Graphene Nanoribbons from Carbon Nanotubes

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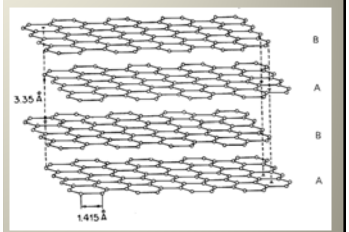
Graphite and Graphene

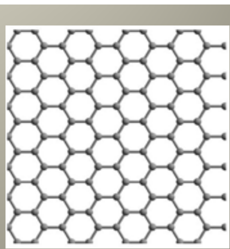
doi:10.1351/goldbook.G02683

graphene layer

A single carbon layer of the graphite structure, describing its nature by analogy to a polycyclic aromatic hydrocarbon of quasi infinite size.
Note:
Previously, descriptions such as graphite layers, carbon layers or carbon sheets have been used for the term graphene. Because graphite designates that modification of the chemical element carbon, in which planar sheets of carbon atoms, each atom bound to three neighbours in a honeycomb-like structure, are stacked in a three-dimensional regular order, it is not correct to use for a single layer a term which includes the term graphite, which would imply a three-dimensional structure. The term graphene should be used only when the reactions, structural relations or other properties of individual layers are discussed.

Source:
PAC, 1995, 67, 473 (*Recommended terminology for the description of carbon as a solid (IUPAC Recommendations 1995)*) on page 491





"Graphene is the name given to a flat monolayer of carbon atoms tightly packed into a two-dimensional (2D) honeycomb lattice"
Geim & Novoselov, Nature Materials 2007

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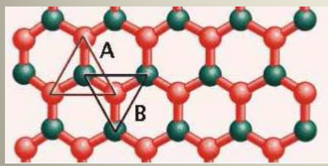
Graphene



Graphene lattice. The unit cell contains two atoms A and B.

(T. Chakraborty, *Physics in Canada*, vol. 66, No. 4, 1 Nov 2010)

The knowledge accumulated along the past 6-7 years shows that:

- Balistic transport and very high electron mobility when suspended (zero band gap)
- Can withstand large current densities
- Is almost impermeable to gases
- Is chemically stable
- Has high thermal conductivity
- …

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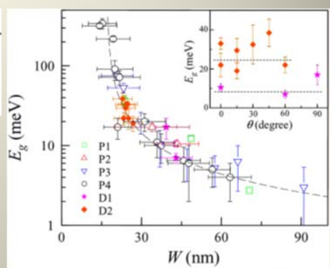
Graphene nanoribbons

PRL **98**, 206805 (2007) PHYSICAL REVIEW LETTERS

Energy Band-Gap Engineering of Graphene Nanoribbons

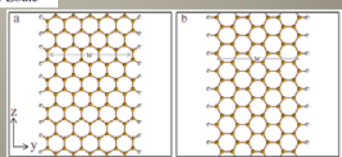
Melinda Y. Han,[1] Barbaros Özyilmaz,[2] Yuanbo Zhang,[2] and Philip Kim[2]



PRL **99**, 186801 (2007) PHYSICAL REVIEW LETTERS

Quasiparticle Energies and Band Gaps in Graphene Nanoribbons

Li Yang,[1,2] Cheol-Hwan Park,[1,2] Young-Woo Son,[3] Marvin L. Cohen,[1,2] and Steven G. Louie[1,2]



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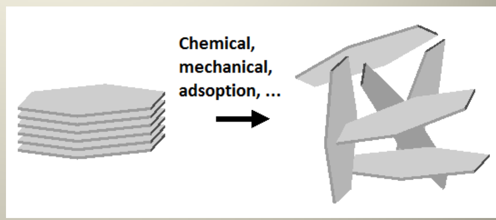
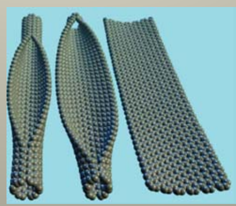
Graphene and graphene nanoribbons

Production in "large" scale:

- Exfolation of graphite



- Exfoliation of carbon nanotubes



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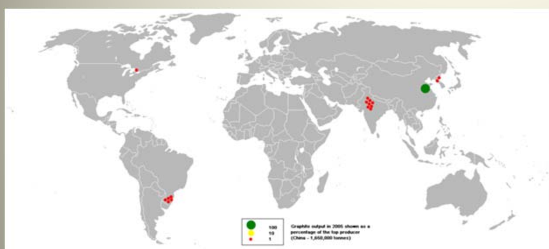
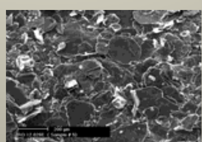
Graphene from Graphite



Natural graphite is formed in either metamorphic or igneous environments. It is generally classified into three types:

- flake graphite



- vein graphite

- amorphous graphite (microcrystalline graphite?)

Synthetic graphite: high temperature treatment of amorphous carbon materials

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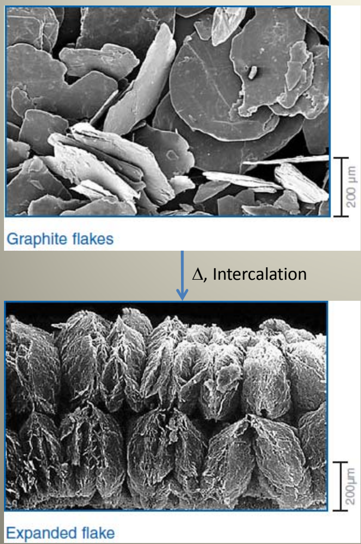
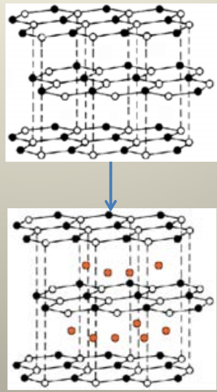
Graphene from graphite: exfoliation



Graphit Kropfmühl AG



Carbon content: 85 – 98 %

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Graphene from graphite: exfoliation

H. He, J. Klinowski, M.I Forster, A. Lerf, A new structural model for graphite oxide, *Chemical Physics Letters 287 (1998) 53–56*

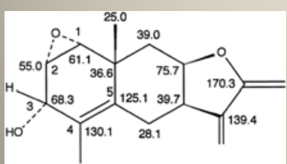

Fig. 3. Model compound for the assignment of the ^{13}C NMR spectra of GO.

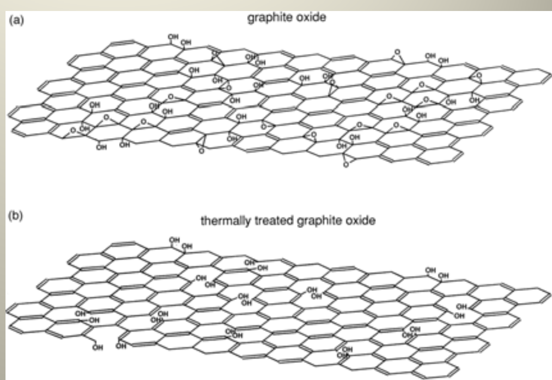

Fig. 4. New structural model for a. as-prepared GO; b. GO after thermal treatment at 100 °C for 24 h in vacuum.

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Graphene from graphite: exfoliation

Table 1. Methods for the oxidation of graphite to graphite oxide.

	Brodie	Staudenmaier	Hummers	Modified Hummers	
Year	1859	1898	1958	1999	2004
Oxidants	$KClO_3$, HNO_3	$KClO_3$ (or $NaClO_3$), HNO_3, H_2SO_4	$NaNO_3$, $KMnO_4$, H_2SO_4	pre-ox: $K_2S_2O_8$, P_2O_5, H_2SO_4 ox: $KMnO_4$, H_2SO_4	$NaNO_3$, $KMnO_4$, H_2SO_4
C:O ratio	2.16[27] 2.28[36]	N/A[28] 1.85[36]	2.25[29] 2.17[36]	1.3[60]	1.8[119]
Reaction time	3–4 days[27] 10 h[36]	1–2 days[28] 10 days[36]	≈2 h[29] 9–10 h[36]	6 h pre-ox + 2 h ox[60]	≈5 days[119]
Intersheet spacing [Å]	5.95[36]	6.23[36]	6.67[36]	6.9[60]	8.3[119]

small 2010, 6, No. 6, 711–723 www.small-journal.com 713

Oxidation of graphite, followed by reduction:

Graphene oxide

↓ Sodium borohydride; Hydrazine; Viatmin C; etc.

Reduced graphene

Figure 4. Digital pictures of unreduced graphene oxide suspensions in water, DMF, and NMP (top), together with their deoxygenated counterparts 4 weeks after reduction with vitamin C (bottom).

M. J. Fernández-Merino, L. Guardia, J. I. Paredes, S. Villar-Rodil, P. Solís-Fernández, A. Martínez-Alonso, and J. M. D. Tascón, *J. Phys. Chem. C*, 2010, *114*, 6426–6432

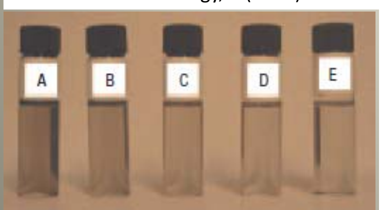
Graphene from graphite: exfoliation

High-yield production of graphene by liquid-phase exfoliation of graphite

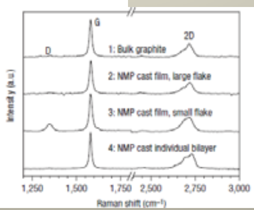
YENNY HERNANDEZ[1†], VALERIA NICOLOSI[1†], MUSTAFA LOTYA[1], FIONA M. BLIGHE[1], ZHENYU SUN[1,2], SUKANTA DE[1,2], I. T. McGOVERN[1], BRENDAN HOLLAND[1], MICHELE BYRNE[1], YURII K. GUN'KO[2,3], JOHN J. BOLAND[2,3], PETER NIRAJ[2,3], GEORG DUESBERG[2,3], SATHEESH KRISHNAMURTHY[2,3], ROBBIE GOODHUE[4], JOHN HUTCHISON[5], VITTORIO SCARDACI[6], ANDREA C. FERRARI[6] AND JONATHAN N. COLEMAN[1,2*]

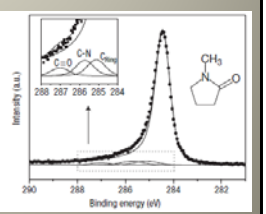
Nature Nanotechnology, 3 (2008) 563-568

graphene dispersions produced by exfoliation of graphite in organic solvents such as *N-methyl-pyrrolidone*



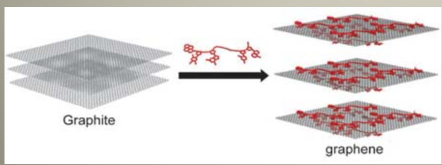
6 μg ml^{-1} to 4 μg ml^{-1}, after centrifugation





Graphene from graphite: exfoliation

P. Laaksonen et al., Interfacial Engineering by Proteins: Exfoliation and Functionalization of Graphene by Hydrophobins, *Angewandte Chemie International Edition*, Volume 49, Issue 29, 4946–4949 (2010).



F. Liu, J. Young Choi, T. S. Seo, DNA mediated water-dispersible graphene fabrication and gold nanoparticle-graphene hybrid, *Chem. Commun.*, 2010, 46, 2844-2846

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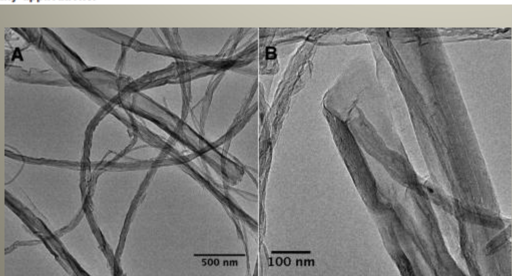
Graphene nanoribbons from carbon nanotubes

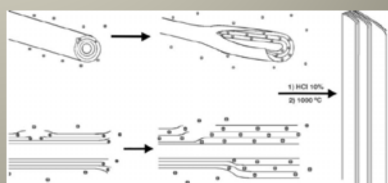
Ex-MWNTs: Graphene Sheets and Ribbons Produced by Lithium Intercalation and Exfoliation of Carbon Nanotubes

Abraham G. Cano-Márquez,[†] Fernando J. Rodríguez-Macías,[†] Jessica Campos-Delgado,[†] Claudia G. Espinosa-González,[†] Ferdinando Tristán-López,[†] Daniel Ramírez-González,[†] David A. Cullen,[‡] David J. Smith,[‡] Mauricio Terrones,[†] and Yadira I. Vega-Cantú[*,†]

NANO LETTERS
2009
Vol. 9, No. 4
1527-1533

We found that multiwalled carbon nanotubes (MWNTs) can be opened longitudinally by intercalation of lithium and ammonia followed by exfoliation. Intercalation of open-ended tubes and exfoliation with acid treatment and abrupt heating provided the best results. The resulting material consists of: (i) multilayered flat graphitic structures (nanoribbons), (ii) partially open MWNTs, and (iii) graphene flakes. We called the completely unwrapped nanotubes ex-MWNTs, and their large number of edge atoms makes them attractive for many applications.





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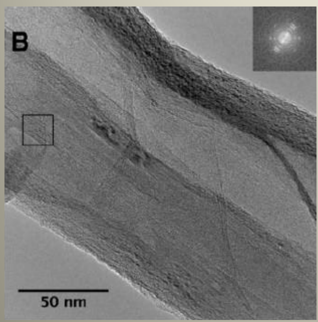
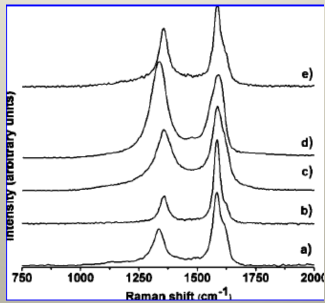
Graphene nanoribbons from carbon nanotubes





HCl exfoliated after thermal treatments

Li-NH_3 intercalated, exfoliated with HCl

cut-MWNTs treated only with NH_3;

cut- MWNTs;

as synthesized;

Nano Letters, Vol.9, No.4, 1527 (2009)

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Graphene nanoribbons from carbon nanotubes

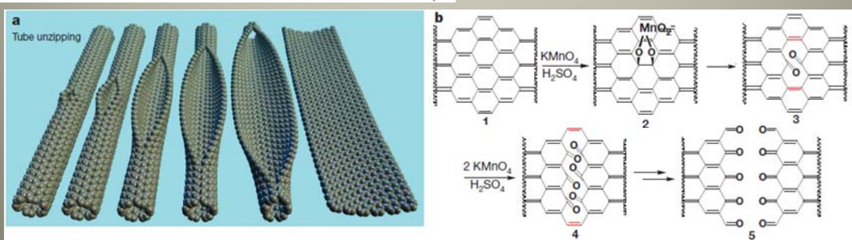
nature Vol 458|16 April 2009|doi:10.1038/nature07872

LETTERS

Longitudinal unzipping of carbon nanotubes to form graphene nanoribbons

Dmitry V. Kosynkin[1], Amanda L. Higginbotham[1], Alexander Sinitskii[1], Jay R. Lomeda[1], Ayrat Dimiev[1], B. Katherine Price[1] & James M. Tour[1,2,3]

... a simple solution-based oxidative process for producing a nearly 100% yield of nanoribbon structures by lengthwise cutting and unravelling of multiwalled carbon nanotube (MWCNT) side walls. ... Ribbon structures with high water solubility are obtained. Subsequent chemical reduction of the nanoribbons from MWCNTs results in restoration of electrical conductivity.



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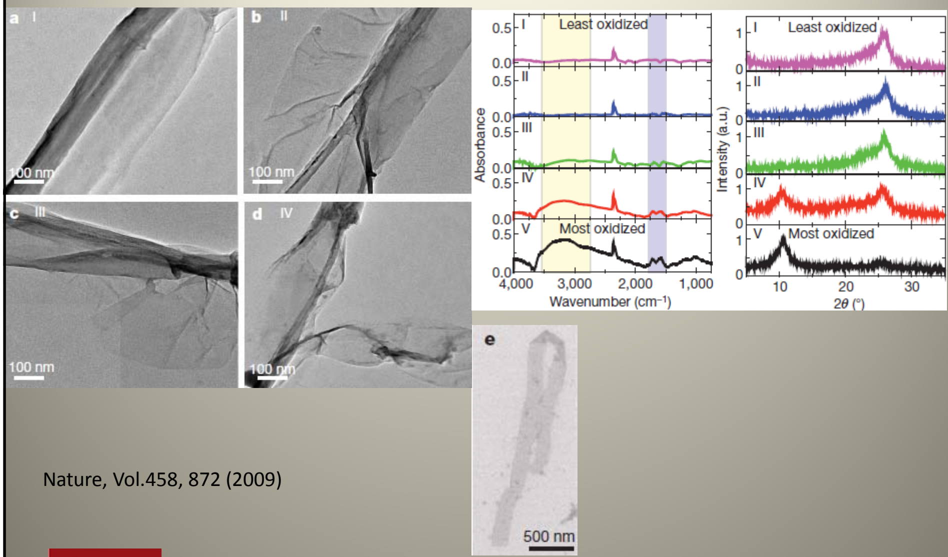
Graphene nanoribbons from carbon nanotubes



Nature, Vol.458, 872 (2009)

Graphene nanoribbons from carbon nanotubes

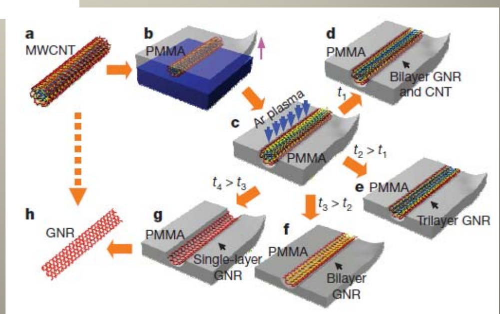
Vol 458 | 16 April 2009 | doi:10.1038/nature07919 nature

LETTERS

Narrow graphene nanoribbons from carbon nanotubes

Liying Jiao[1]*, Li Zhang[1]*, Xinran Wang[1], Georgi Diankov[1] & Hongjie Dai[1]

… unzipping multi-walled carbon nanotubes by plasma etching of nanotubes partly embedded in a polymer film. The GNRs have smooth edges and a narrow width distribution (10–20 nm). Raman spectroscopy and electrical transport measurements reveal the high quality of the GNRs.



Making GNRs from CNTs: **a**, A pristine MWCNT. **b**, The MWCNT was deposited on a Si substrate and then coated with a PMMA film. **c**, The PMMA–MWCNT film was peeled from the Si substrate, turned over and then exposed to an Ar plasma.

Graphene nanoribbons from carbon nanotubes

Atomic force microscopy

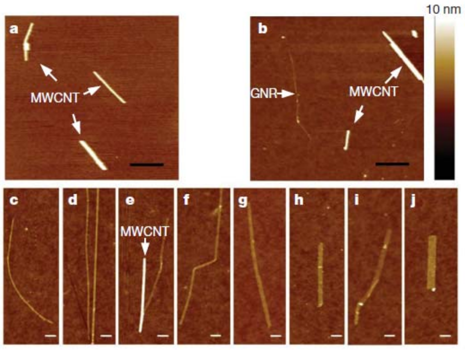
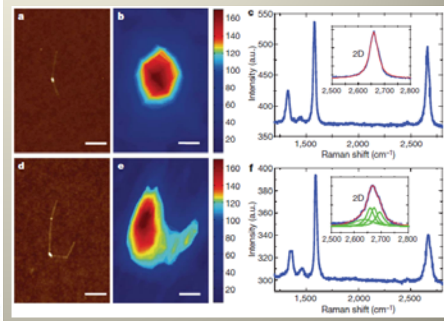


Figure 2 | Images of GNRs converted from MWCNTs. **a**, An AFM image of raw MWCNTs dispersed on a Si substrate. **b**, An image of the substrate after the GNR conversion process, showing coexistence of MWCNTs and GNRs. Scale bars, 1 μm. **c–j**, Single- or few-layer GNRs of different widths and heights: respectively 7 and 1.8 nm (**c**), 8 and 1.8 nm (left, **d**), 13 and 2.0 nm (right, **d**), 15 and 0.9 nm (**e**), 17 and 1.0 nm (**f**), 25 and 1.1 nm (**g**), 33 and 1.4 nm (**h**), 45 and 0.8 nm (**i**) and 51 and 1.9 nm (**j**). Scale bars, 100 nm. The height scale for all the AFM images is 10 nm. In **e**, an arrow points to a leftover MWCNT.

Confocal Raman mapping



Nature, Vol.458, 877 (2009)

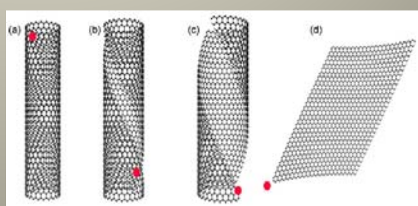
Graphene nanoribbons from carbon nanotubes

Applied Catalysis A: General 371 (2009) 22–30
Contents lists available at ScienceDirect
Applied Catalysis A: General
journal homepage: www.elsevier.com/locate/apcata

Catalytic unzipping of carbon nanotubes to few-layer graphene sheets under microwaves irradiation

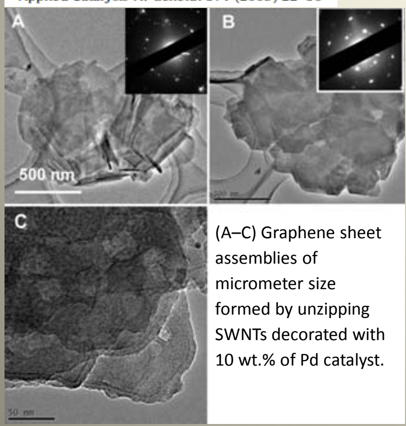
Izabela Janowska [a,*], Ovidiu Ersen [b], Timo Jacob [c], Philippe Vennégues [d], Dominique Bégin [a], Marc-Jacques Ledoux [a], Cuong Pham-Huu [a]

Catalytic unzipping of single-, double-, and multi-walled carbon nanotubes (SWNTs, DWNTs, and MWNTs), in the presence of Pd nanoparticles and an oxygen-containing liquid medium, to yield the few-layer graphene sheets, was performed under microwaves irradiation. In this unzipping process, the palladium particles act as a pair of scissors to cut the nanotube lengthways. Theoretical simulations with Reactive Forcefields confirm that the presence of Pd nanocatalysts and oxygen next to vacancies facilitate the unzipping process to form graphene from nanotube by significantly lowering the corresponding energy barrier.

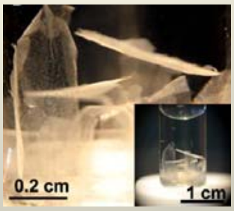


Graphene nanoribbons from carbon nanotubes

Applied Catalysis A: General 371 (2009) 22–30



(A–C) Graphene sheet assemblies of micrometer size formed by unzipping SWNTs decorated with 10 wt.% of Pd catalyst.



Optical photos showing the millimeter size graphene sheet assembly after partial reduction with $NaBH_4$ in a mixture of water–toluene at 80 °C.

(A and B) TEM micrographs of the graphene sheets obtained from MWNTs unzipping.

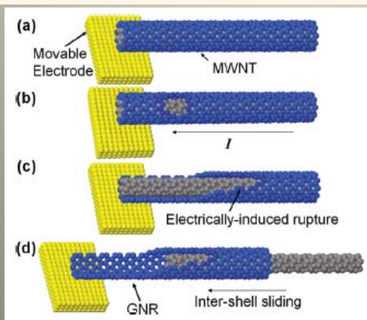
Graphene nanoribbons from carbon nanotubes

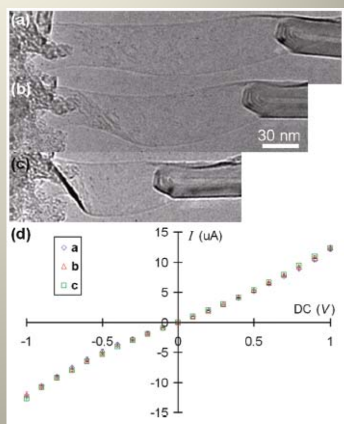
Graphene Nanoribbons Obtained by Electrically Unwrapping Carbon Nanotubes

Kwanpyo Kim, Allen Sussman, and A. Zettl*

ACSNANO VOL. 4 • NO. 3 • 1362–1366 • 2010

ABSTRACT We describe a clean method of graphene nanoribbon (GNR) extraction from multiwall carbon nanotubes (MWNTs), performed in a high vacuum, nonchemical environment. Electrical current and nanomanipulation are used to unwrap a portion of the MWNT and thus produce a GNR of desired width and length. The unwrapping method allows GNRs to be concurrently characterized structurally *via* high-resolution





Flexing of a GNR and concurrent electrical measurement. The two-terminal conductance stays the same even with the dramatic mechanical deformation of the GNR.

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Graphene nanoribbons from carbon nanotubes

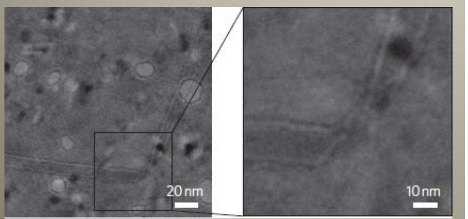
nature nanotechnology LETTERS
PUBLISHED ONLINE: 4 APRIL 2010 | DOI: 10.1038/NNANO.2010.54

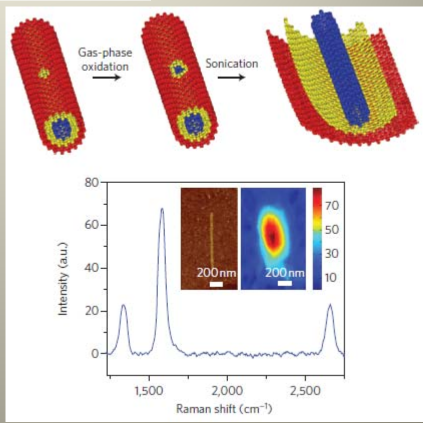
Facile synthesis of high-quality graphene nanoribbons

Liying Jiao, Xinran Wang, Georgi Diankov, Hailiang Wang and Hongjie Dai*

Here, we show that pristine few-layer nanoribbons can be produced by unzipping mildly gas-phase oxidized multiwalled carbon nanotubes using mechanical sonication in an organic solvent.

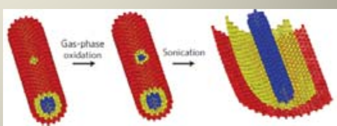
The CNT were calcinated in air at 500 °C to remove impurities and oxidize MWNTs at defect sites and ends. Then they were dispersed in a 1,2-dichloroethane organic solution of poly(m-phenylenevinylene-co-2,5-dioctoxy-p-phenylenevinylene) (PmPV) by sonication

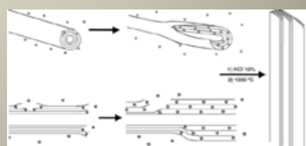




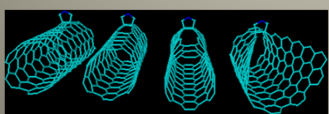
Graphene nanoribbons from carbon nanotubes

- What triggers the unzipping?
- How does it happen?
- What type of edges are formed?





Alexander Sinitskii, James M. Tour / November 2010



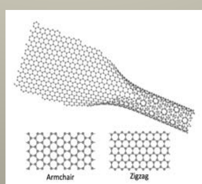
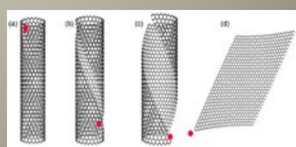


Image: Lawrence Berkeley National Laboratory

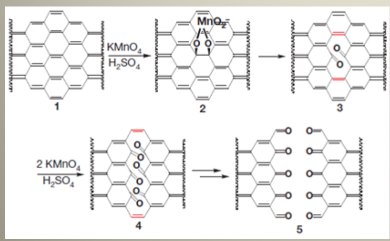


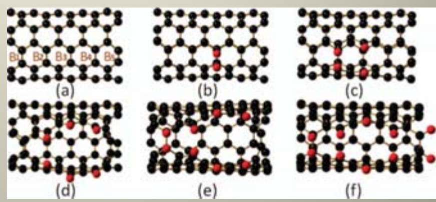
Graphene nanoribbons from carbon nanotubes

THE JOURNAL OF CHEMICAL PHYSICS 131, 031105 (2009)

Mechanism of carbon nanotubes unzipping into graphene ribbons

Norma L. Rangel,[1,2] Juan C. Sotelo,[2,a)] and Jorge M. Seminario[1,2,3,b)]





ab initio density functional theory molecular orbital calculations:

- Pairs of O atoms from MnO_4^{2-} bind and break the internal C-C bonds of the CNT;
- The stretching of the bonds involved in the first reaction weakens the neighbor parallel bonds, becoming vulnerable to the next oxygen pair attack;
- The results suggest that unzipped CNTs end up in zigzag edge ribbons.

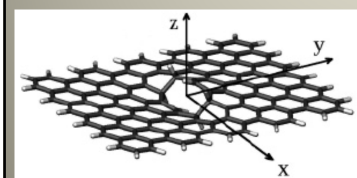
Graphene nanoribbons from carbon nanotubes

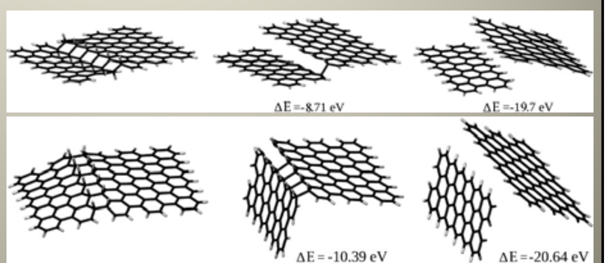
PHYSICAL REVIEW B 83, 195442 (2011)

Zipping and unzipping of nanoscale carbon structures

Julia Berashevich and Tapash Chakraborty

Simulated the zipping and unzipping of carbon systems in vacuum using quantum chemistry methods





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Graphene nanoribbons from carbon nanotubes

Unzipping of functionalized carbon nanotubes:

- Functionalization of CNT
- STM observation of CNT unzipping
- Formation of GNR in solution

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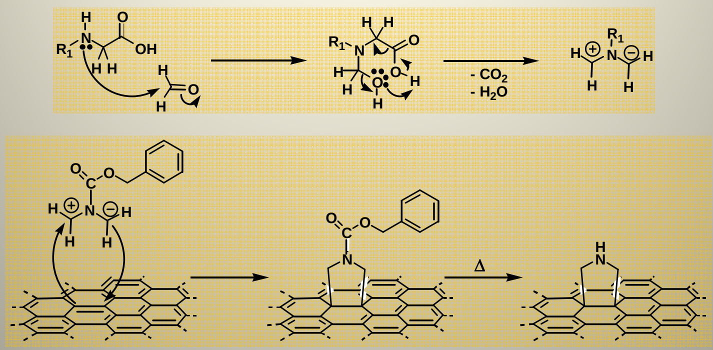
CNT functionalization: the 1,3-dipolar cycloaddition reaction



V. Georgakilas, K. Kordatos, M. Prato, D. M. Guldi, M. Holzinger, A. Hirsch, *J. Am. Chem. Soc*. 2002, 124, 760

R. Araújo, M. C. Paiva, M. F. Proença, C. J. R. Silva, *Composites Science and Technology*, 2007, 67, 806-810

- In solution (DMF)
- Reaction time – 5 days
- In solvent-free conditions
- With heat: Reaction time – 2 to 7 hours
- With microwaves: a few seconds

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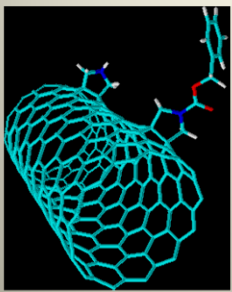
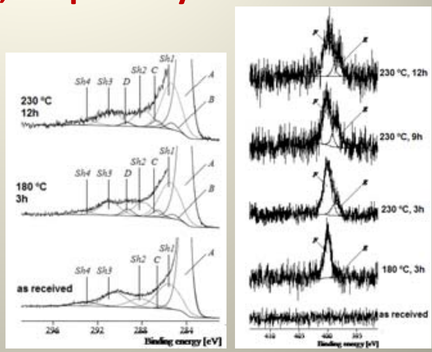
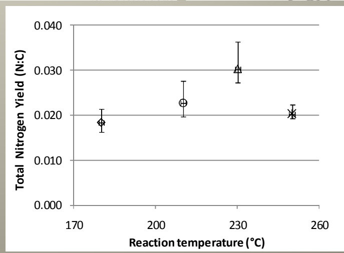
CNT functionalization: the 1,3-dipolar cycloaddition reaction





- Thermogravimetric analysis
- X-ray photoelectron spectroscopy
- Scanning tunneling microscopy
- Infrared spectroscopy



Controlled Functionalization of Carbon Nanotubes by a Solvent-free Multicomponent Approach
VOL. 4 • NO. 12 • 7379–7386 • 2010 ACS NANO
M. C. Paiva, F. Simon, R. M. Novais, T. Ferreira, M. F. Proença, W. Xu, F. Besenbacher

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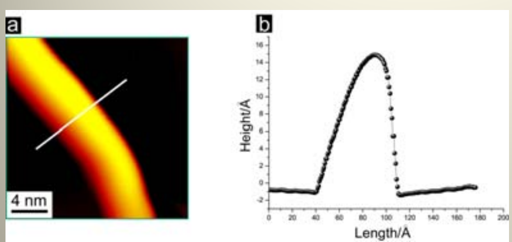
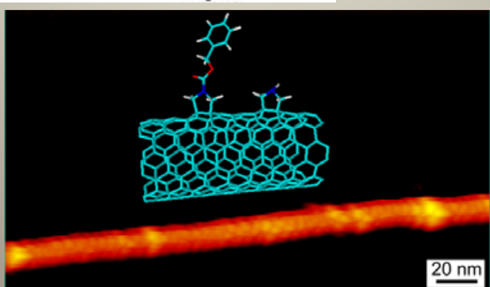
The 1,3-dipolar cycloaddition reaction - STM



- UHV chamber (3×10^{-10} Torr), Pt/Ir tip
- constant current mode
- Typical bias voltage and tunneling current:
  - ✓ nanometer scale - 1000-2000 mV and 0.8-1.2 nA
  - ✓ Atomic scale - 30-100 mV and 1.5-2.0 nA



iNANO Research Center at Aahrus University, DK

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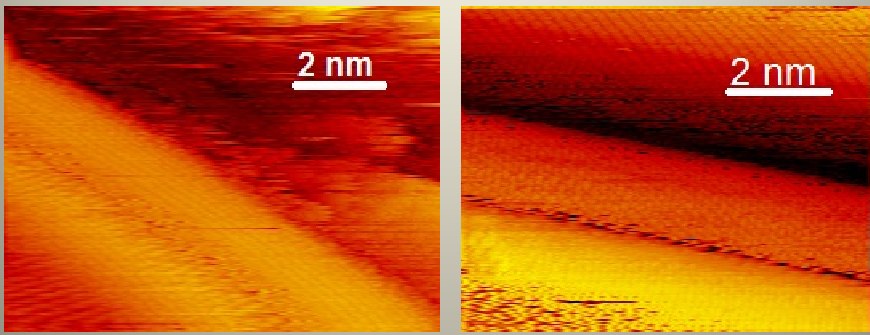
Graphene nanoribbons from carbon nanotubes



Instability of the outer graphene layer of the functionalized CNT under atomic scale STM imaging conditions

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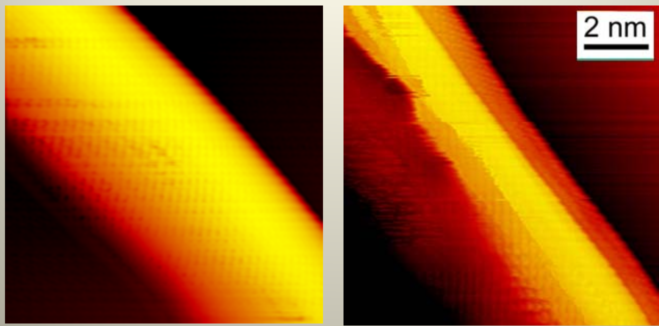
Graphene nanoribbons from carbon nanotubes



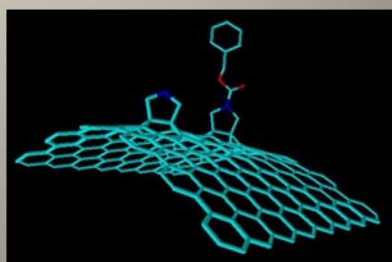
- **constant current mode**
- **Typical bias voltage and tunneling current:**

  Atomic scale - 30-100 mV and 1.5-2.0 nA


NANO LETTERS DOI: 10.1021/nl100240n | Nano Lett. 2010, 10, 1764–1768

Unzipping of Functionalized Multiwall Carbon Nanotubes Induced by STM

M. Conceição Paiva,*,† Wei Xu,†,‡ M. Fernanda Proença,|| Rui M. Novais,† Erik Lægsgaard,† and Flemming Besenbacher*,†



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Graphene nanoribbons from carbon nanotubes, in solution

CNT functionalization

↓

“Exfoliation” in solution

↓

Filtration

↓

Analysis of the solution and of the solid obtained by evaporation on a surface

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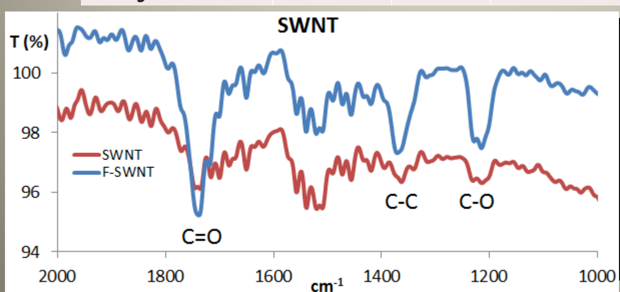
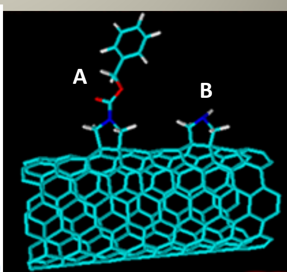
Carbon nanotube functionalization

Carbon nanotubes	Diameter (nm)	Purity	Functionalization conditions	TGA wt loss (%)
SWNT HiPco ***Carbon Nanotechnology Inc.***	1 - 2	>90%	200 °C; 14h	21
MWNT_NC3100 ***Nanocyl***	7 - 10	>95%	250 °C; 3h	14
MWNT_NC7000 ***Nanocyl***	7 - 10	90%	230 °C; 5h	17
MWNT_Aldrich ***Sigma Aldrich***	110 - 170	>90%	250 °C; 3h	17

A (SWNT HiPco)

B (MWNT_NC3100, MWNT_NC7000, MWNT_Aldrich)





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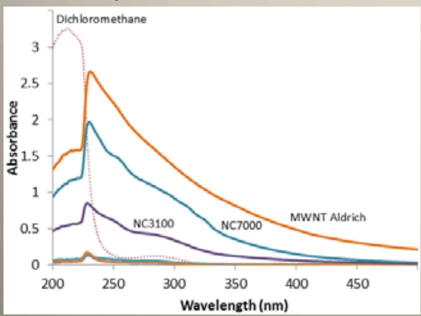
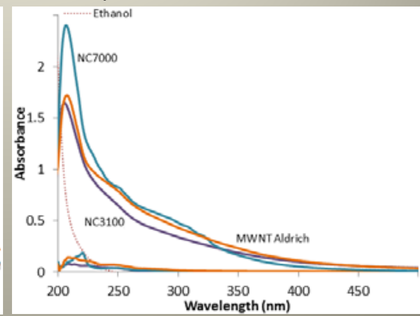
Graphene nanoribbons from carbon nanotubes

Solvent	λ_{max} (nm) SWNT	MWNT_NC3100	MWNT_NC7000	MWNT_Aldrich
Dichloromethane	230	229	229	230
Ethanol	-	207	207	207

UV-Vis spectra of GNR solutions in DCM



UV-Vis spectra of GNR solutions in Ethanol



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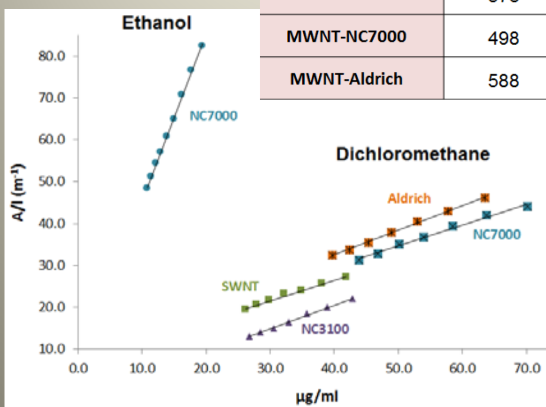
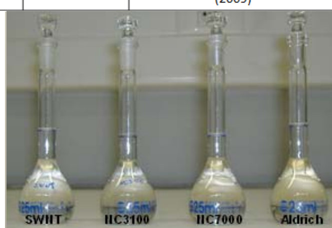
Graphene nanoribbons from carbon nanotubes

Graphene solution obtained from:	α_{DCM} (L g^{-1} m^{-1})	$\alpha_{Ethanol}$ (L g^{-1} m^{-1})	$\alpha_{Ref.}$ (L g^{-1} m^{-1})
SWNT- HiPCO	488	-	**2460 (660nm)** Hernandez, Y. et al. Nature 3, 563 (2008) **5300 (230 nm)** Wang, G., et al. Carbon 47, 68 (2009) **2400 (419nm)** Xu, B., et al. Adv. Mater. 21, 1275 (2009)
MWNT-NC3100	573	-	
MWNT-NC7000	498	3983	
MWNT-Aldrich	588	-	





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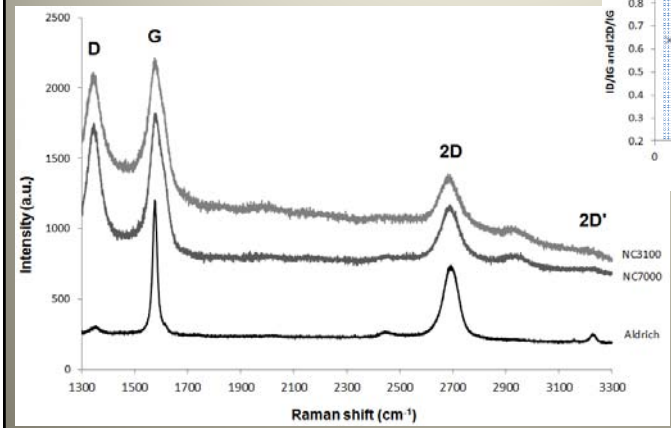
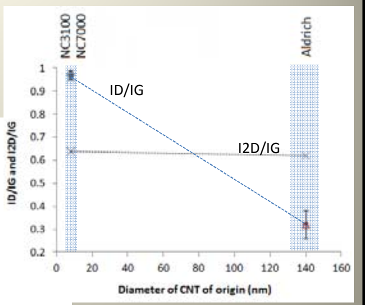
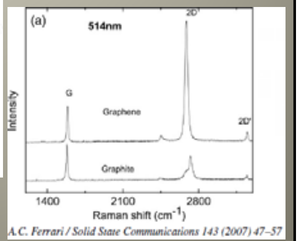
Graphene nanoribbons from carbon nanotubes

Raman spectra (532 nm) obtained for the GNR deposited by solvent evaporation (ethanol solutions) on a Si surface.







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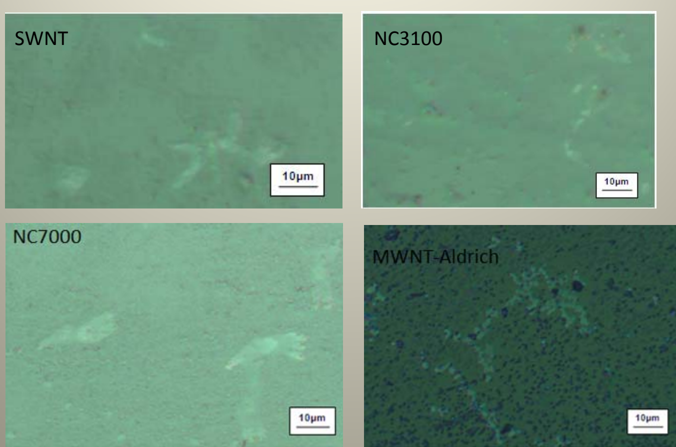
Graphene nanoribbons from carbon nanotubes

Optical micrographs of the GNR residue obtained by solvent evaporation on a quartz surface



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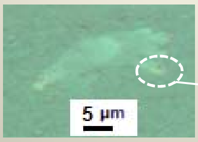
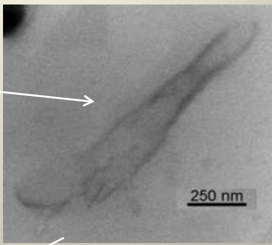
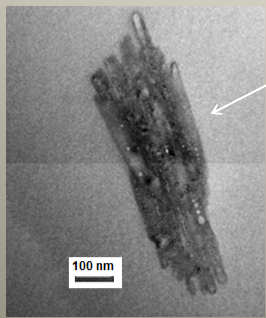
Graphene nanoribbons formed in DCM

Optical microscopy





Electron microscopy - STEM



Electron microscopy - TEM

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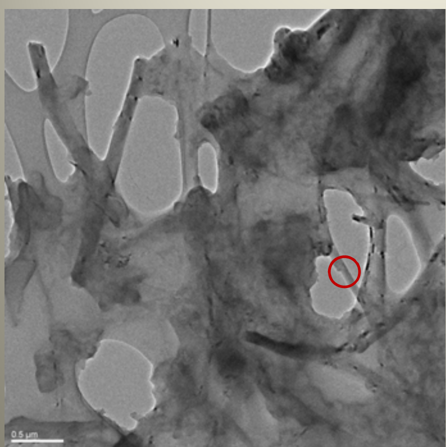
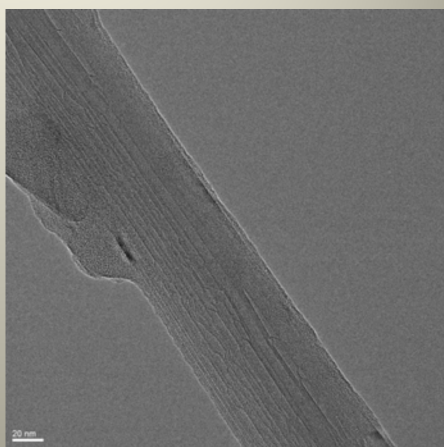
Graphene nanoribbons formed in ethanol

TEM of the GNR obtained from the MWNT from Aldrich





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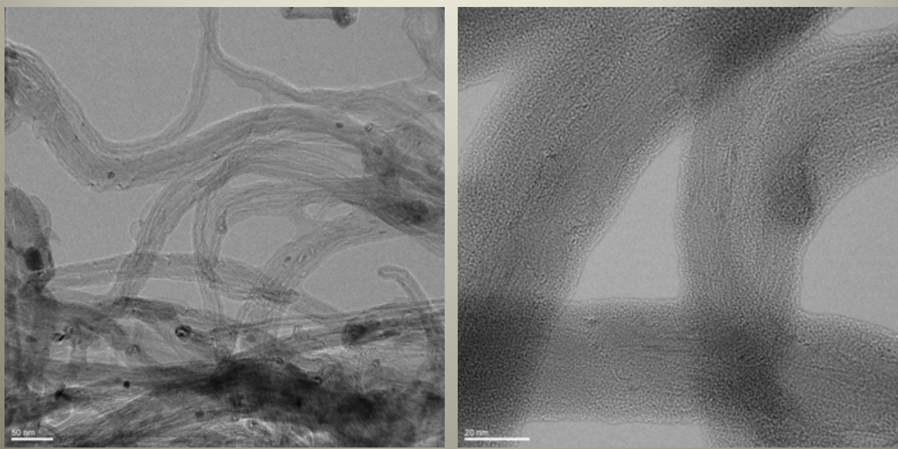
Graphene nanoribbons formed in ethanol

TEM of the GNR obtained from NC 3100



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Contributions to the work presented:

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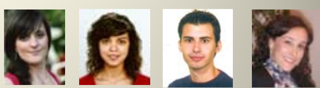
  Eunice Cunha
  Joana Fernandes
  Rui M. Novais
  Tânia Ferreira



- **Centro de Química U. Minho**

  CNT organic modification: M. Fernanda Proença, Rui Araújo

- **I3N/Physics of Semiconductors, Optoelectronics and disordered Systems**

  Raman analysis: Florinda Costa, António José Fernandes

- **iNANO Research Center at Aahrus University (Denmark)**

  STM analysis: Flemming Besenbacher, Erik Lægsgaard, Wei Xu

- **Leibniz Institute of Polymer Research, Dresden (Germany)**

  XPS analysis: Frank Simon

- **TEM facilities at CICECO, U. Aveiro** (Rui Silva, Marta Ferro)
- **TEM facilities at INMETRO, Rio de Janeiro**

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