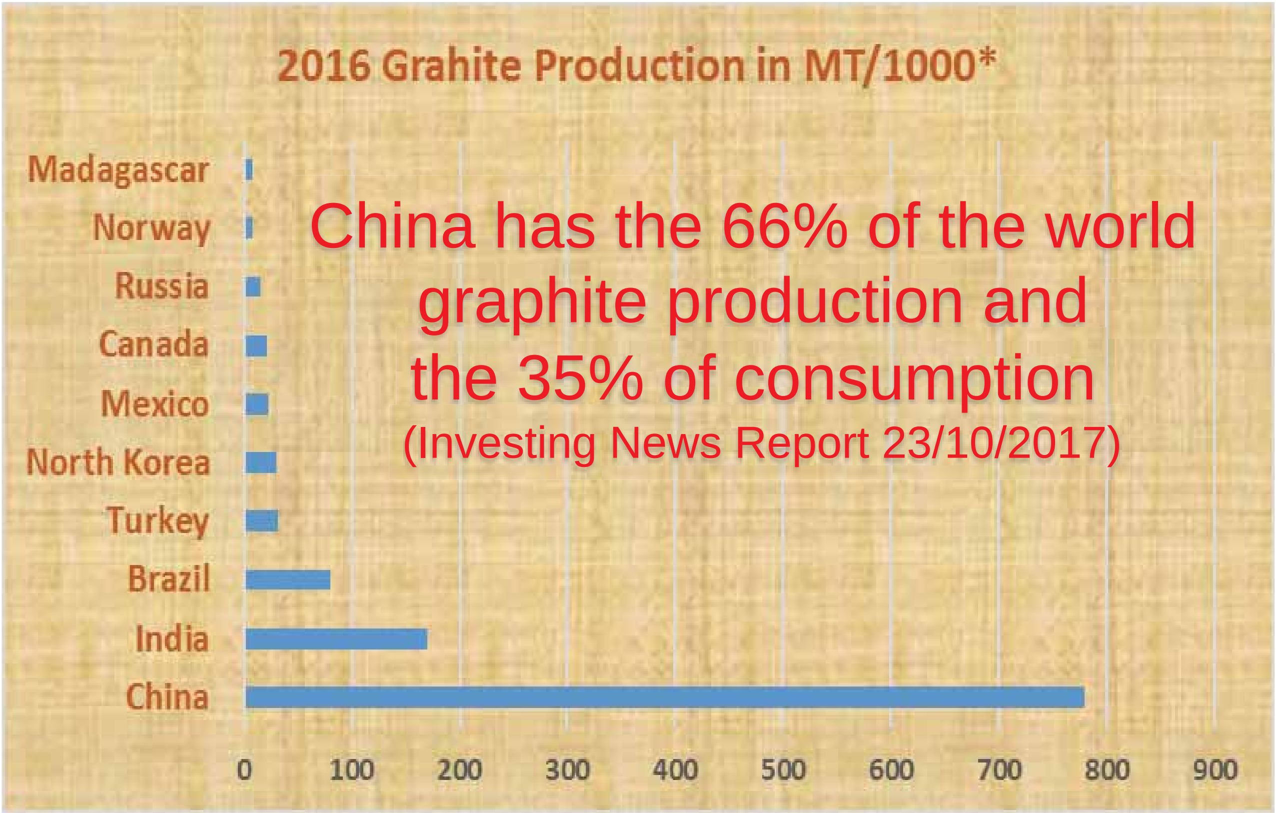
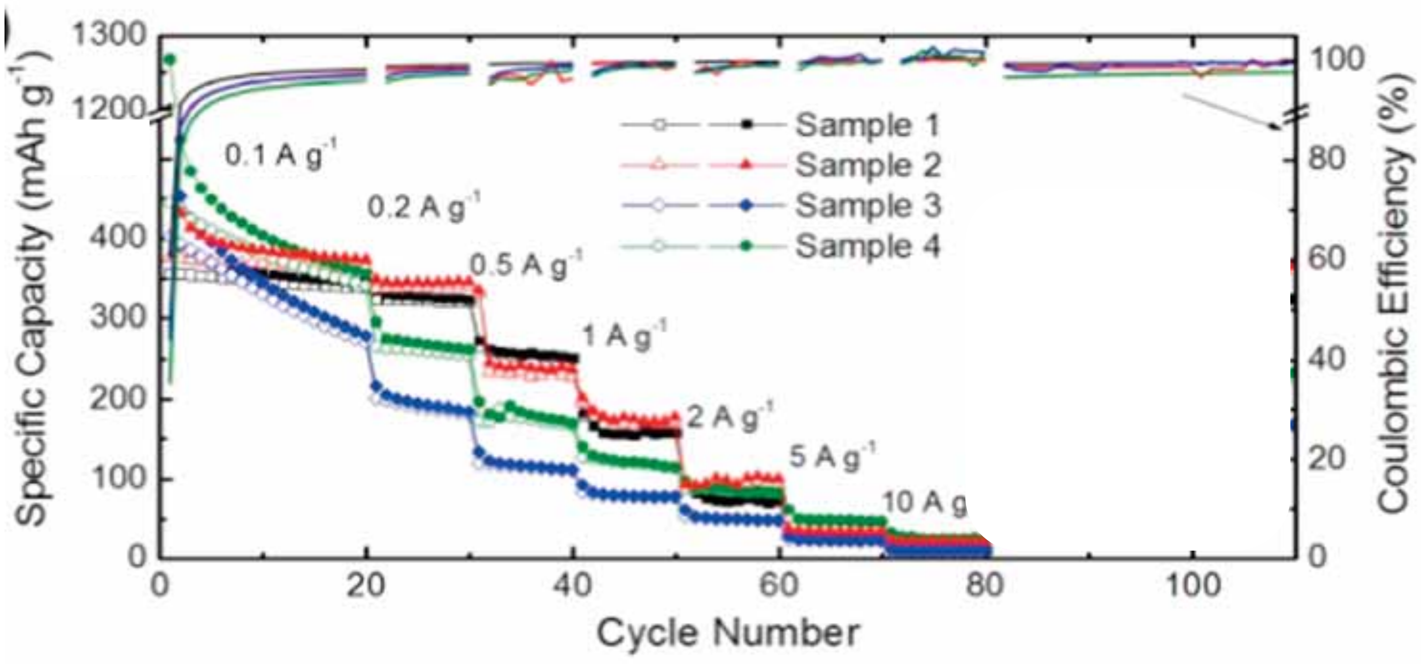
E. Greco et al, J. Material Chemistry A **5**, 19306 (2017)

# The first ten producers of graphite in the world




** data from US Geological Survey (2017)*

# Li-ion: can Graphene replace graphite ?




(anode) oxidation reaction : $Li_2C_6 \rightarrow 2Li^+ + C_6 + 2e^-$ **744 mAh/g**

F. Bonaccorso et al., Science (2015)
H. Sun al., Solid State Comm **251**, 88 (2017)
H. Sun et al., Journal of Mater. Chem. A **4**, 6886 (2016)
J. Hassoun et al., NanoLetters **14**, 4901 (2014)

- Marginal improvement vs graphite
- Irreversible processes
- Cost

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