Magnetism in carbon structures filled with ferromagnetic crystals: filling-control, phase-transitions and shell manipulation

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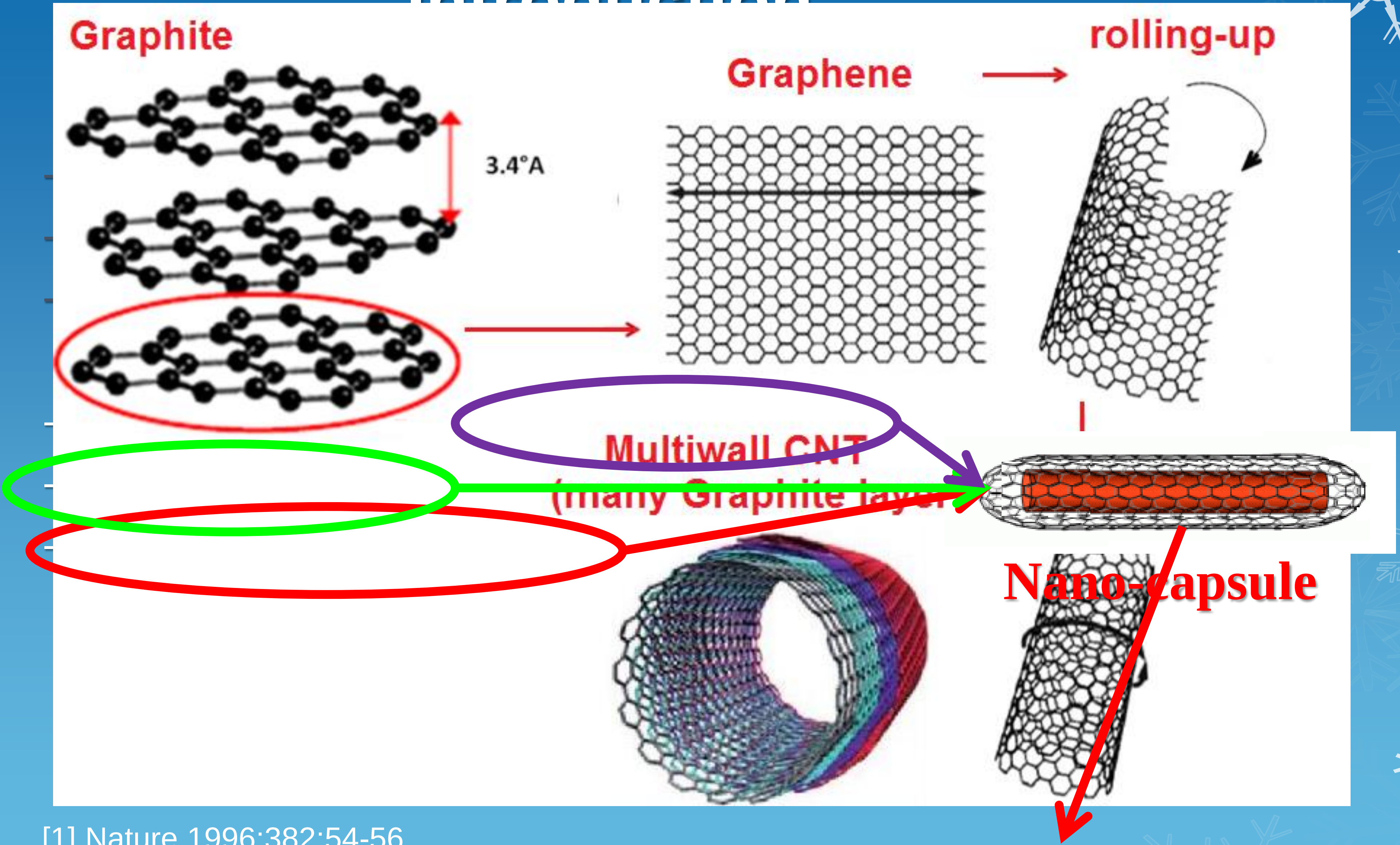
Outline

- Introduction of iron filled carbon nanotubes
- Applications of filled carbon nanotubes
- Problem of Conventional CVD methodology
- Perturbed vapour CVD:
- Substrate-Hole Approach
- Boundary Layer Approach
- Other solutions towards filling control: the role of Chlorine
- The filled CNT buckypaper
- Investigating phase transformations and enhancing magnetization
- Unusual magnetic transitions
- Sulfur as an additional filling promoter
- Ferromagnetically filled carbon foam, a carbon material fully filled with iron
- Mass Production through fusion of filled CNOs
- Manipulation of the carbon shells, the key role of sulfur
- Conclusion and Future Work
- Acknowledgments

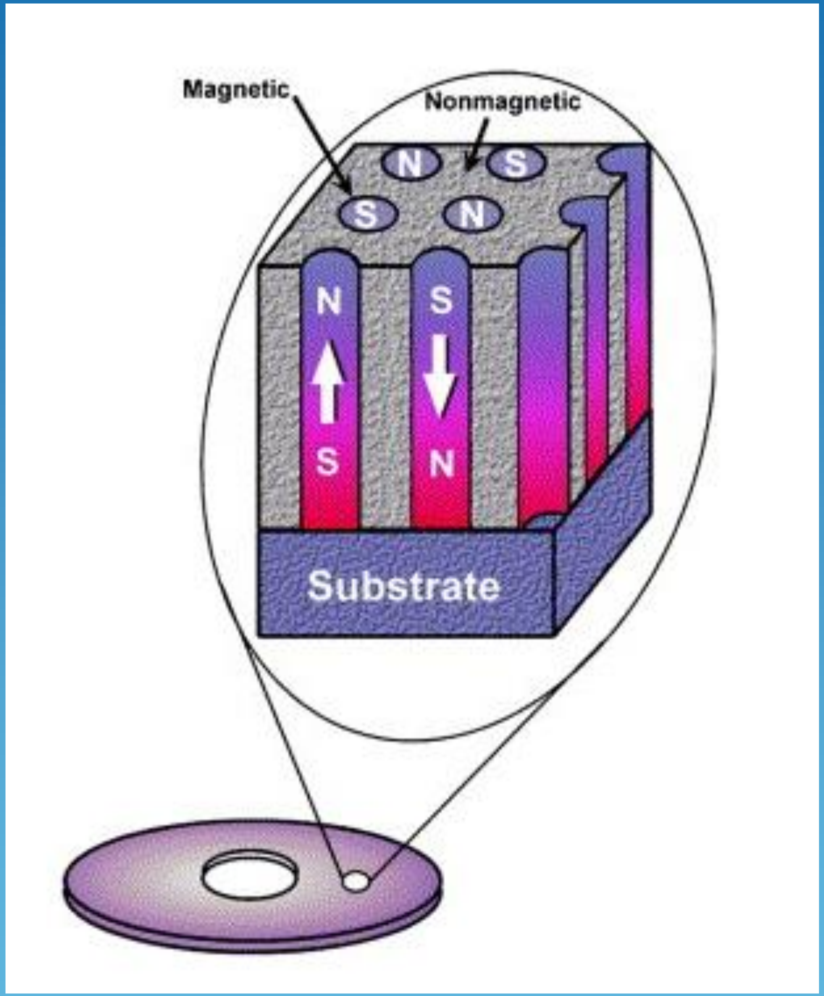
Introduction



Magnetic applications!

[1] Nature 1996;382:54-56
[2] Science 2000; 287(5453): 637-640
[3] Materials 2010; 3(8): 4387-4427

Possible Application as Quantum Disk systems



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Solid State Sciences

Volume 8, Issues 3–4, March–April 2006, Pages 303-320

Magnetism in Fe-based and carbon nanostructures: Theory and applications

H. Terrones, F. López-Urías, E. Muñoz-Sandoval, J.A. Rodríguez-Manzo, A. Zamudio, A.L. Elías, M. Terrones

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Possible Applications as **multifunctional nano-container**

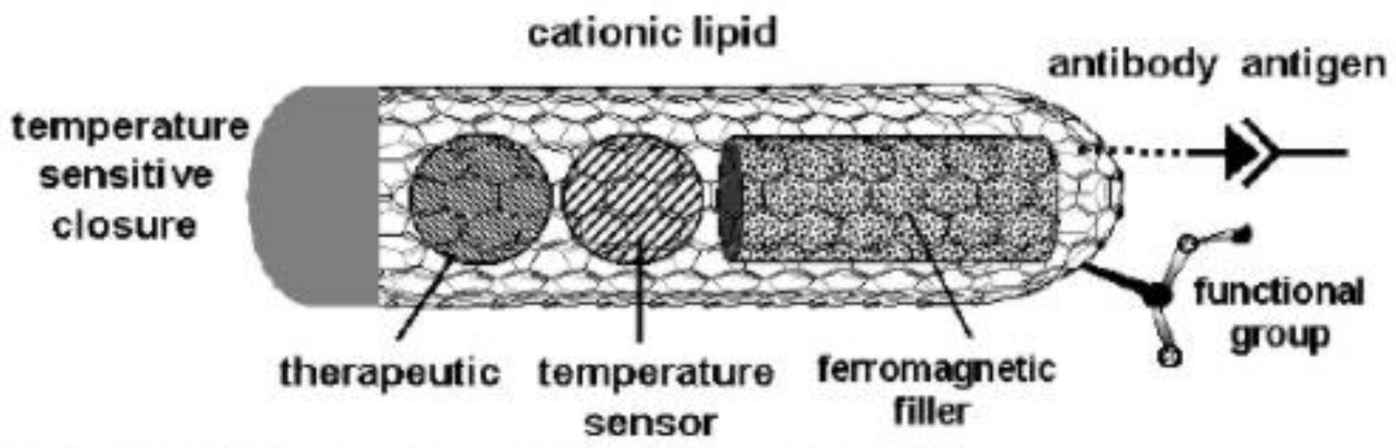
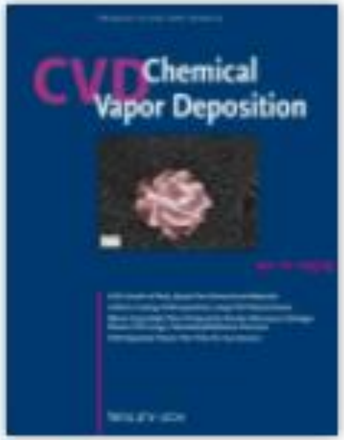


Fig. 9. Schematic diagram of a multifunctional nanocontainer [3].



Volume 12, Issue 6

Special Issue: Carbon Nanotubes

June, 2006

Pages 380-387

Chemical Vapor Deposition

Full Paper | Full Access

Synthesis, Properties, and Applications of Ferromagnetic-Filled Carbon Nanotubes

A. Leonhardt, S. Hampel, C. Müller, I. Mönch, R. Koseva, M. Ritschel, D. Elefant, K. Biedermann, B. Büchner

First published: 19 June 2006 | https://doi.org/10.1002/cvde.200506441 | Citations: 102

Challenges towards applications and device fabrication

- Control of nanotube filling rate which is generally limited by the precursor stoichiometry

- Phase Control: Nanowires comprise multiple phases of Fe_3C, α-Fe and exchange coupled α-Fe/γ-Fe

- Control of Nanowires anisotropy (K) requires phase control

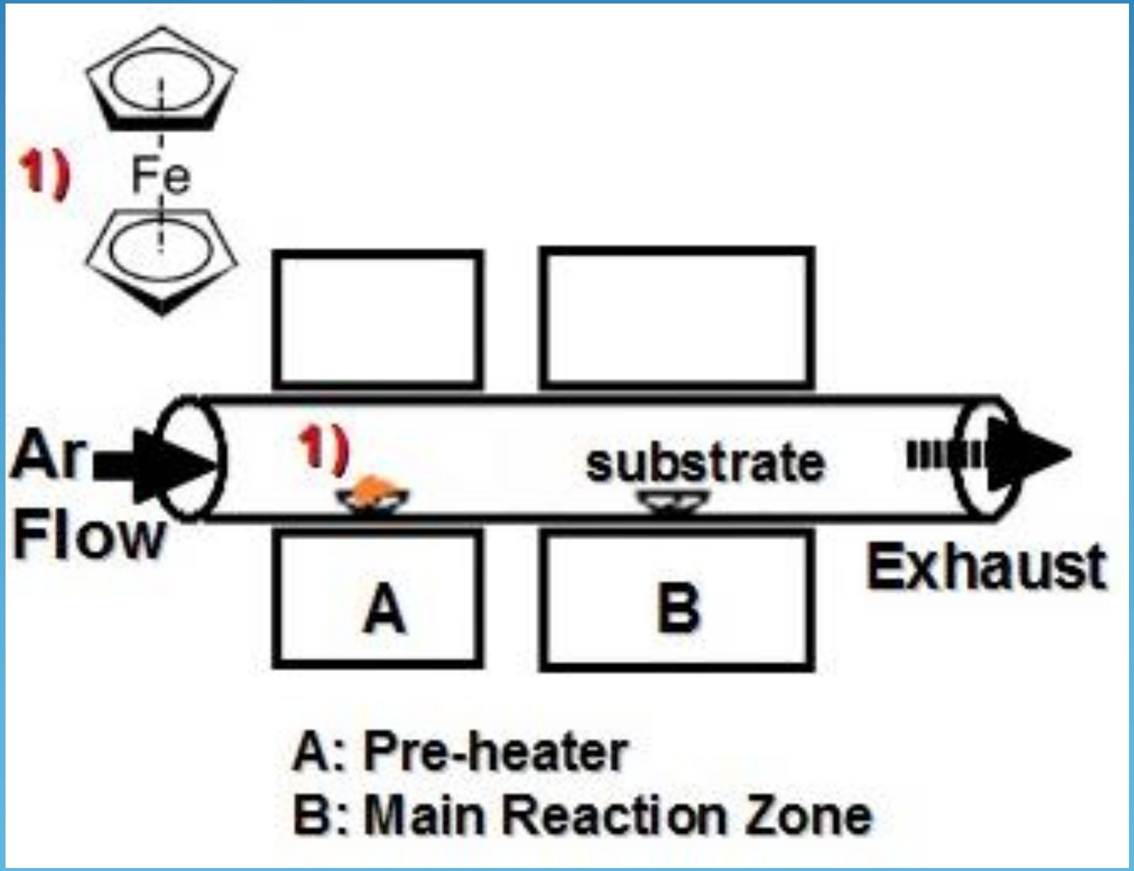
- Control of the sharpness of the interface

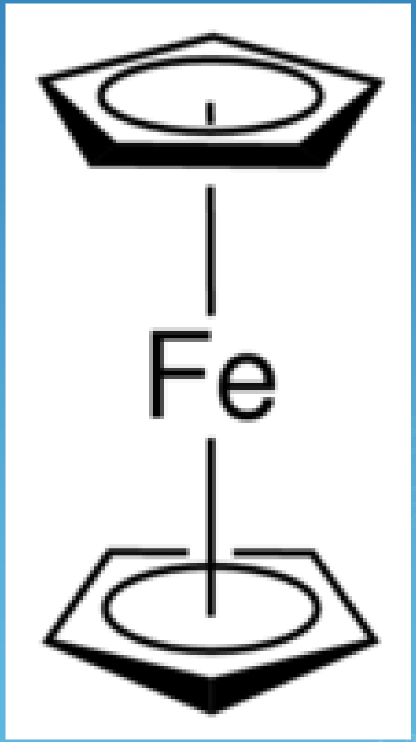
- Manipulation of the shell-capsule properties for device applications

CNT as capsule for ferromagnetic nanowires

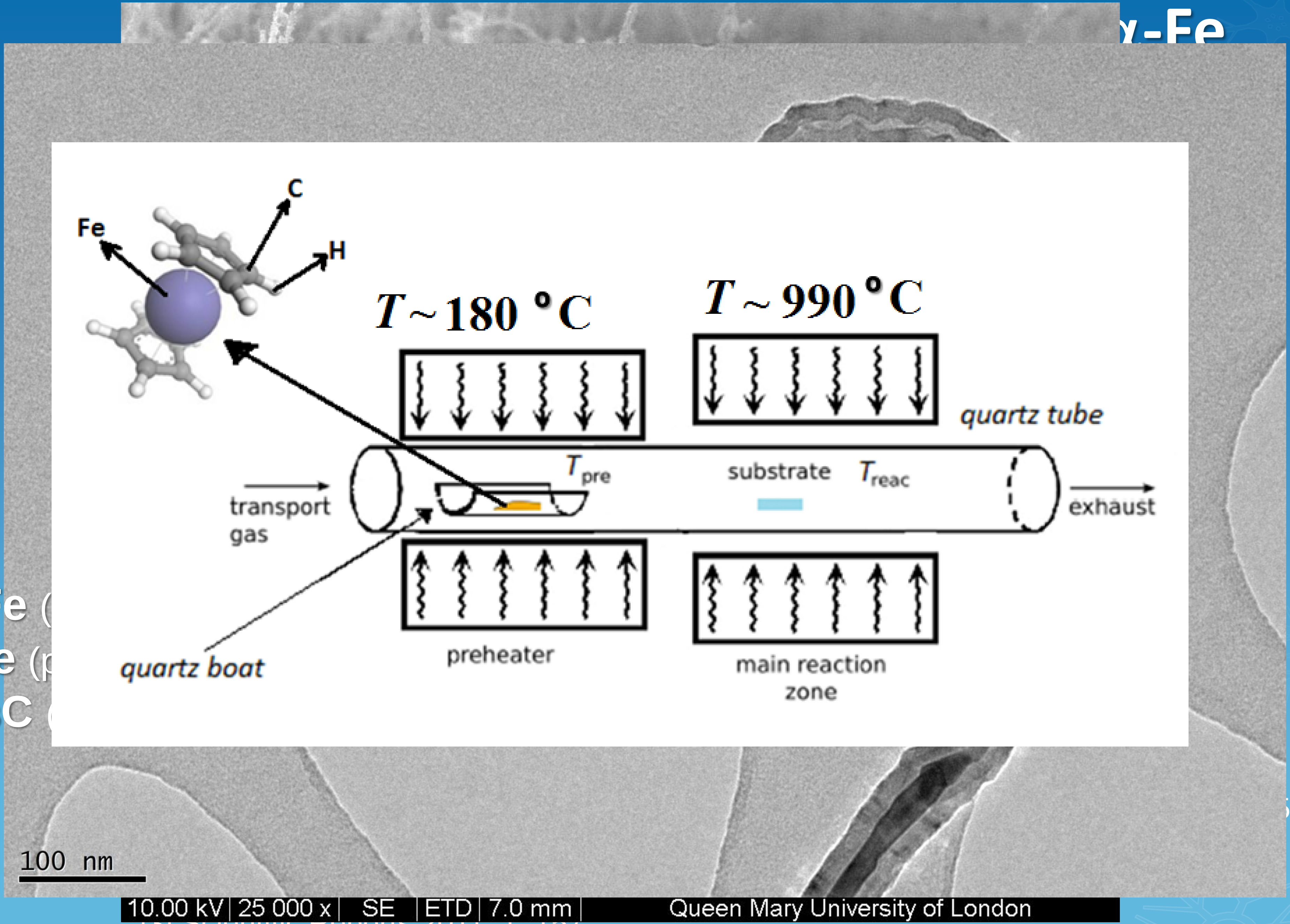
- **Laminar flow approaches**

- Based on the conventional pyrolysis of ferrocene (relatively low filling rates)





γ-Fe



- α-Fe (
– γ-Fe (p
- Fe_3C (

501.

[3] Scientific Reports 2011, 1: 157.

Perturbed-Vapour CVD

- **Early solutions developed towards filling-rate control:**
- Two approaches:

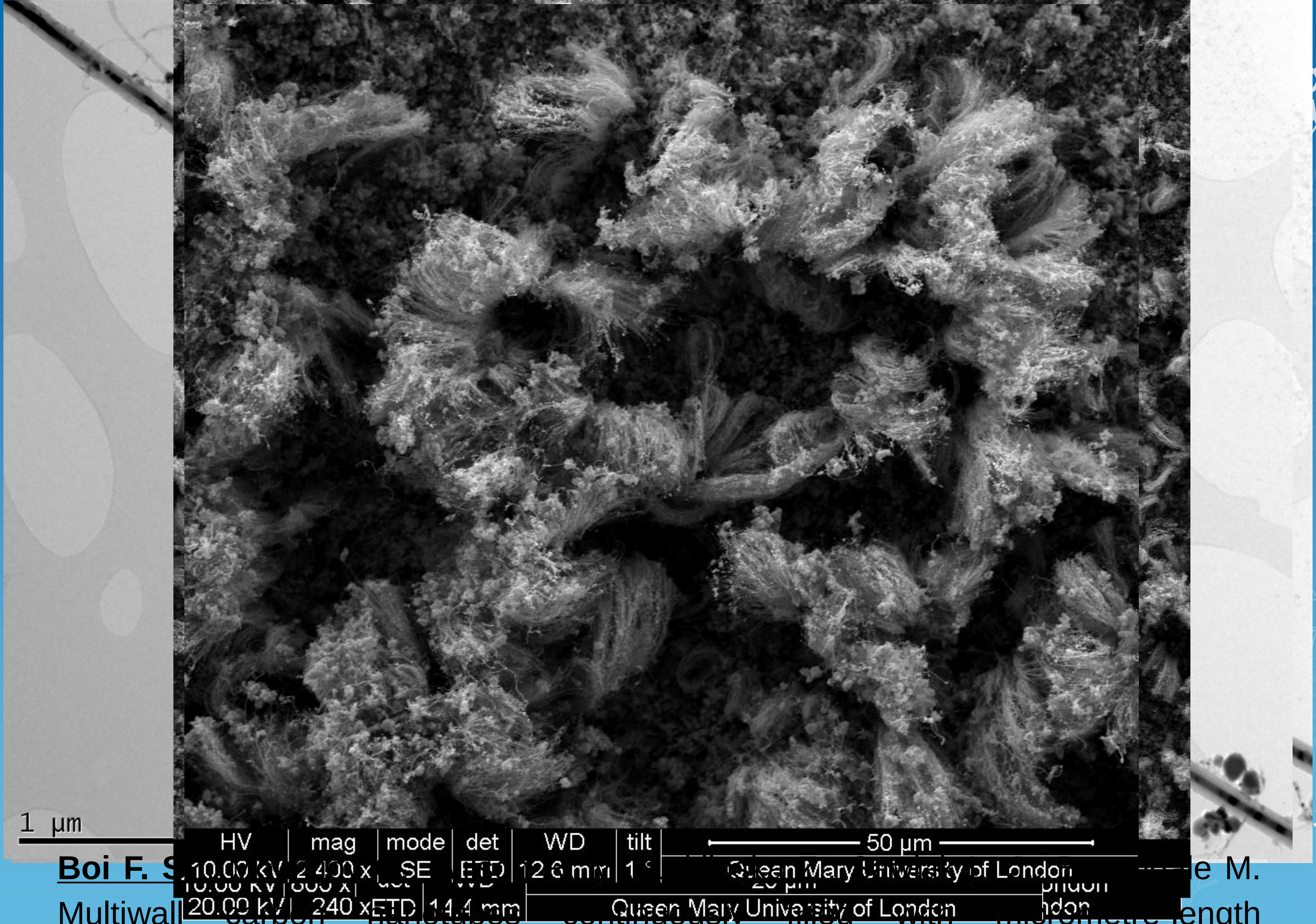
1) **Substrate-hole approach:**
2) **Boundary-layer approach:**

- Perturbed-vapour CVD method

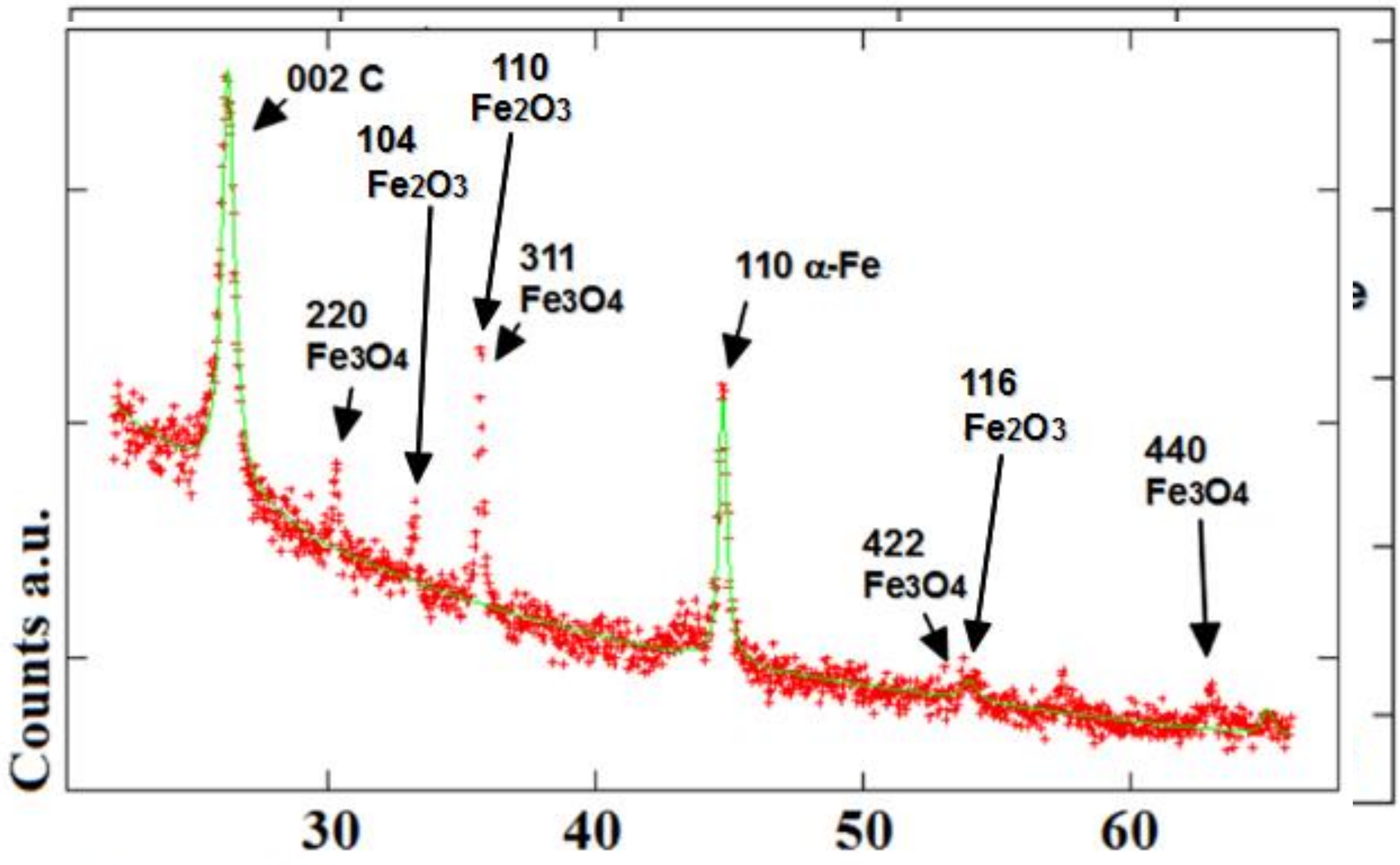
Involves the creation of local perturbation into a ferrocene-Ar vapour flow

Boi F. S.*, Mountjoy G., Wilson R. M., Luklinska Z., Sawiak L. J., Baxendale M. Multiwall carbon nanotubes continuously filled with micrometre-length ferromagnetic α-Fe nanowires. Carbon 2013; 64: 351-8.

Boi F. S., Mountjoy G., Baxendale M*. Boundary layer chemical vapor synthesis of self-organized radial filled-carbon-nanotube structures.Carbon 2013; 64: 516-26.



Boi F. S … e M. Multiwall carbon nanotubes continuously filled with micrometre length ferromagnetic α-Fe nanowires. Carbon 2013; 64: 351-8.



Boi F. S.*, Mountjoy G., Wilson R. M., Luklinska Z., Sawiak L. J., Baxendale M. Multiwall carbon nanotubes continuously filled with micrometre-length ferromagnetic α-Fe nanowires. Carbon 2013; 64: 351-8.

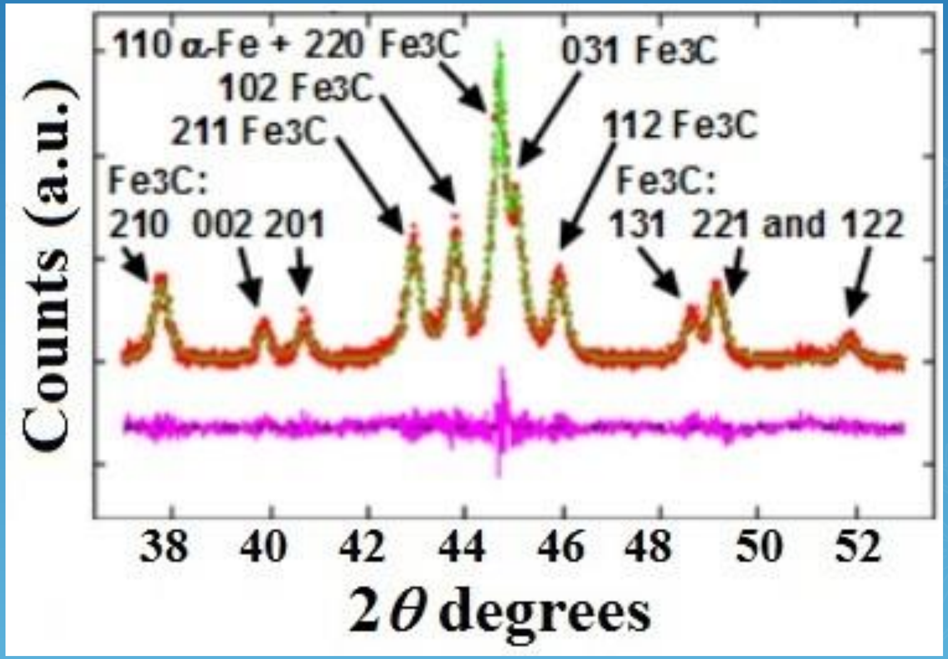
Particularly high filling rates

Fast cooling allows Fe_3C encapsulation

XRD diffractogram (red line) and Rietveld refinement (green line) of the Fe_3C single crystals quenched at 950 °C revealing the following phase abundances: 95% of Fe_3C and 5% of α-Fe. Each peak is indicated with the labelled reflection of the corresponding phase. The pink line corresponds to the difference between the XRD diffractogram and the Rietveld theoretical model.

Unpublished (in preparation)

quenched at 950 °C

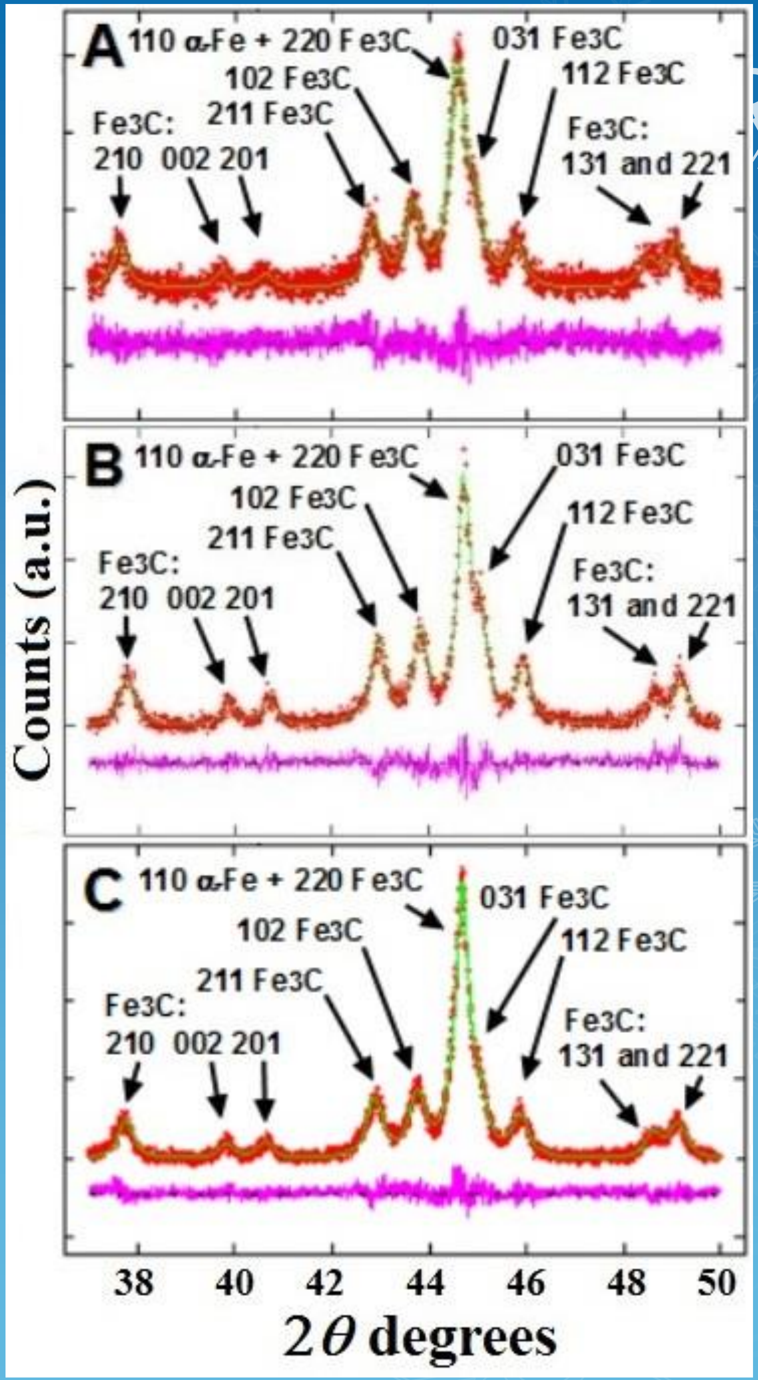


XRD diffractogram (red line) and Rietveld refinement (green line) of the Fe_3C single crystals quenched at the furnace temperatures of 830 ºC (A), 800 ºC (B) and 770 ºC (C) with a cooling time of ~ 10 min. Each peak is indicated with the labelled reflection of the corresponding phase.
The pink line corresponds to the difference between the XRD diffractogram and the Rietveld theoretical model.
The following phase abundances are extracted: 95% of Fe_3C and 5% of α-Fe (A), 98% of Fe_3C and 2% of α-Fe (B) and 85% of Fe_3C and 15% of α-Fe (C).

830 ºC

800 ºC

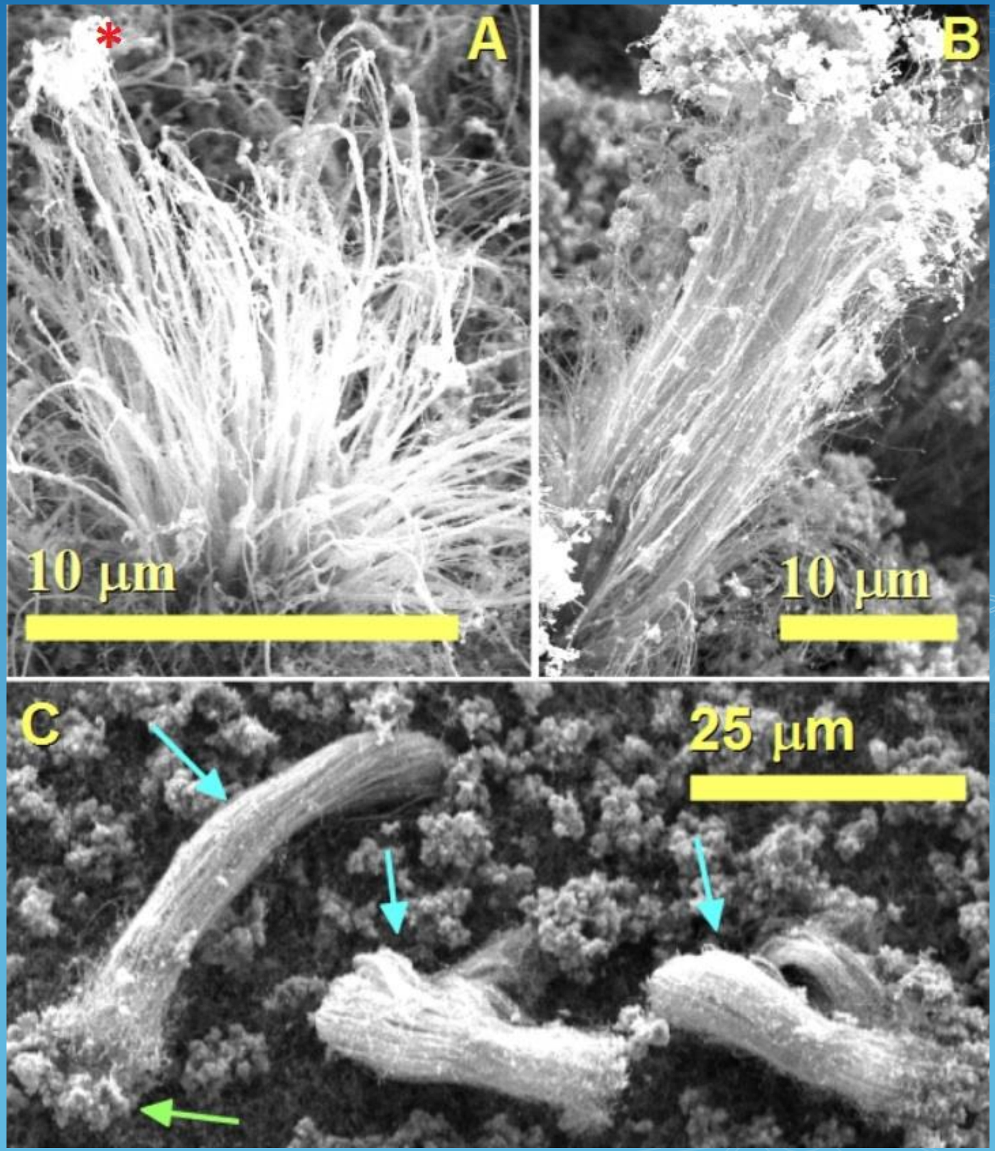
770 ºC



Unpublished (in preparation)

Morphological control

Unpublished (in preparation)

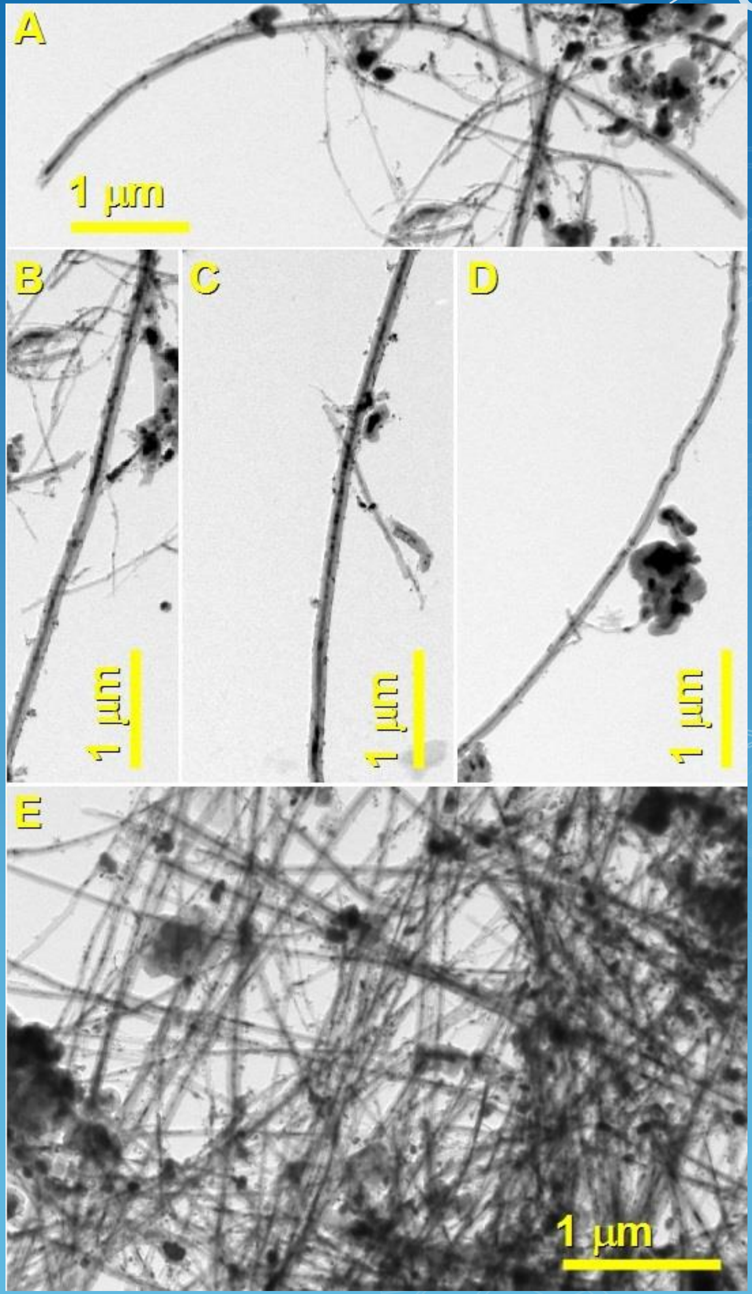


SEM micrographs of typical flower-like structures obtained with ferrocene sublimation-temperatures of 140 ºC (A), 180 ºC (B) and 500 ºC (C). Note the progressive change of the flower morphology from open-like to closed structures. The red star in A and the green arrow in C indicates catalyst particles connected to a MWCNT-flower. The cyan arrows indicate typical closed-flower-like structures.

Morphological control

Unpublished (in preparation)

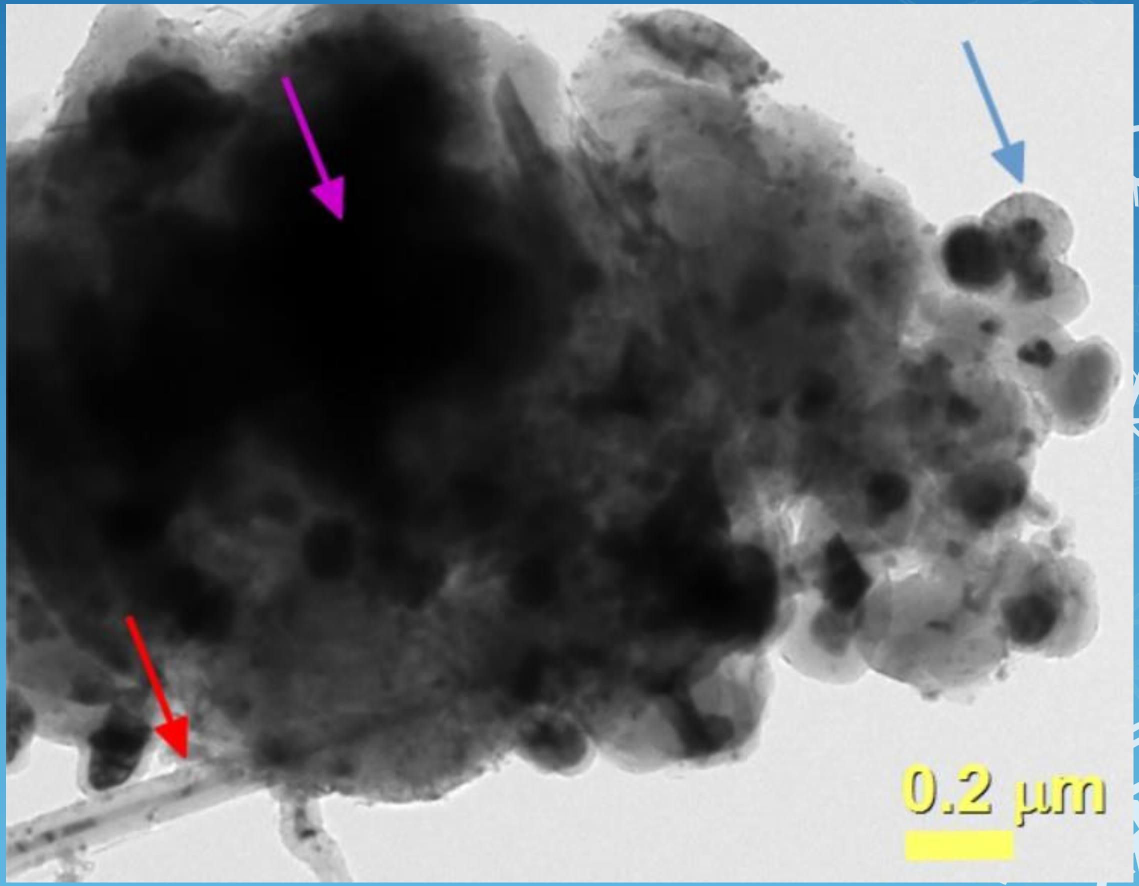
- Typical examples of individual continuously filled CNTs obtained with evaporation temperature of 500 ºC. are shown in A-D while a micrograph including multiple continuously filled CNTs structures is shown in E.



Morphological control

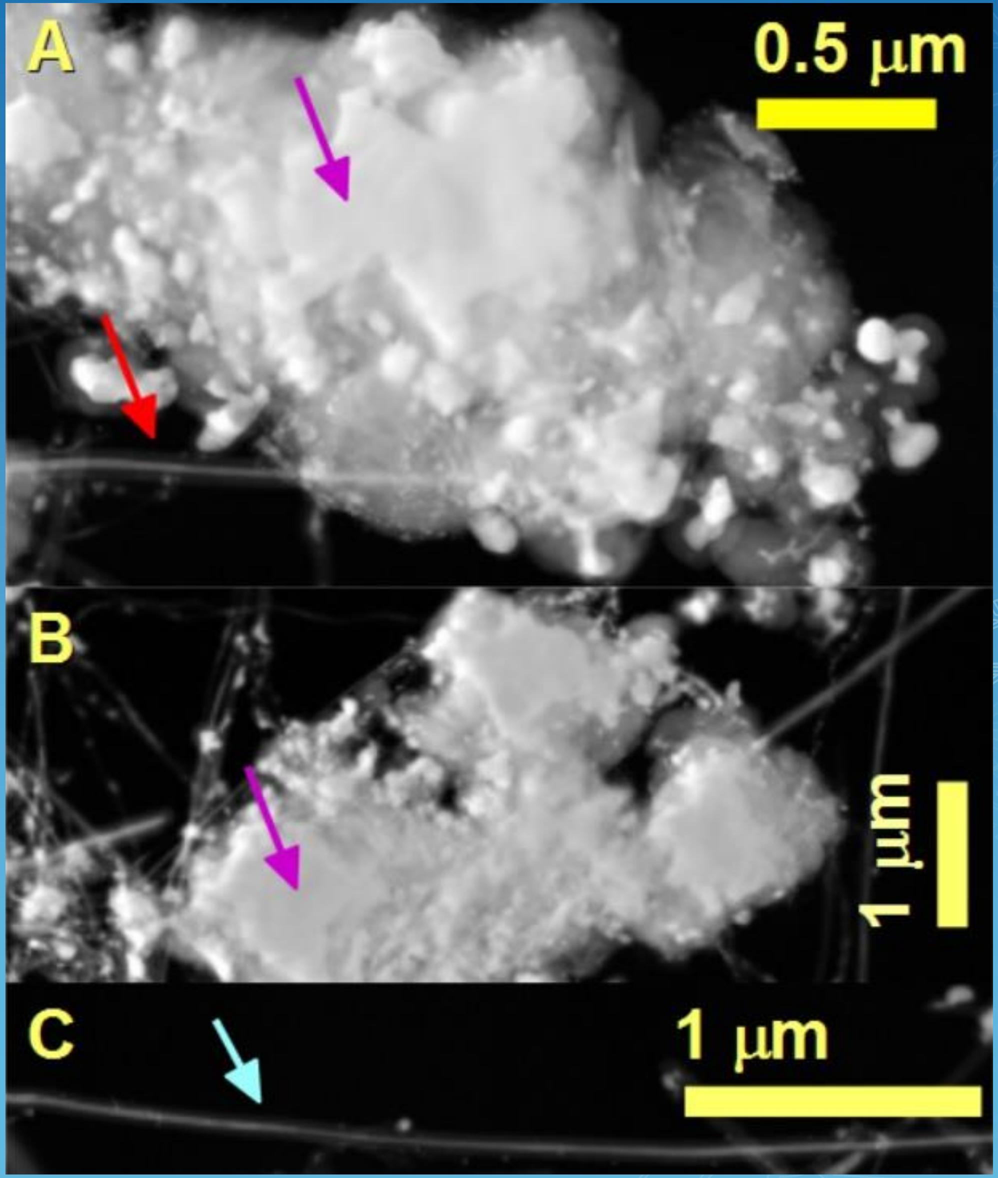
TEM micrograph showing a large catalyst particle (possibly resulting from homogenous nucleation) with high metal content (magenta arrow) connected to a continuously filled CNT (red arrow). The formation of onion-like structures is also found (blue arrow).

Unpublished (in preparation)



Morphological control

Unpublished (in preparation)



STEM micrographs showing in A an example of typical continuously filled CNT (indicated by the red arrow) connected to a large catalyst particle (see magenta arrow). In B and C two more examples of large catalyst particle (see magenta arrow) and continuously filled CNT (see cyan arrow) are shown. The bright regions in A, B and C indicate the regions of high metal content.

New approaches: Perturbed-Vapour CVD

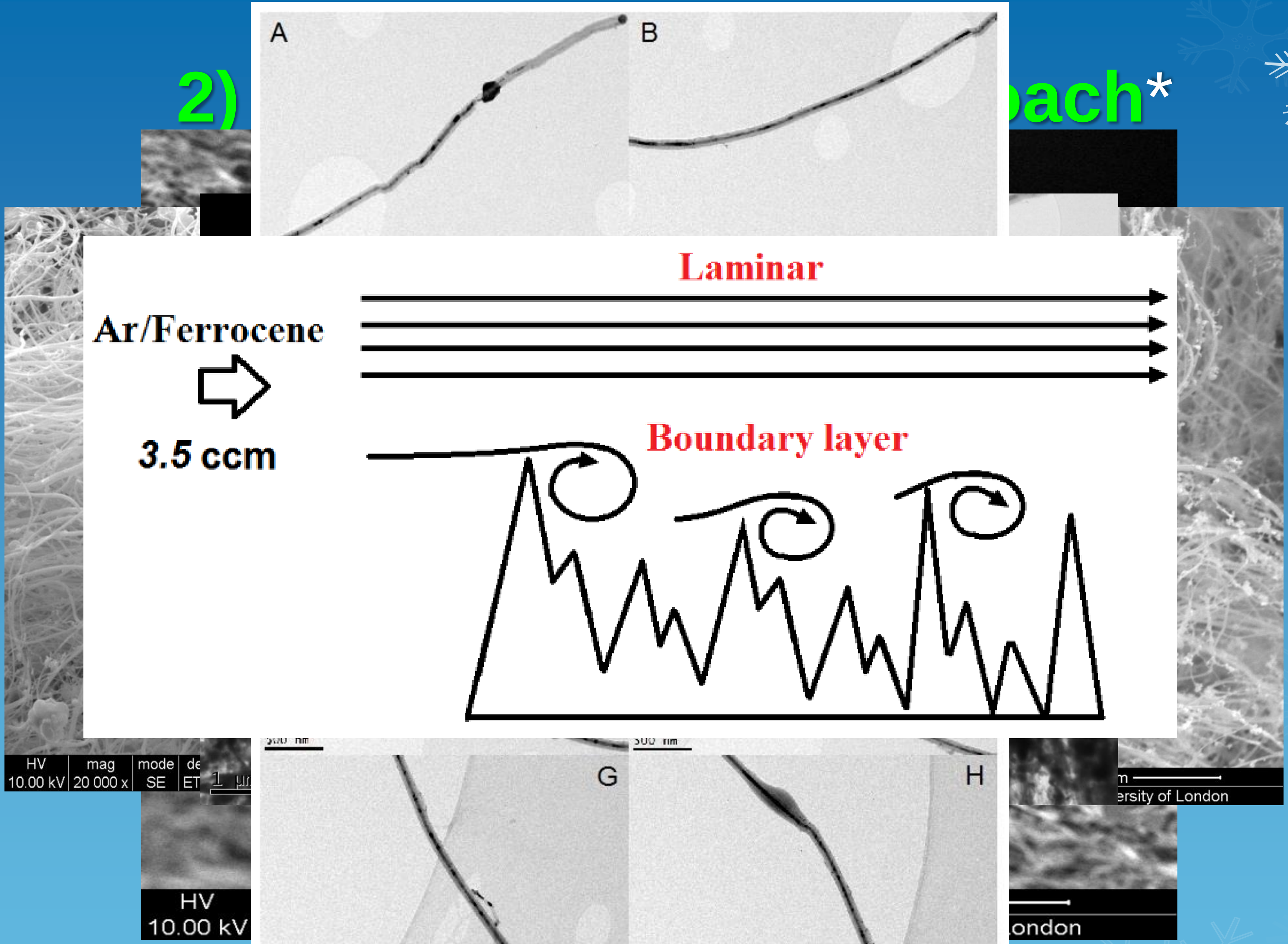
- Involves the perturbation of the ferrocene-Ar vapour flow

- Two approaches:

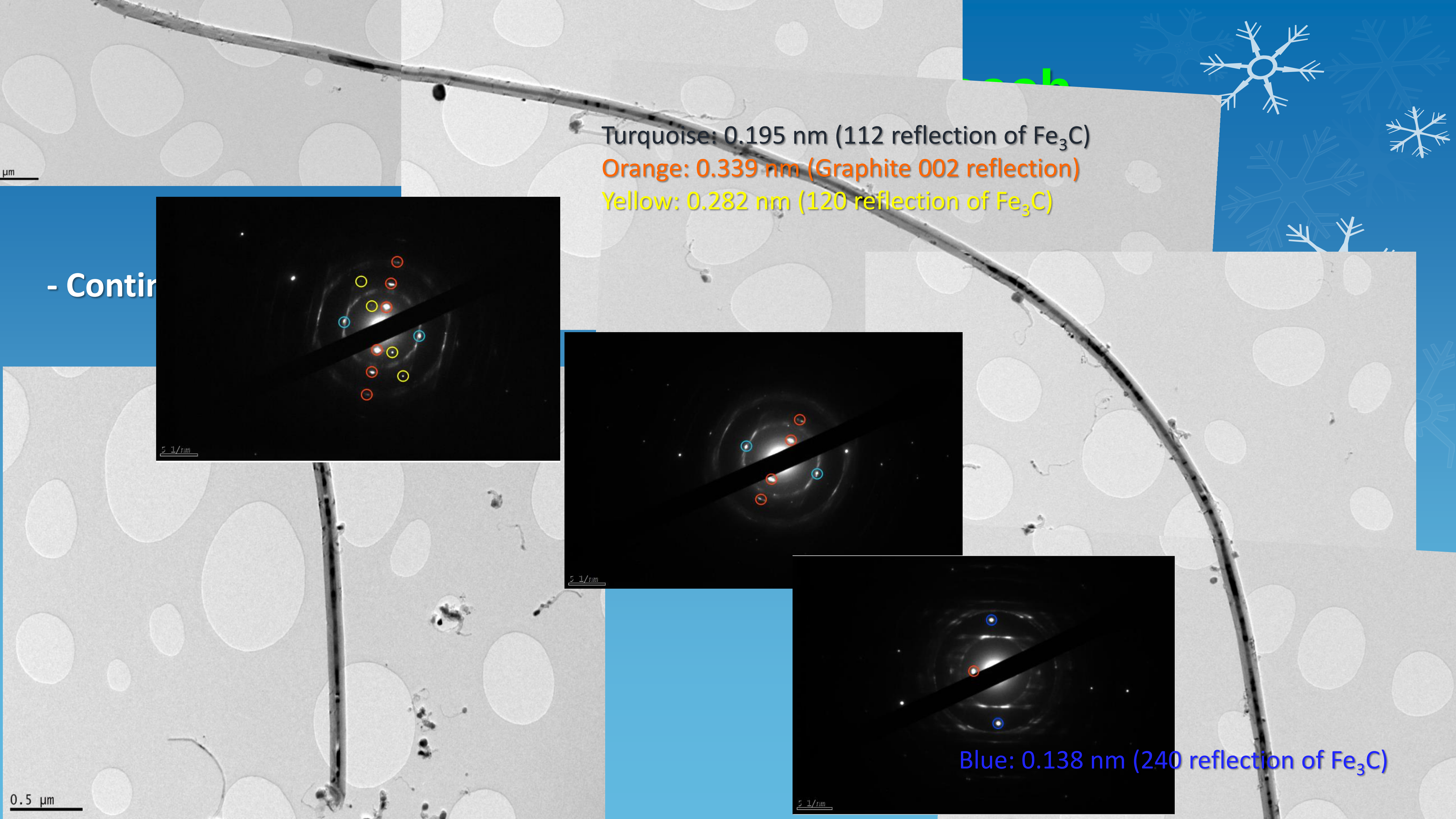
  1) **Substrate-hole approach:**
  2) **Boundary-layer approach:**

- Use identical sublimation and pyrolysis temperatures (180 ºC and 990 ºC).

2) ach*



<u>Boi F. S.</u>, Mountjoy G., Baxendale M*. Boundary layer chemical vapor synthesis of self-organized radial filled-carbon-nanotube structures.Carbon 2013; 64: 516-26.



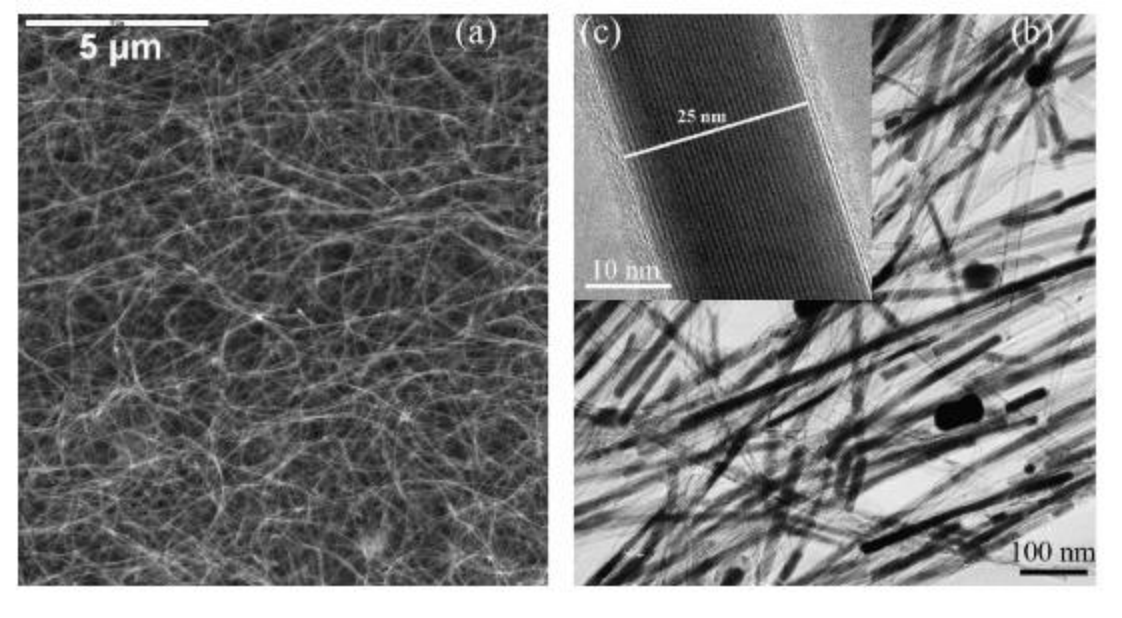
Turquoise: 0.195 nm (112 reflection of Fe_3C)
Orange: 0.339 nm (Graphite 002 reflection)
Yellow: 0.282 nm (120 reflection of Fe_3C)
- Contin
5 1/nm
5 1/nm
5 1/nm
0.5 µm
Blue: 0.138 nm (240 reflection of Fe_3C)

Additional Solutions towards filling control:

Chlorine and Sulfur as CNT- filling promoters

Chlorine as a CNT- filling promoter

In presence of Cl radicals, the formation of CCl_4 species, induce changes in the carbon to metal ratio value, with substantial consequences on the degree of ferromagnetic filling of the CNTs, which self-arrange into an entangled ensemble, known as buckypaper



ELSEVIER
Carbon
Volume 47, Issue 4, April 2009, Pages 1141-1145

In situ synthesis and magnetic anisotropy of ferromagnetic buckypaper

Ruitao Lv [a], Shinji Tsuge [b], Xuchun Gui [c], Kazuyuki Takai [b], Feiyu Kang [a], Toshiaki Enoki [b], Jinquan Wei [c], Jialin Gu [a], Kunlin Wang [c], Dehai Wu [c]

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ELSEVIER
Materials Chemistry and Physics
Volume 113, Issues 2–3, 15 February 2009, Pages 634-637

The decisive roles of chlorine-contained precursor and hydrogen for the filling Fe nanowires into carbon nanotubes

Xuchun Gui [a, b], Kunlin Wang [a, b], Wenxiang Wang [a, b], Jinquan Wei [a, b], Xianfeng Zhang [a, b], Ruitao Lv [c], Yi Jia [a, b], Qinke Shu [a, b], Feiyu Kang [c], Dehai Wu [a, b]

- Variable filling rate at low-Cl concentrations

Guo J., He Y., Wang S. and **Boi F. S.*** Mapping the transition from free-standing vertically-aligned Fe3C-filled carbon nanotube films to entangled randomly-oriented carbon nanotube buckypapers in presence of a great excess of ferrocene. **Carbon 2016; 102: 372-82.**

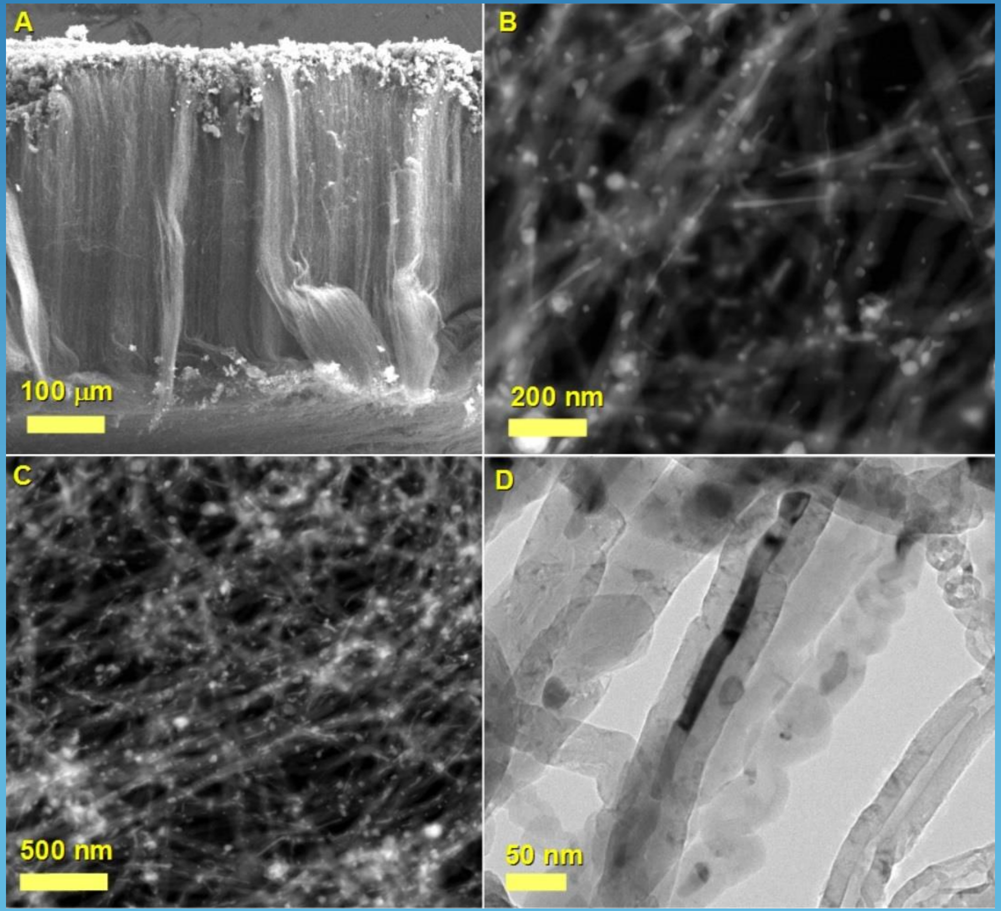
Mater. Res. Express **6** (2019) 015040

Increasing ferrocene quantity

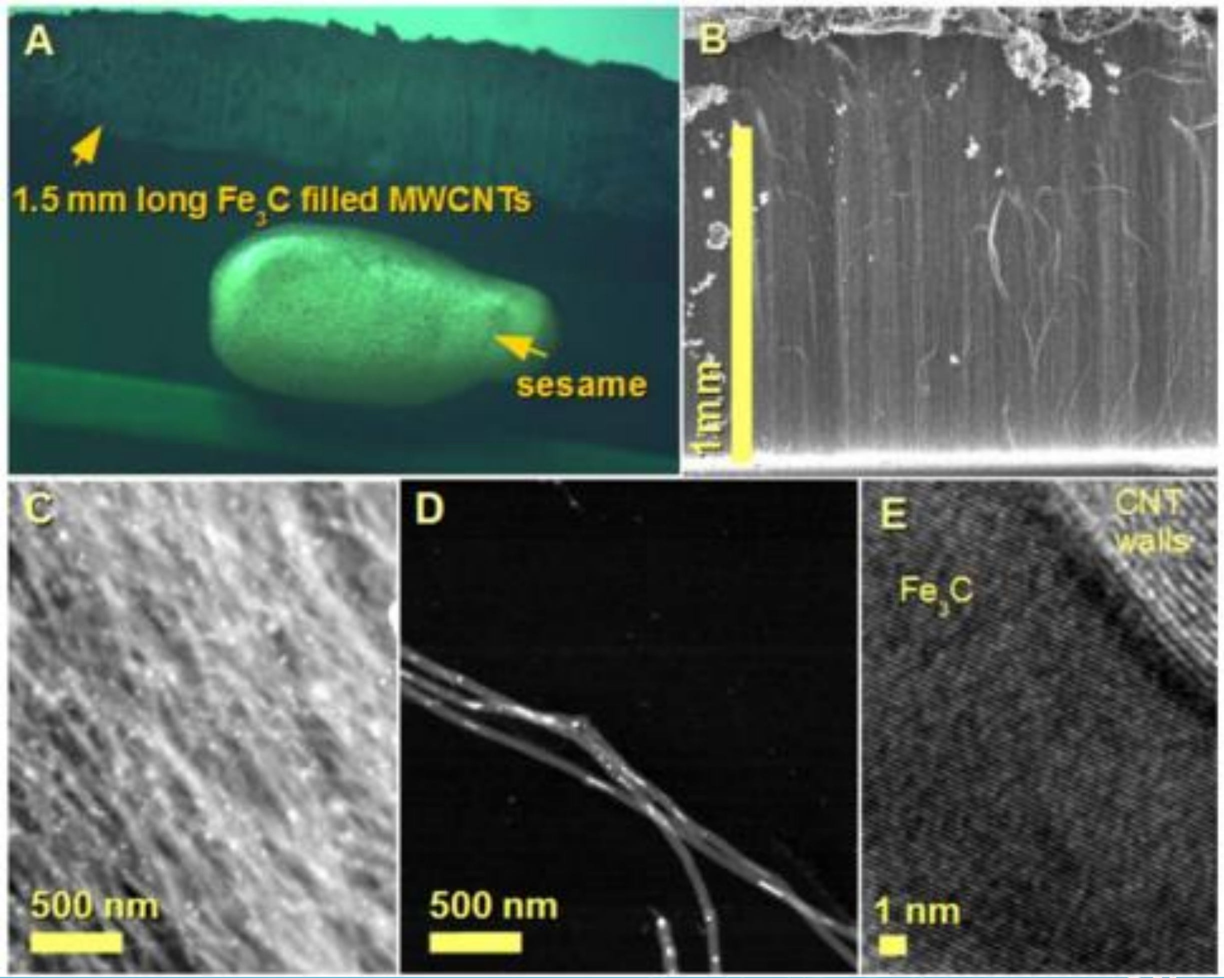
Chlorine-assisted synthesis of Fe_3C-filled mm-long vertically aligned arrays of multiwall carbon nanotubes

Omololu Odunmbaku[1,2], Filippo S Boi[1,2,4], Jian Guo[1], Mu Lan[1], Yi He[3], Tian Yu[1], Shanling Wang[3] and Gang Xiang[1,4]

High evap. T Fixed $C_6H_4Cl_2$ (0. 15 ml)



Free Standing vertically aligned



- Variable filling rate at low-Cl concentrations

Guo J., He Y., Wang S. and **Boi F. S.*** Mapping the transition from free-standing vertically-aligned Fe3C-filled carbon nanotube films to entangled randomly-oriented carbon nanotube buckypapers in presence of a great excess of ferrocene. **Carbon 2016; 102: 372-82.**

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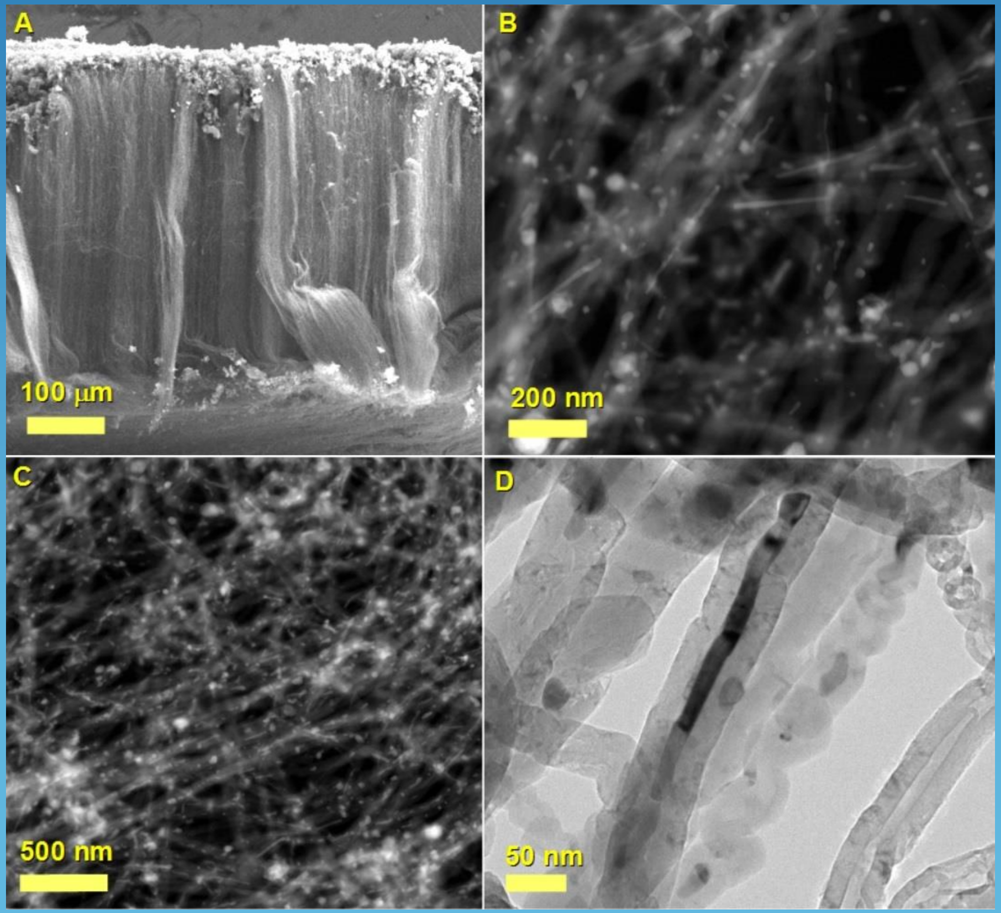
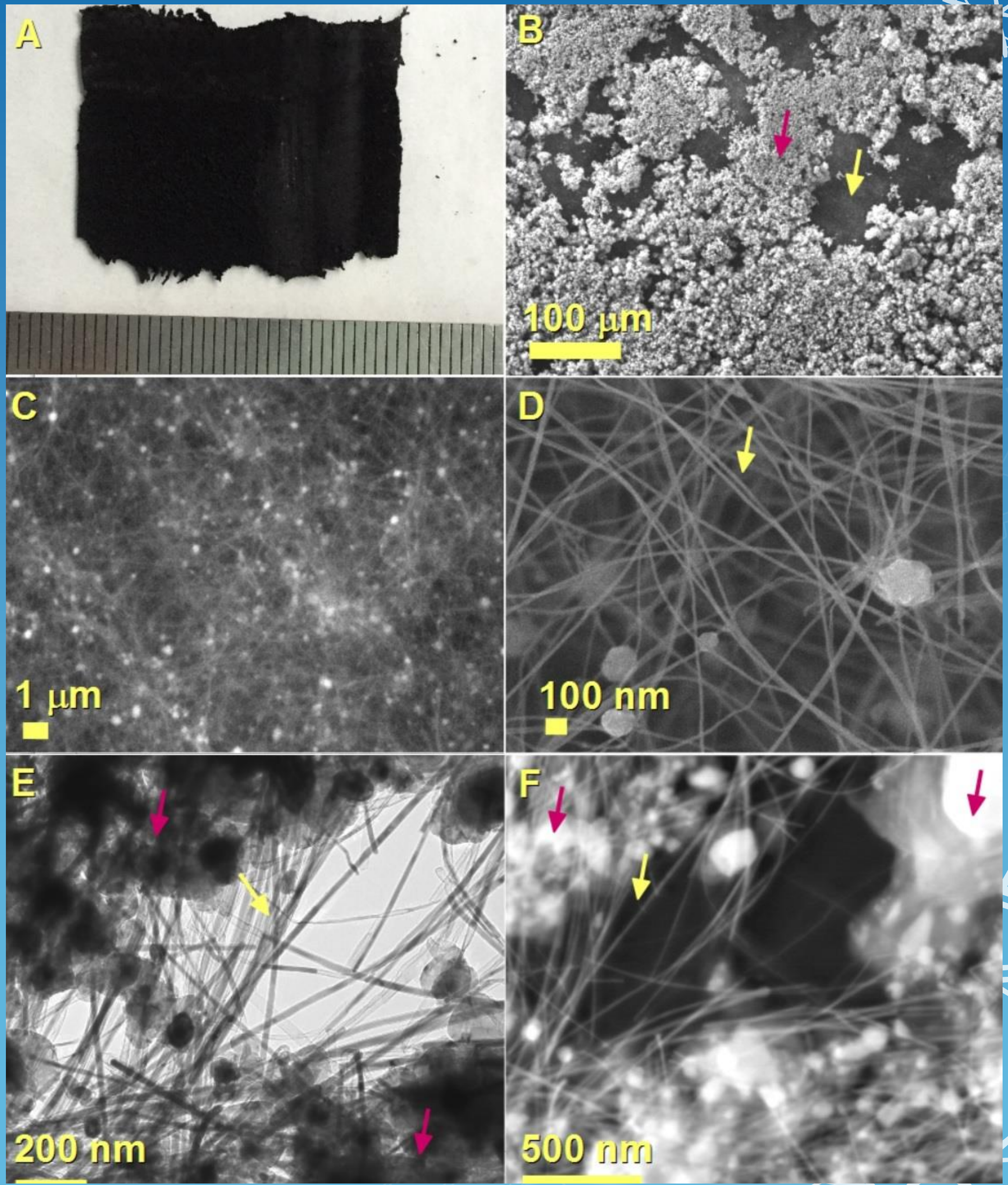


Free Standing vertically aligned

Low evap. T Fixed $C_6H_4Cl_2$ 0.15 ml



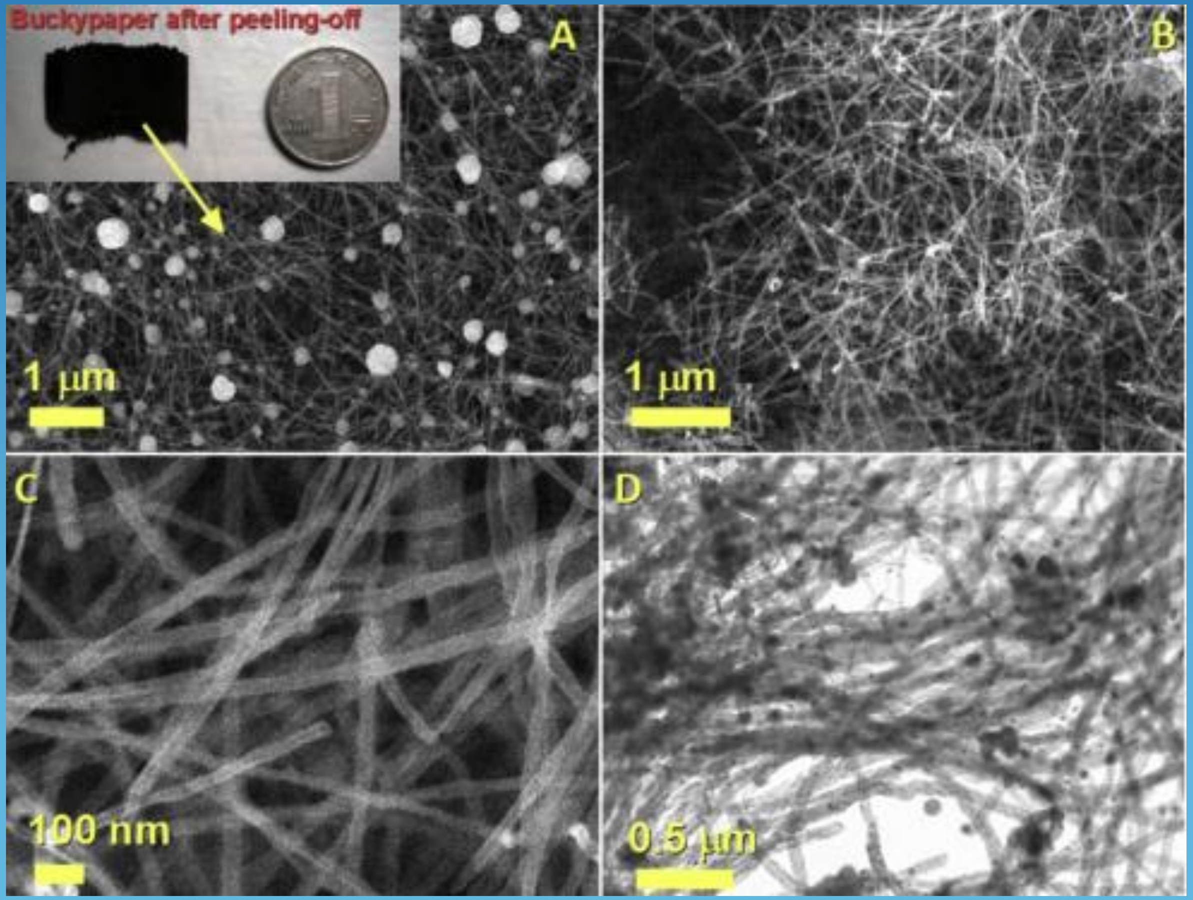
Free Standing horizontally oriented

- Variable filling rate at low-Cl concentrations

Guo J., He Y., Wang S. and **Boi F. S.*** Mapping the transition from free-standing vertically-aligned Fe3C-filled carbon nanotube films to entangled randomly-oriented carbon nanotube buckypapers in presence of a great excess of ferrocene. **Carbon 2016; 102: 372-82.**

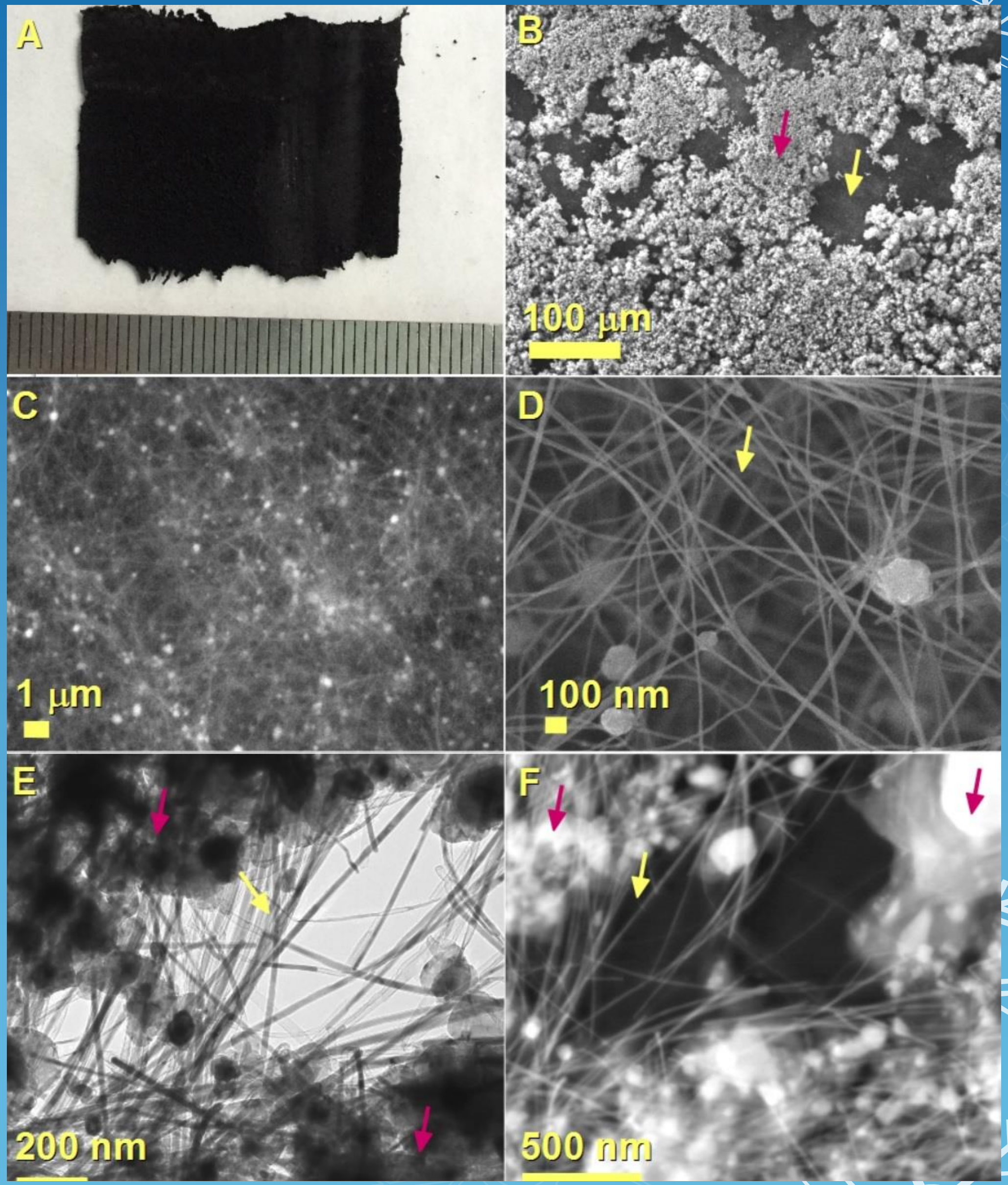
Low evap. T Fixed $C_6H_4Cl_2$ 0.15 ml

15 ml/min



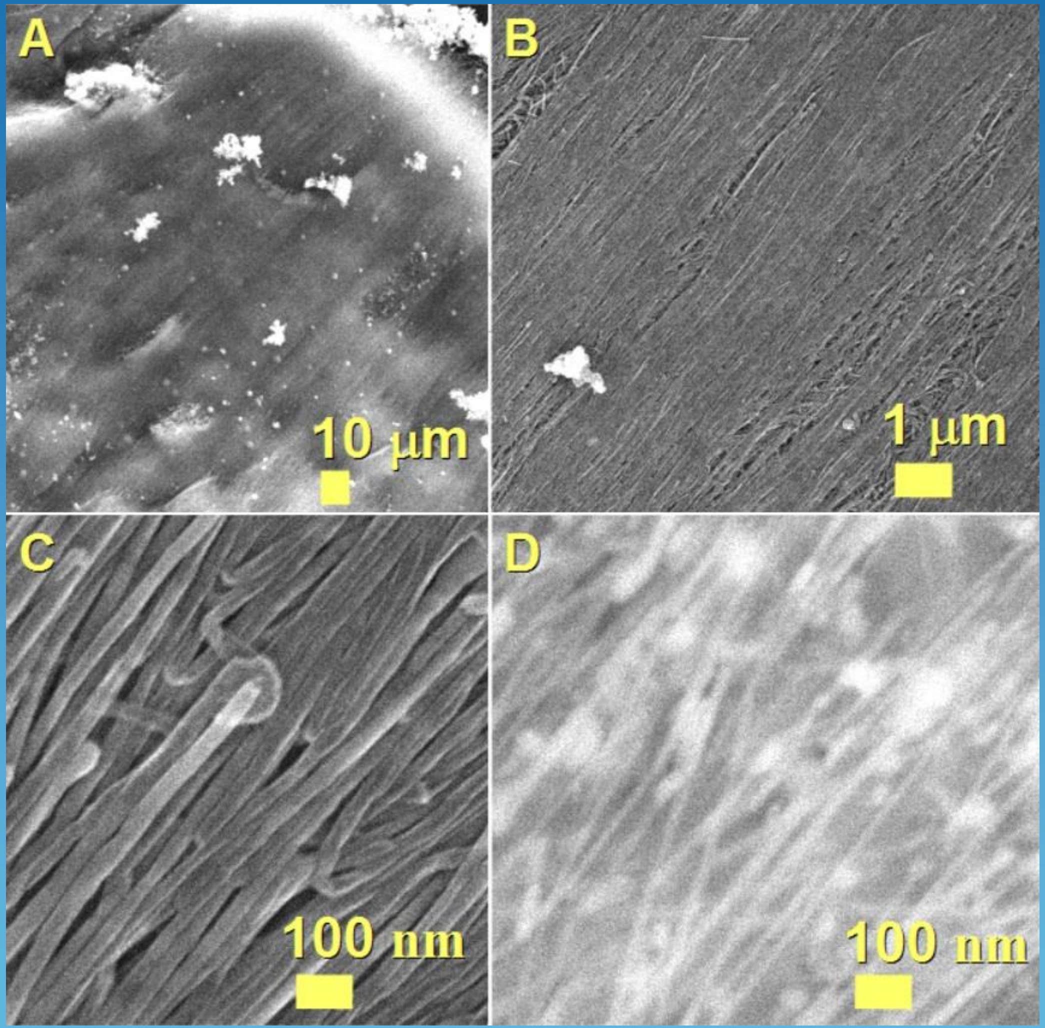
5 ml/min

Free Standing horizontally oriented

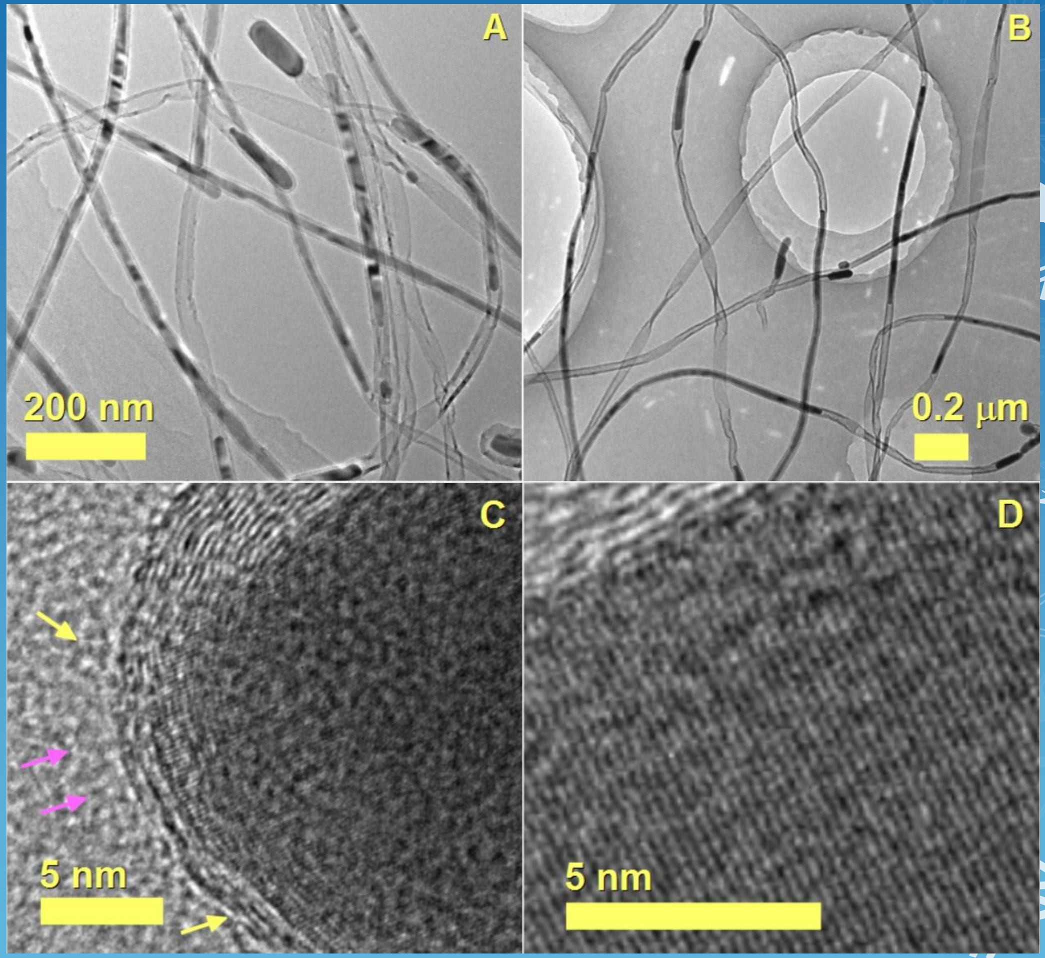


- Increasing vapour flow rates and Cl-concentration

Aligned Fe_3C@CNTs buckypaper Fe_3C@CNTs, resulting from pyrolysis of mixtures of dichlorobenzene (0.65 mL) and ferrocene (1 gram)



100 ml/min



[1] Boi F. S.*, Guo J., Wang S., He Y., Xiang G., Zhang X.* and Baxendale M.* Fabrication of cm scale buckypapers of horizontally aligned multiwall carbon nanotubes highly filled with Fe3C: the key roles of Cl and Ar-flow rate. Chem. Comm. 2016; 52: 4195-98.

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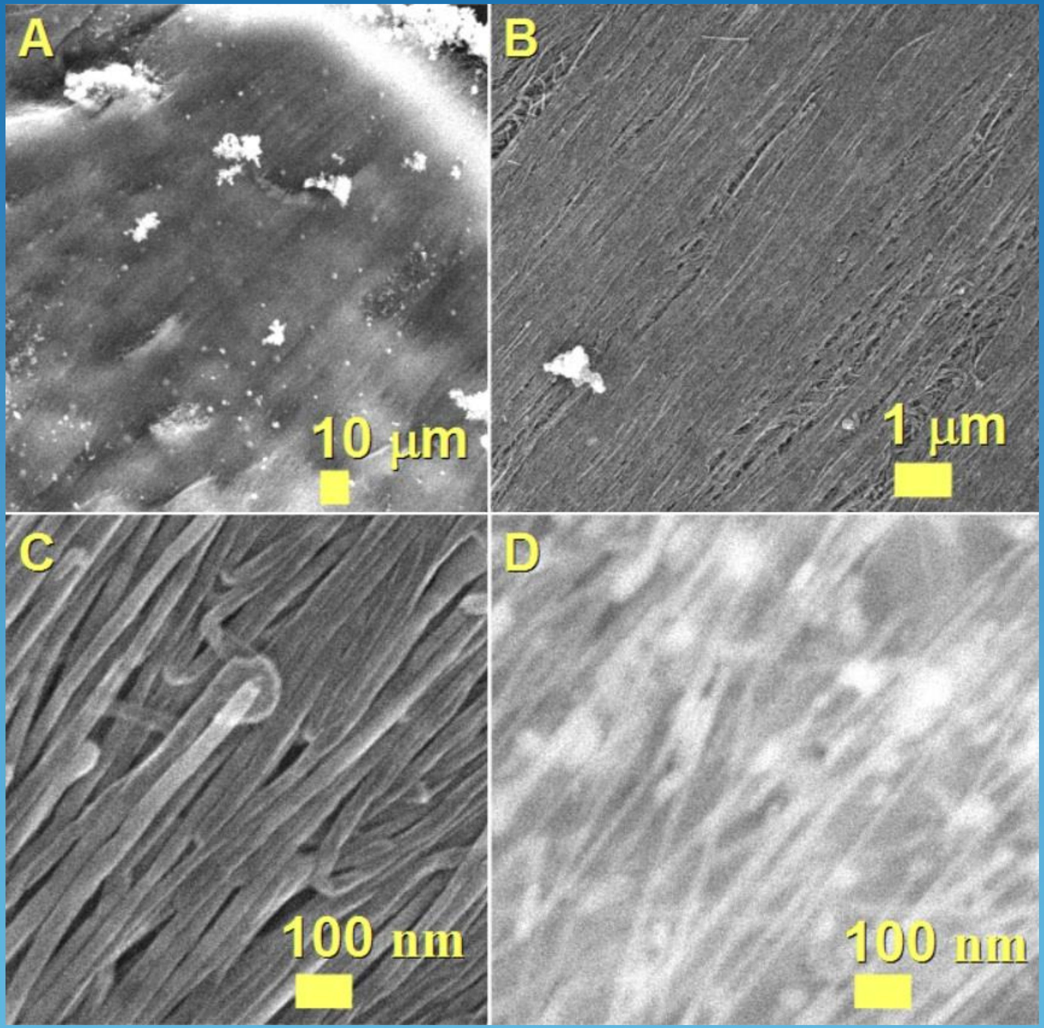
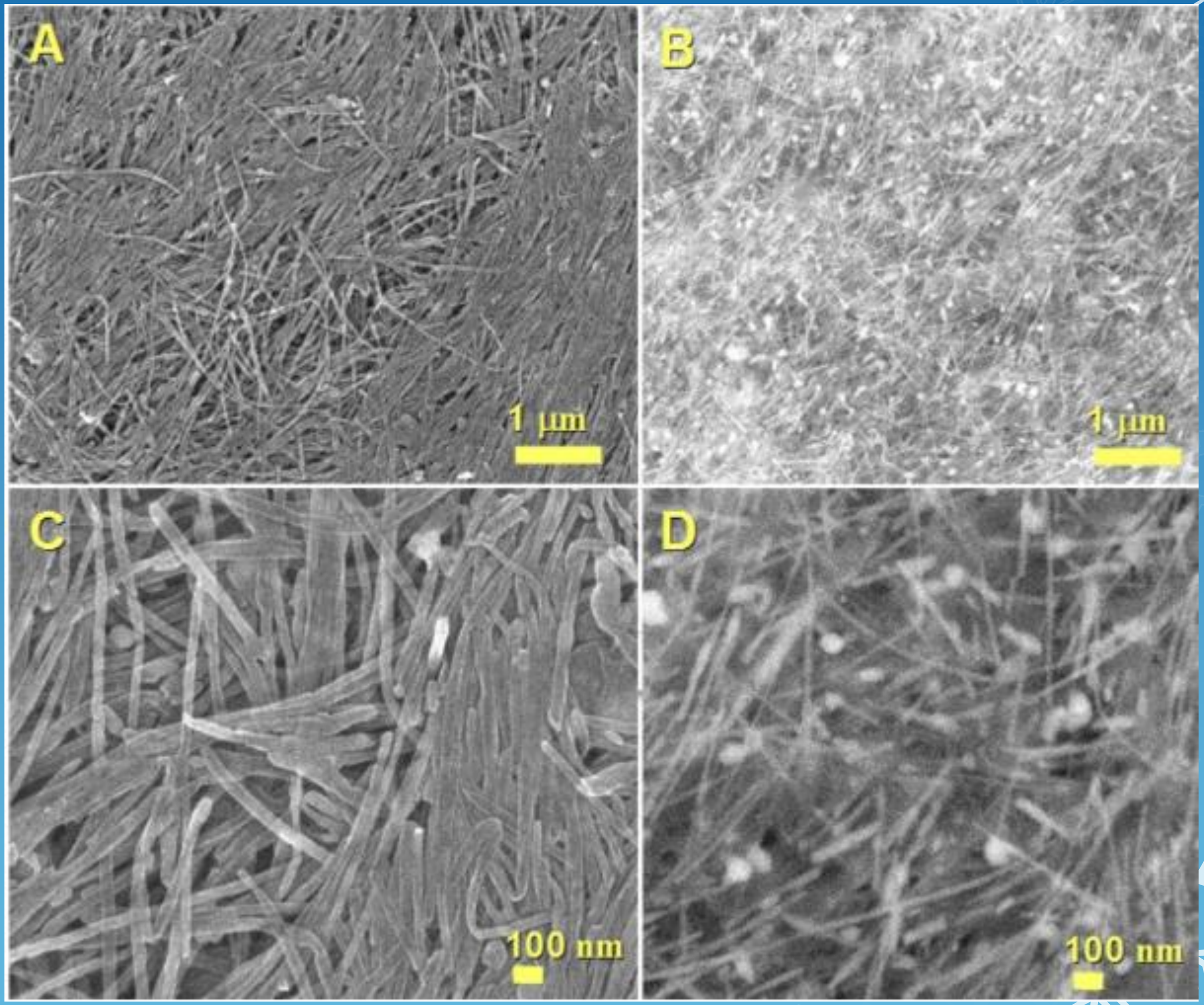
100 ml/min



20 ml/min



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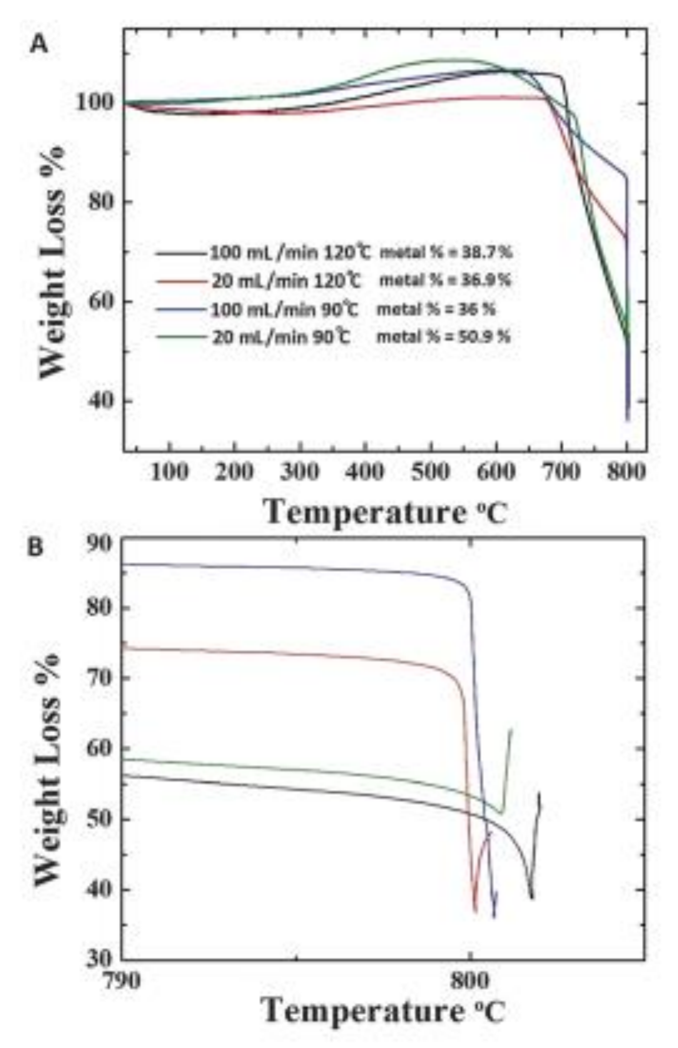

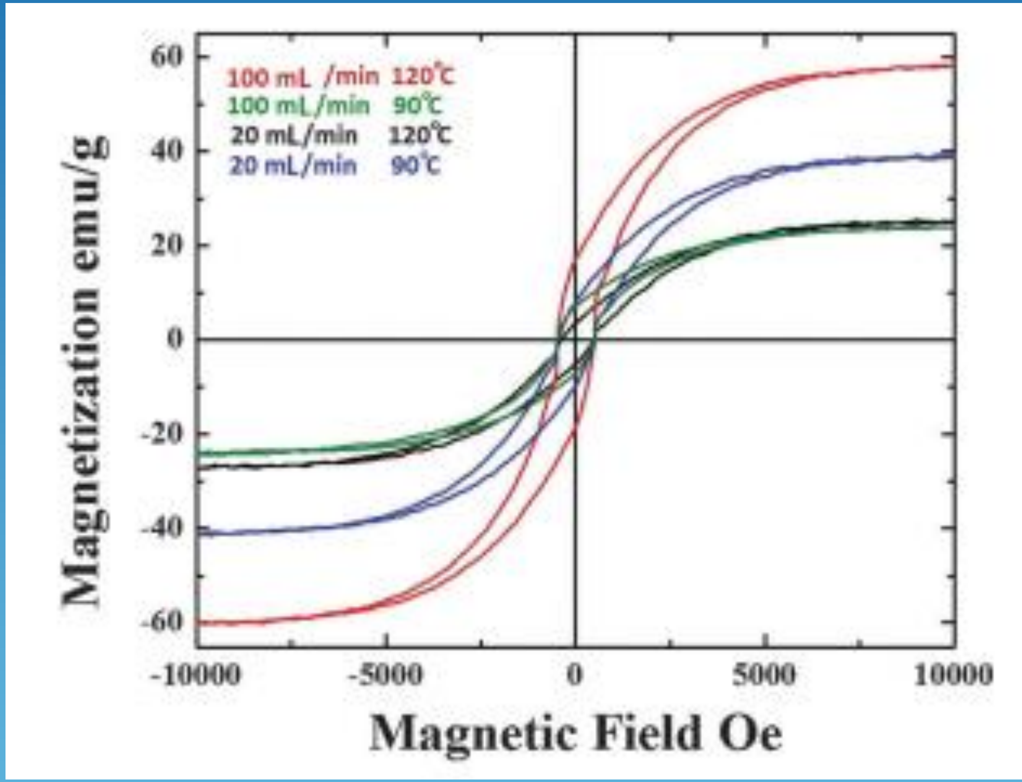

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- Investigating phase transformations

DOI: 10.1039/C7RA03144K (Paper) *RSC Adv.*, 2017, **7**, 25025-25030

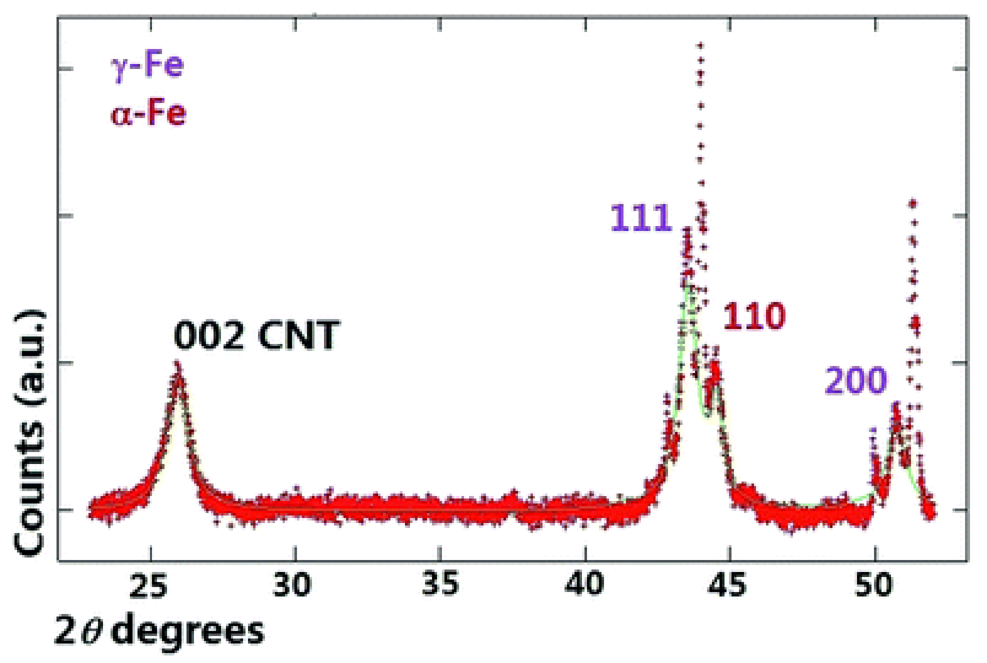
New insights on the dynamics of the γ-Fe/α-Fe phase-transition inside iron-filled carbon nanotubes†

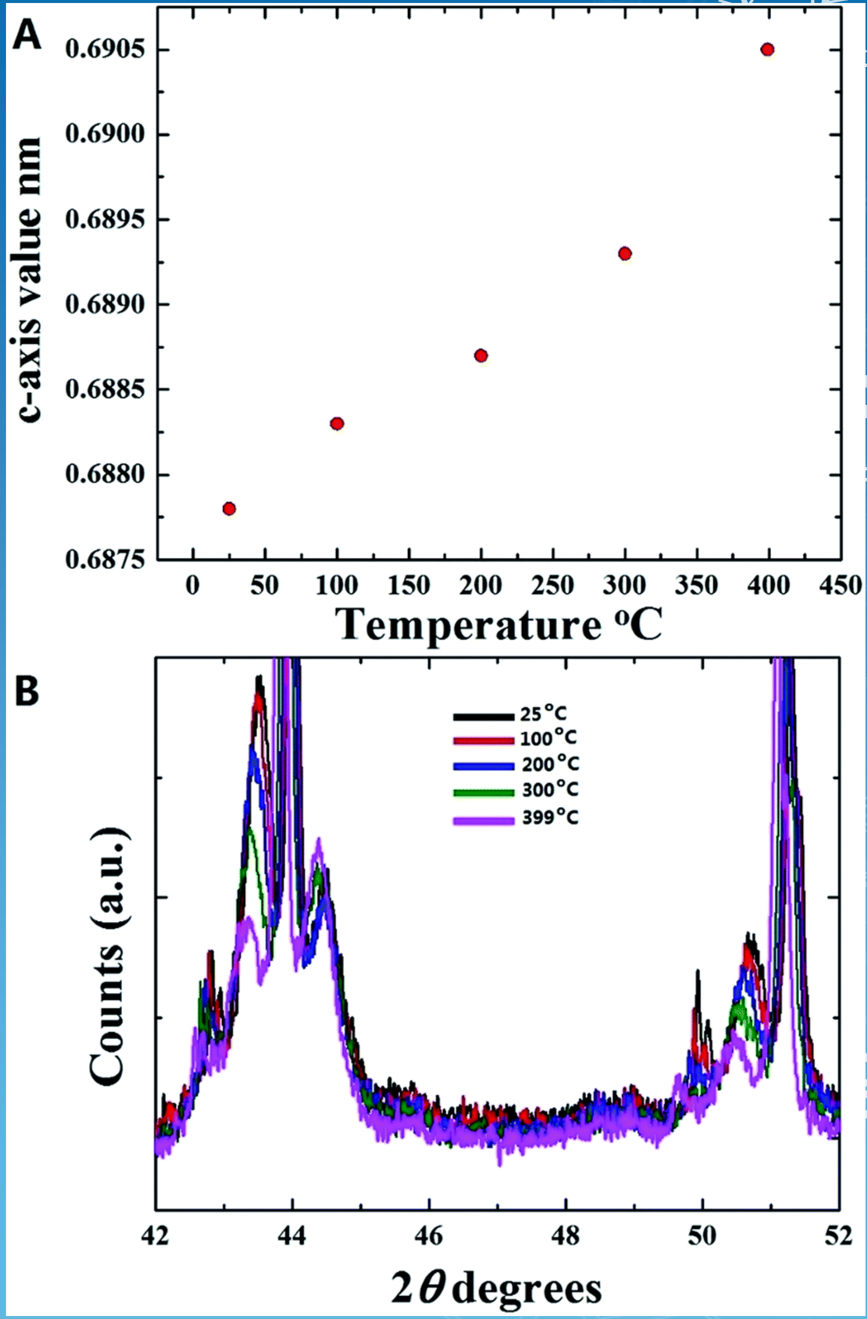
Filippo S. Boi *[a], **Yuzhong Hu**[a] **and Jiqiu Wen**[b]

[a] *College of Physical Science and Technology, Sichuan University, Chengdu, China. E-mail: f.boi@scu.edu.cn*

[b] *Analytical and Testing Center, Sichuan University, Chengdu, China*

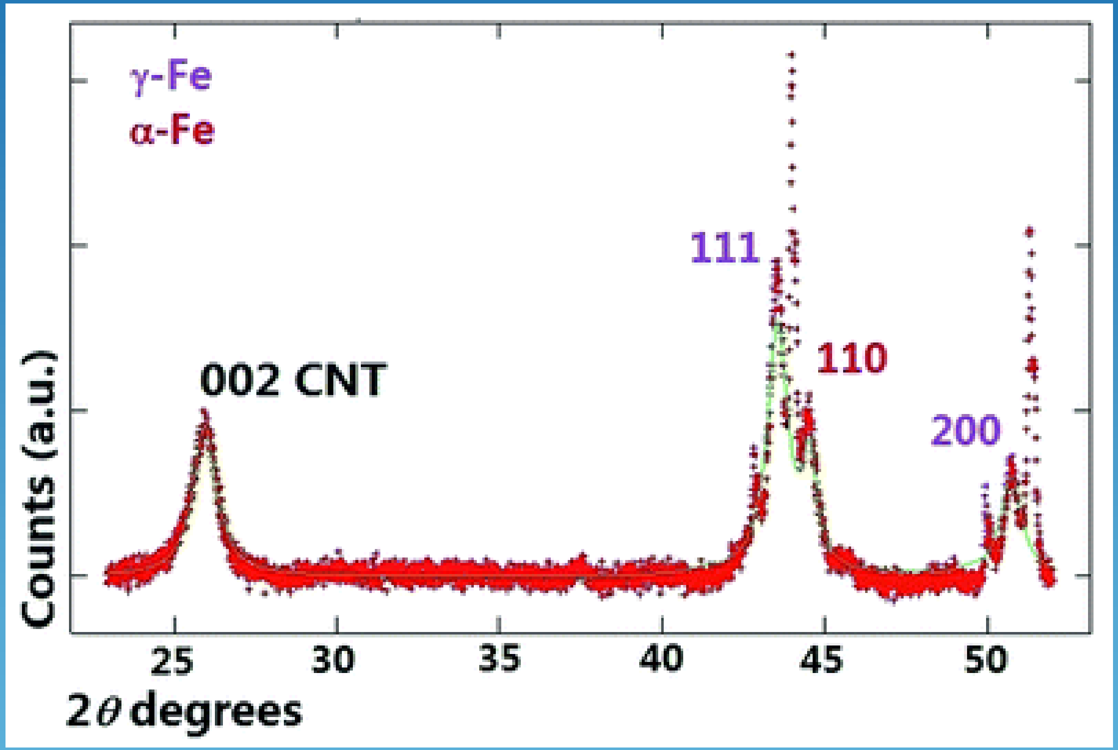
Received 16th March 2017 , Accepted 3rd May 2017

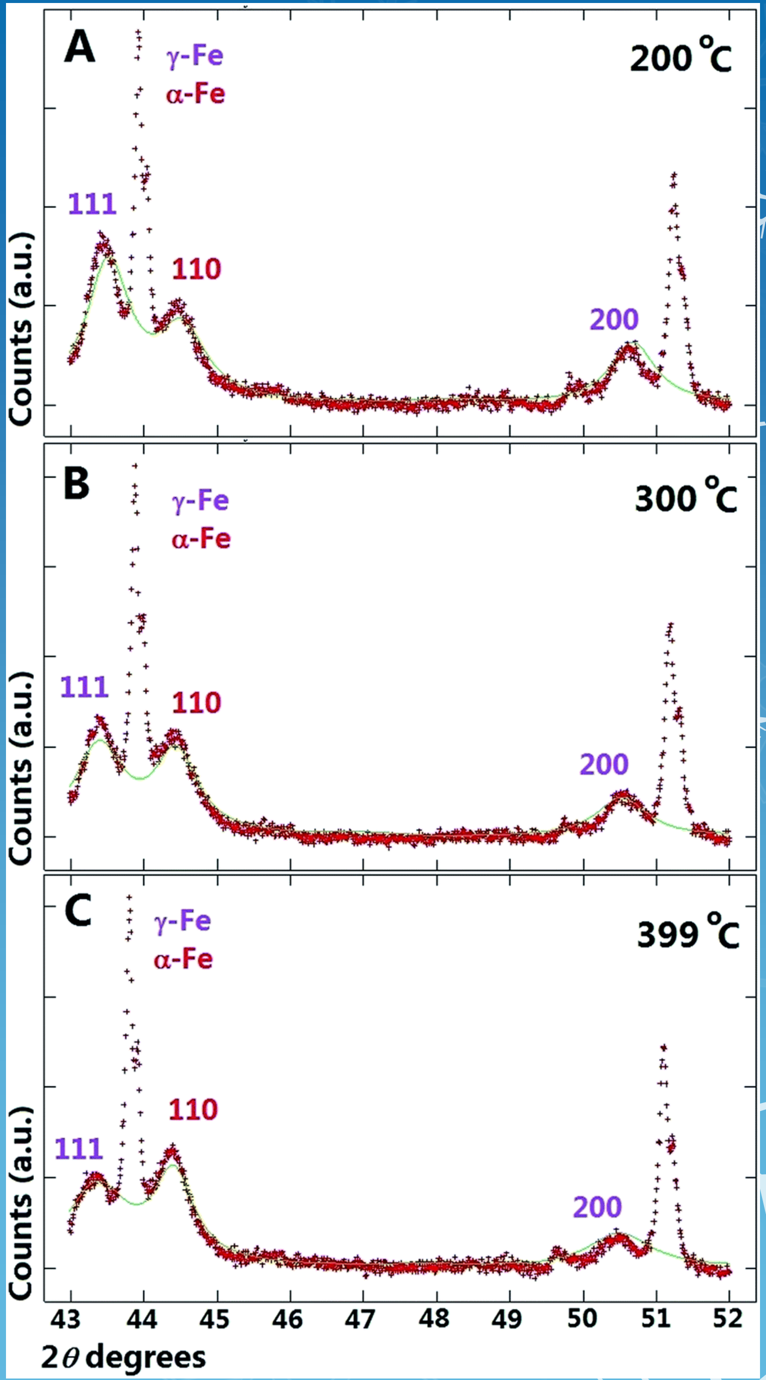




- Investigating phase transformations

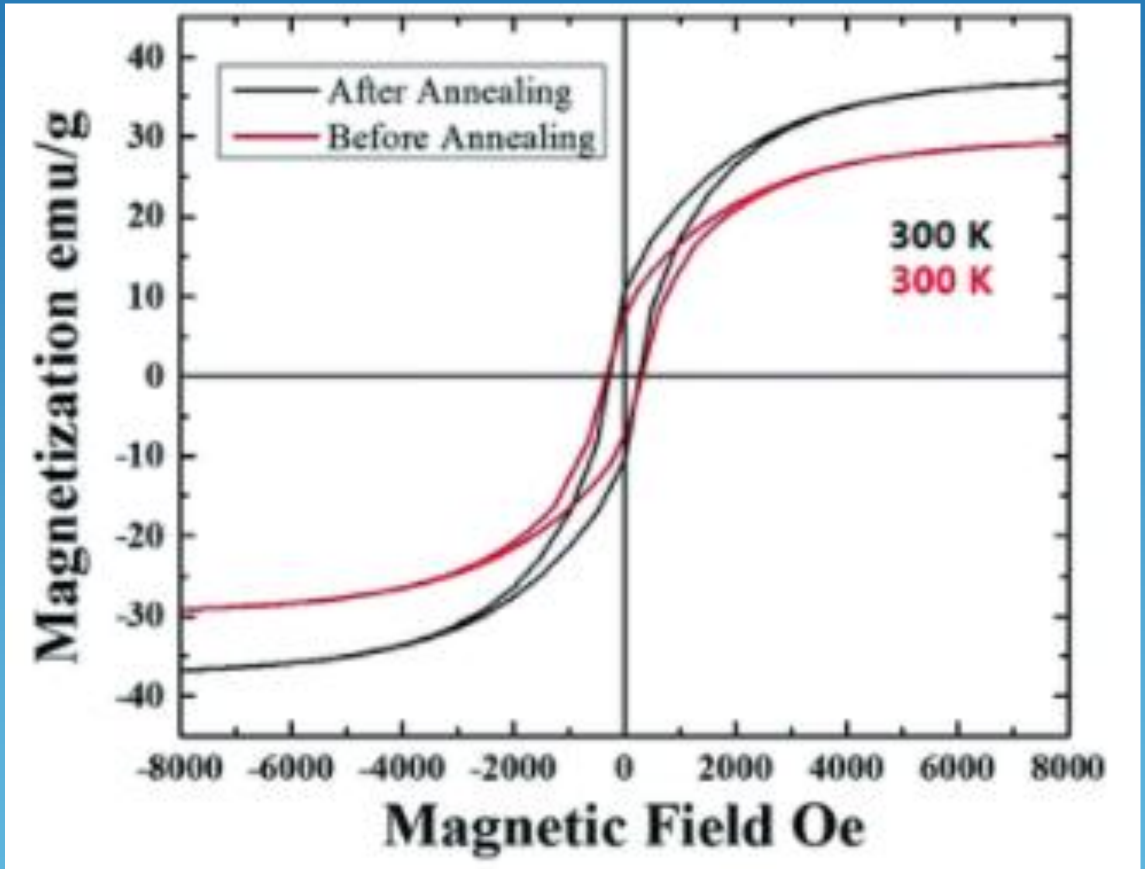
Ensemble of alpha and gamma Fe phases





- Investigating phase transformations

Enhanced magnetization in post annealed samples



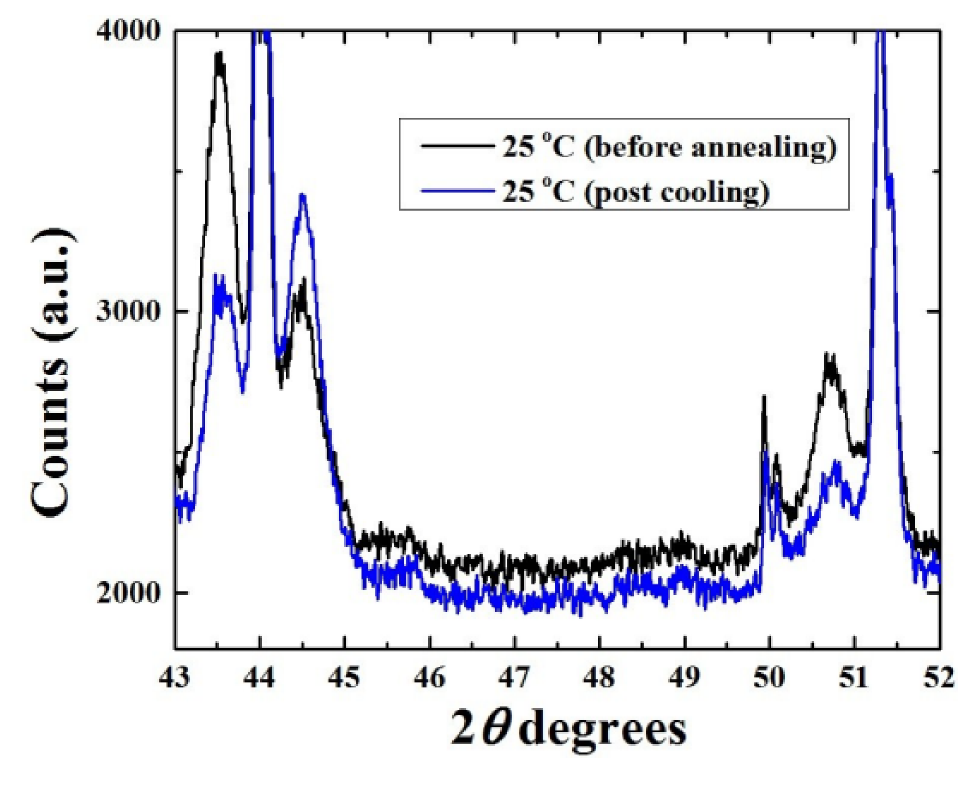


Figure 3 Supp.: Plot showing the variation in the γ-Fe and α-Fe intensities before the annealing treatment in vacuum and after annealing (after cooling down the sample with liquid nitrogen).

- Observation in agreement with previous work by Leonhardt et al.

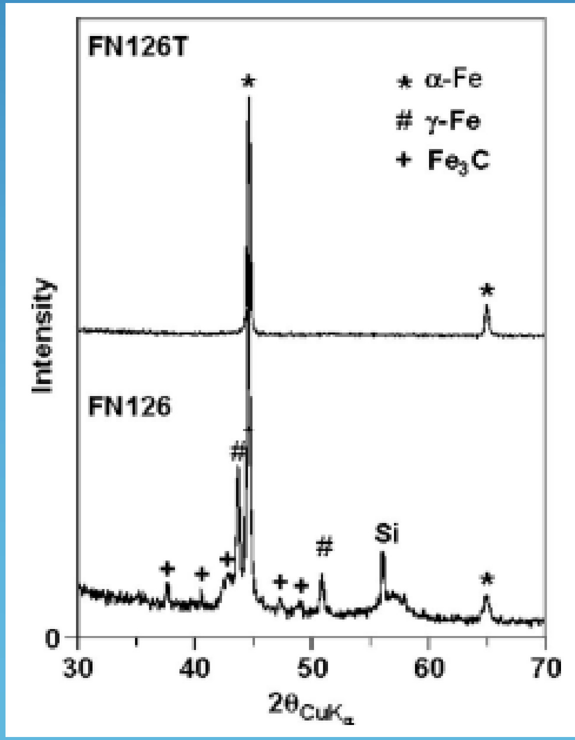
Home > Journal of Applied Physics > Volume 98, Issue 7 > 10.1063/1.2058181

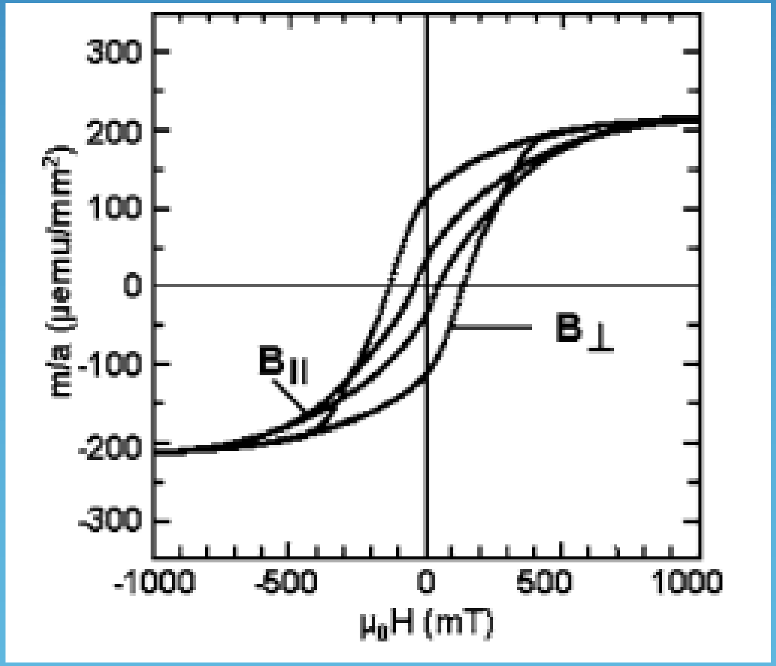
Full . Submitted: 18 April 2005 . Accepted: 10 August 2005 . Published Online: 14 October 2005

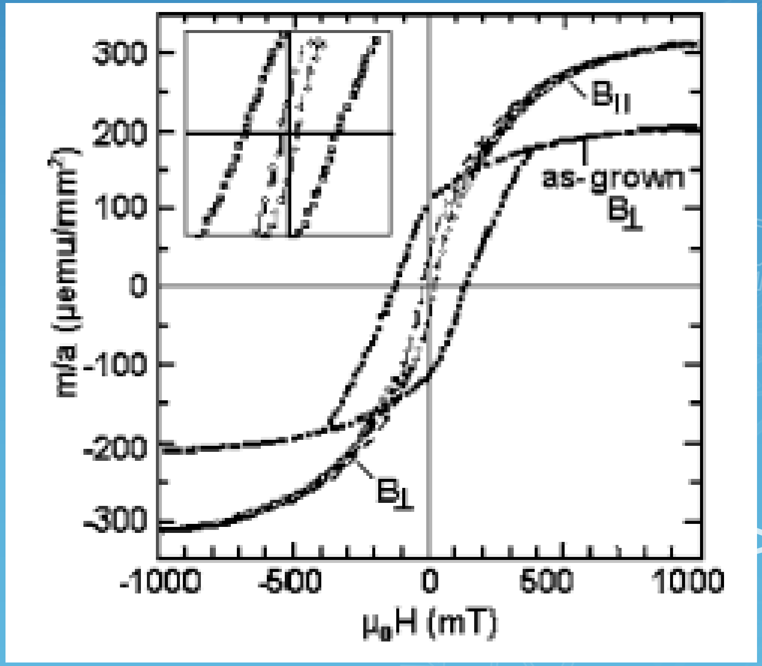
Enhanced magnetism in Fe-filled carbon nanotubes produced by pyrolysis of ferrocene

Journal of Applied Physics **98**, 074315 (2005); https://doi.org/10.1063/1.2058181

A. Leonhardt[a], M. Ritschel, D. Elefant, N. Mattern, K. Biedermann, S. Hampel, Ch. Müller, T. Gemming, *and* B. Büchner



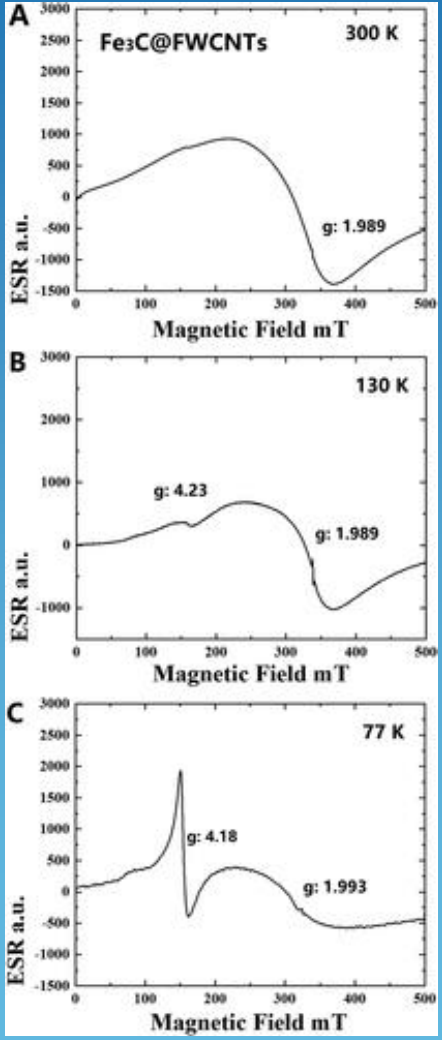




Temperature driven magnetic transitions in $FePd_3$ filled monolayer carbon foam and Fe_3C/α-Fe filled carbon nanotubes

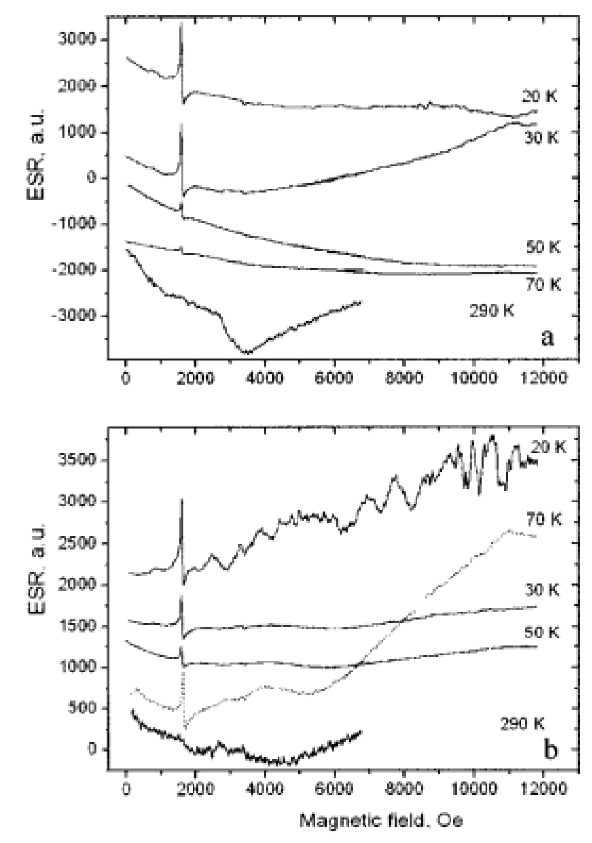
Cite as: J. Appl. Phys. 125, 024302 (2019); doi: 10.1063/1.5064705
Submitted: 7 October 2018 · Accepted: 21 December 2018 ·
Published Online: 9 January 2019

Omololu Odunmbaku,[1] JiaChen Xia,[1] Joanna Borowiec,[1] Shanling Wang,[2] Ayoub Taallah,[1] Yi He,[2] and Filippo S. Boi[1,3,a)]



- Unusual Low Temperature alpha to gamma Transition

Agrees with Sohatsky et al.

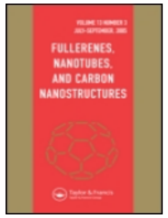


Original Articles

ESR of Fe-Filled Multi-Walled Carbon Nanotubes

V. Sohatsky, S. Kolesnik, D. Makarov, A. Leonhardt, T. Muehl, I. Moench, ...show all

Pages 401-410 | Published online: 06 Feb 2007
Download citation https://doi.org/10.1081/FST-200039389



Taylor & Francis

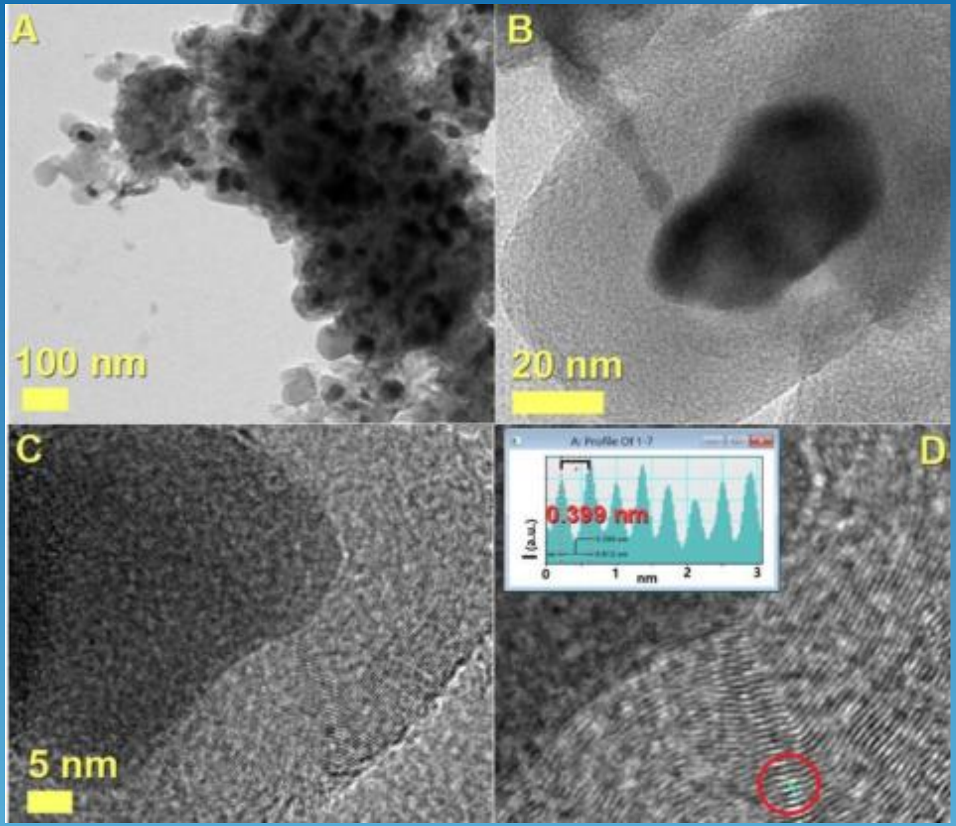
Fullerenes, Nanotubes, and Carbon Nanostructures

ISSN: 1536-383X (Print) 1536-4046 (Online) Journal homepage: http://www.tandfonline.com/loi/lfnn20

ESR of Fe-Filled Multi-Walled Carbon Nanotubes

V. Sohatsky , S. Kolesnik , D. Makarov , A. Leonhardt , T. Muehl , I. Moench , M. Ritschel , R. K. Kozhuharova , J. Schumann & C. M. Schneider

We observe this transition also in free standing iron filled CNOs

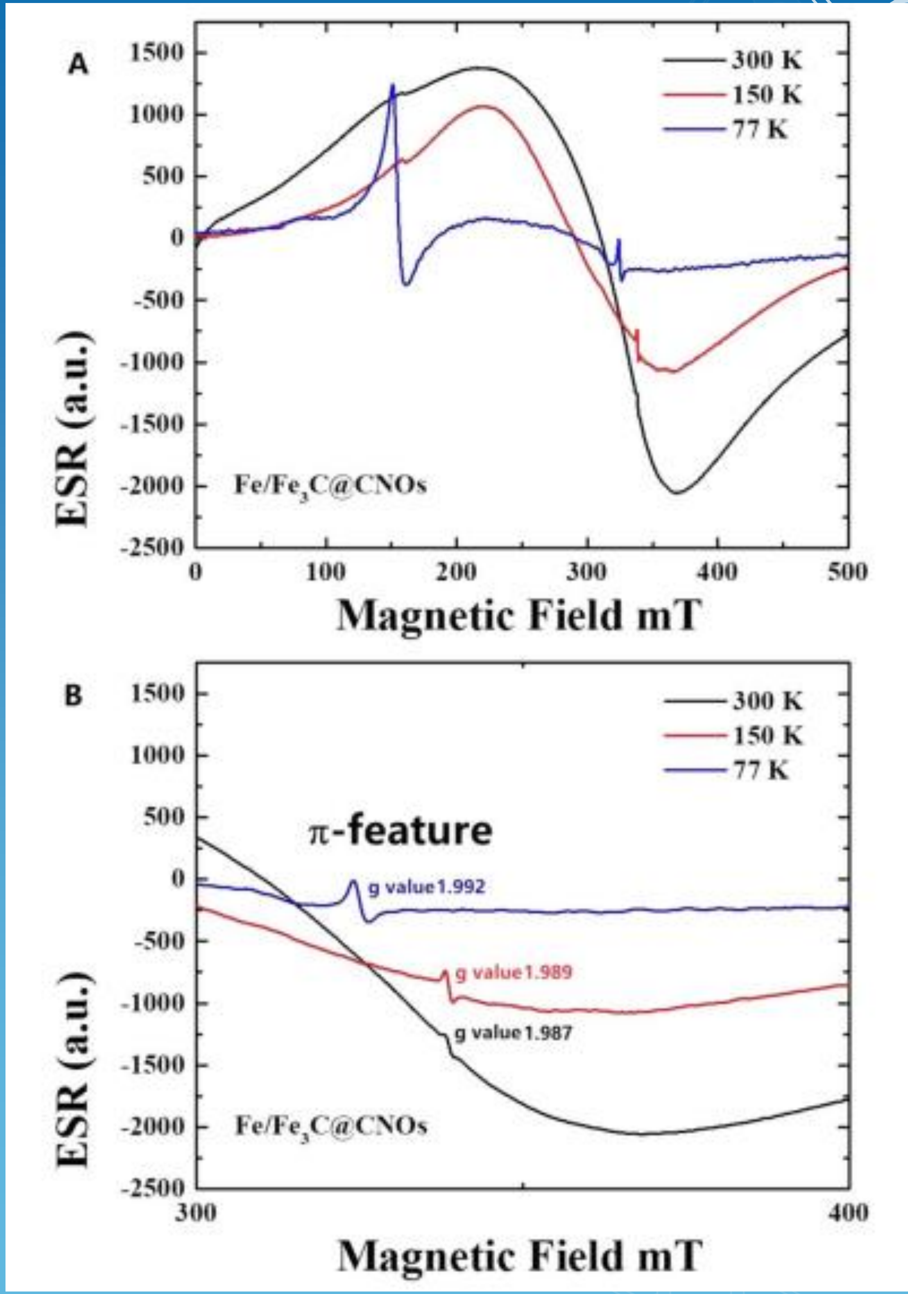


PAPER • OPEN ACCESS

Temperature-dependent c-axis lattice-spacing reduction and novel structural recrystallization in carbon nano-onions filled with Fe_3C/α-Fe nanocrystals

Filippo S Boi[4] [4,1], Jiaxiang Li[1], Omololu Odunmbaku[1], Mengjiao Liu[2], Daniel Medranda[1], Ayoub Taallah[1], Li Lei[3] and Shanling Wang[4] [4,4]

Published 30 July 2020 • © 2020 The Author(s). Published by IOP Publishing Ltd
Nano Express, Volume 1, Number 2



Sulfur as an additional filling promoter in arc discharge methods

Demoncy et al. (**in arc discharge methods**) highlighted a key role of sulfur in promoting the filling rates of numerous types of metals such as chromium, nickel and germanium within the CNTs core. In these processes sulfur was shown to promote the carbon precipitation before being trapped within the metallic catalyst material.

Eur. Phys. J. B 4, 147–157 (1998)

THE EUROPEAN PHYSICAL JOURNAL B
EDP Sciences

Filling carbon nanotubes with metals by the arc-discharge method: the key role of sulfur

N. Demoncy[1,2], O. Stéphan[3], N. Brun[3], C. Colliex[3,4], A. Loiseau[2,a], and H. Pascard[1]

[1] Laboratoire des Solides Irradiés, CEA-CNRS, École Polytechnique, 91128 Palaiseau Cedex, France
[2] Laboratoire d'Étude des Microstructures, ONERA-CNRS, Office National d'Études et de Recherches Aérospatiales, BP 72, 92322 Châtillon Cedex, France
[3] Laboratoire de Physique des Solides[b], Université de Paris-Sud, Bâtiment 510, 91405 Orsay Cedex, France
[4] Laboratoire Aimé Cotton[c], Campus d'Orsay, Bâtiment 505, 91405 Orsay Cedex, France

Received: 20 January 1998 / Received in final form and accepted: 9 April 1998

Abstract. Various filled carbon nanotubes have recently been successfully produced by the arc-discharge method by doping a 99.4% graphite anode with a transition metal like Cr, Ni, a rare earth like Yb, Dy, or a covalent element like S, Ge. In this work, the structural characteristics of these encapsulated nanowires were studied by High Resolution Transmission Electron Microscopy and their chemical composition was investigated using Electron Energy-Loss Spectroscopy with high spatial resolution: this analysis mode provides elemental concentration profiles across or along the filled nanotubes. Except in the case of Ge for which only pure Ge fillings were identified, surprising amounts of sulfur, which was present as an impurity (≈ 0.25%) in the graphite rods, were found within numerous filling materials. When using high purity carbon rods, no filled nanotube was obtained. We chose the case of Cr to clearly evidence that the addition of sulfur in catalytic quantity is responsible for the formation of filled nanotubes, including sulfur free encapsulated nanowires. A growth mechanism based on a catalytic process involving three elements, *i.e.* carbon, a metal and sulfur, and taking into account the experimental results is proposed.

PACS. 61.16.Bg Transmission, reflection and scanning electron microscopy (including EBIC) – 81.05.Tp Fullerenes and related materials; diamonds, graphite

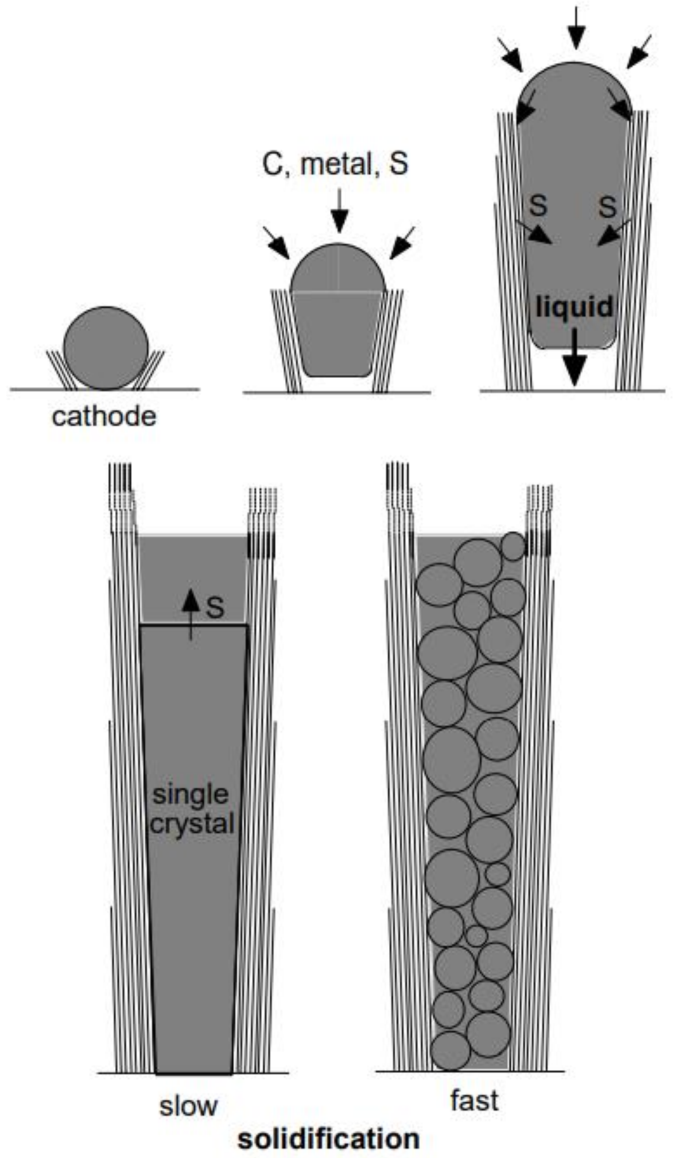
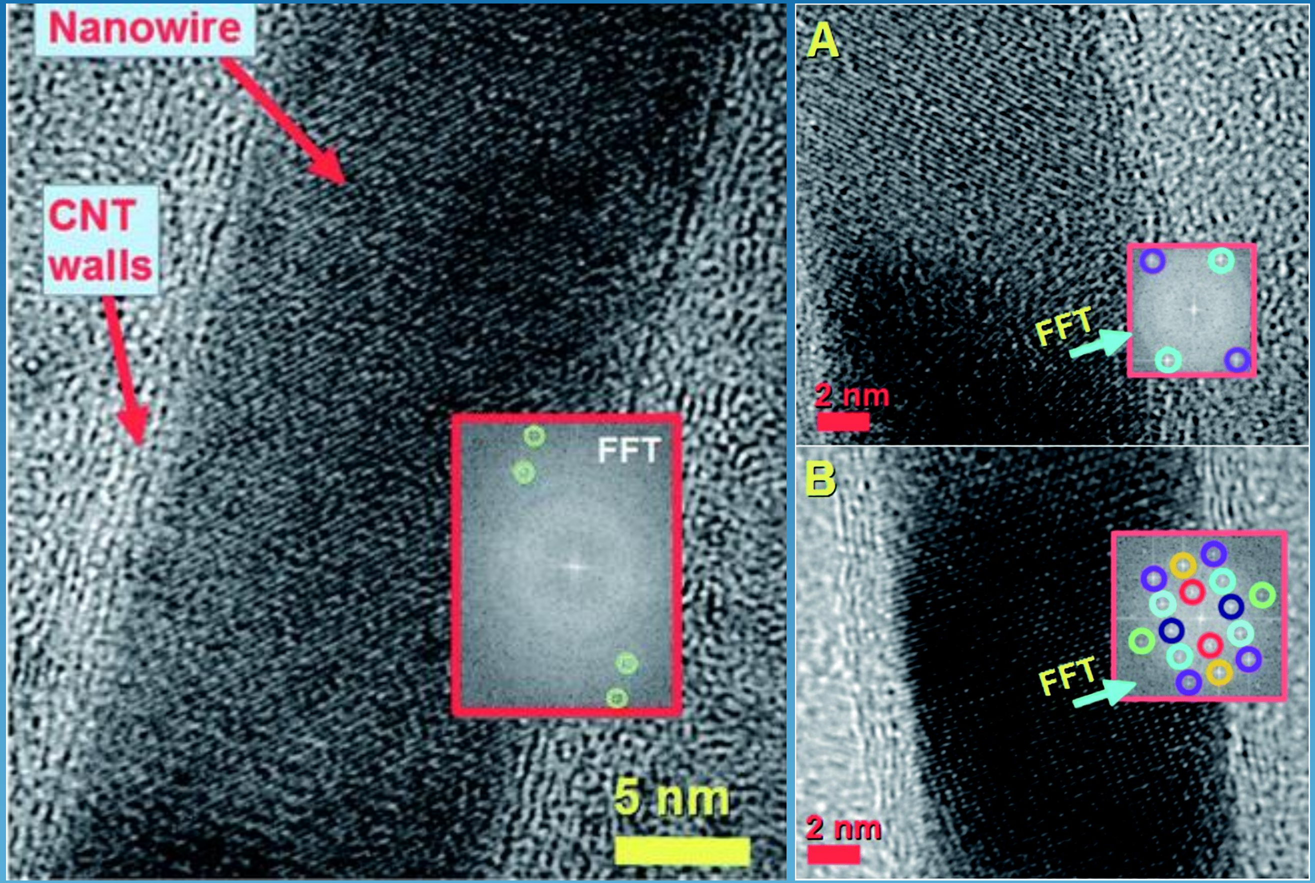
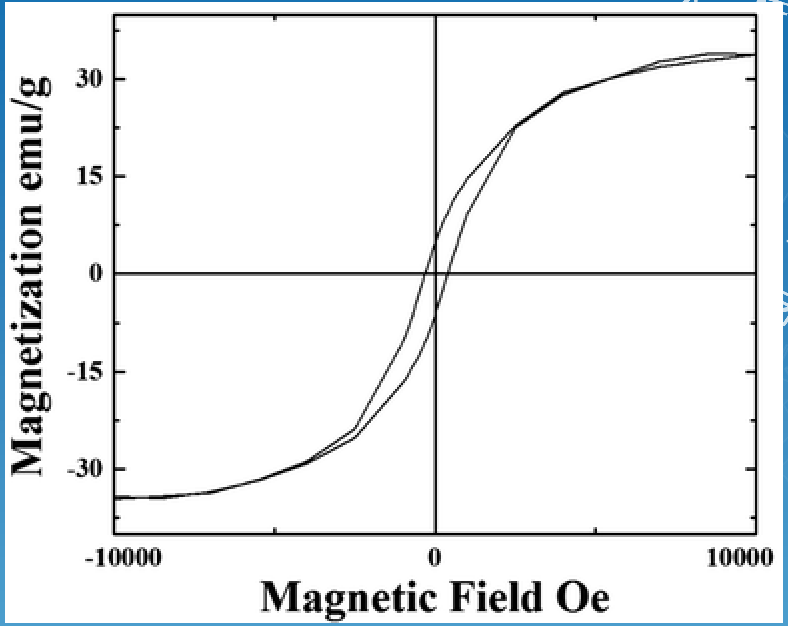
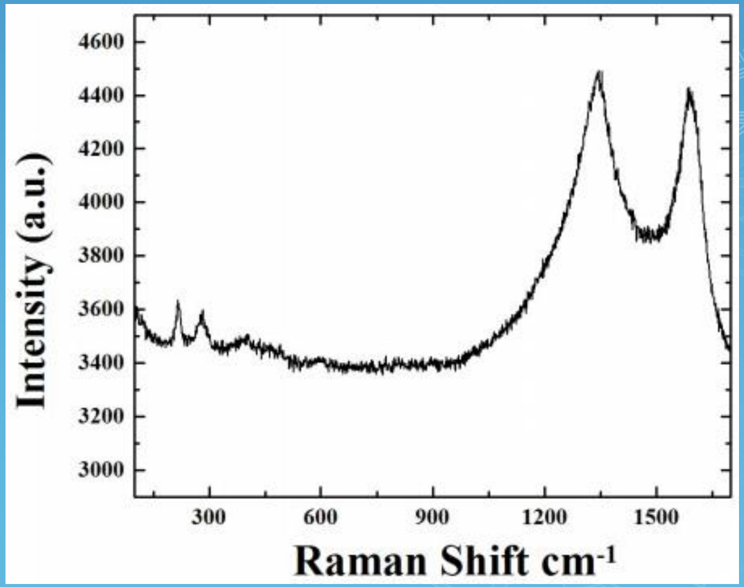


Fig. 15. Schematic growth mechanism proposed for the formation of a carbon nanotube filled with a metal on the cathode of an arc-discharge experiment in presence of sulfur (see text for details).

Sulfur as an additional filling promoter in CVD methods?





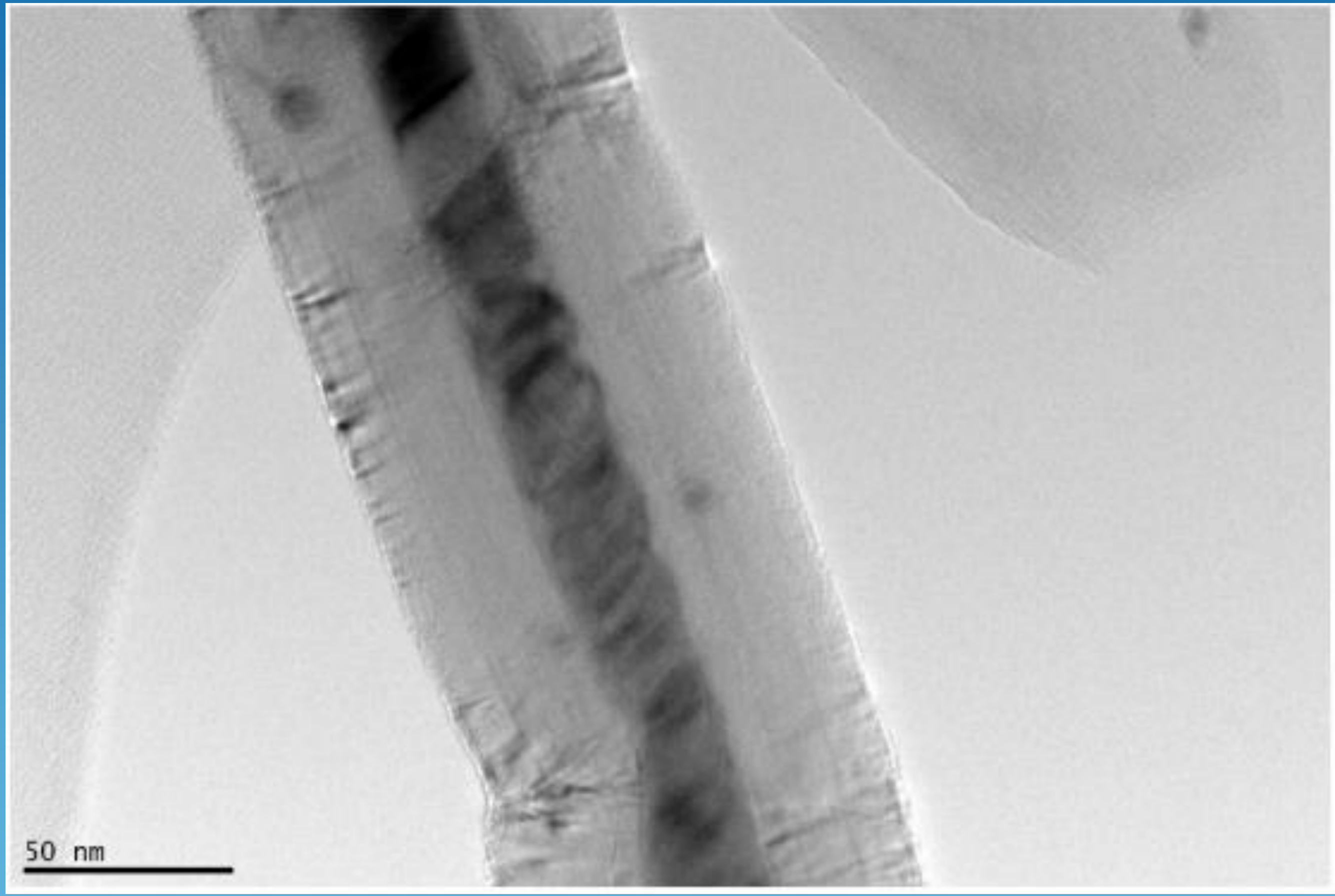


Boi et al.

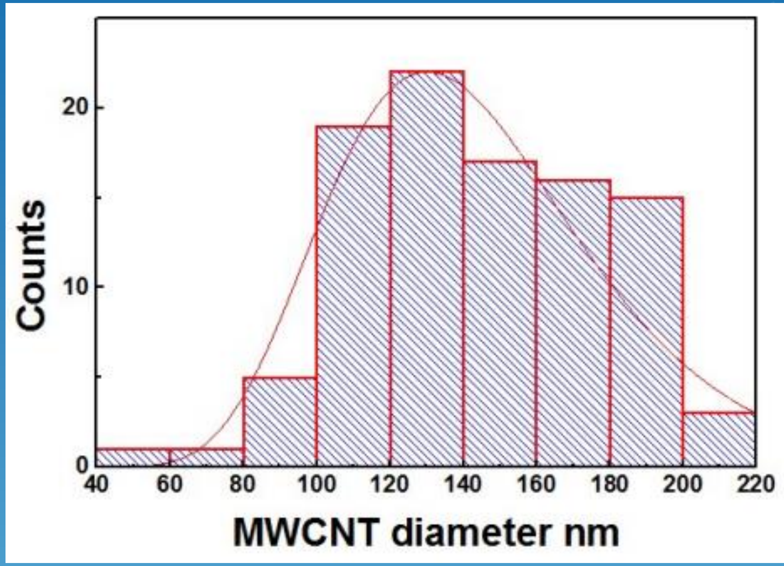
DOI: 10.1039/C7RA00240H (Paper) *RSC Adv.*, 2017, **7**, 13272-13280

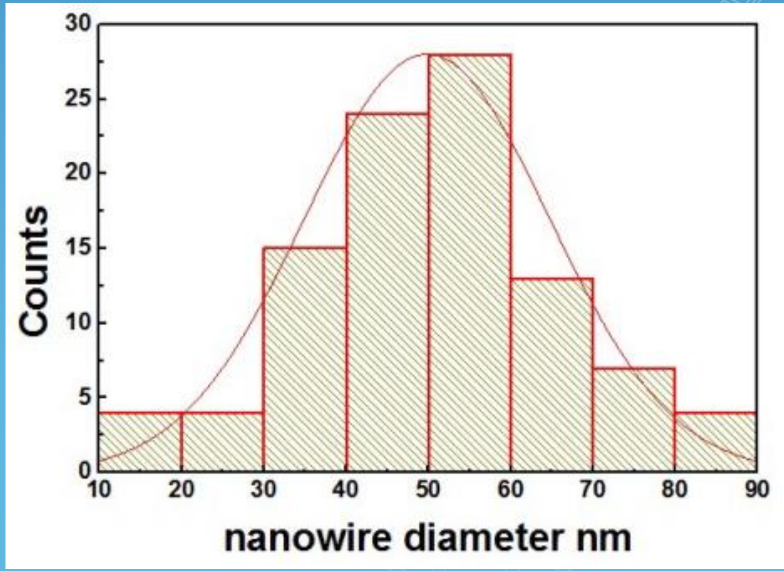
Sulfur as an additional filling promoter in CVD methods?

Only ferrocene



No sulfur

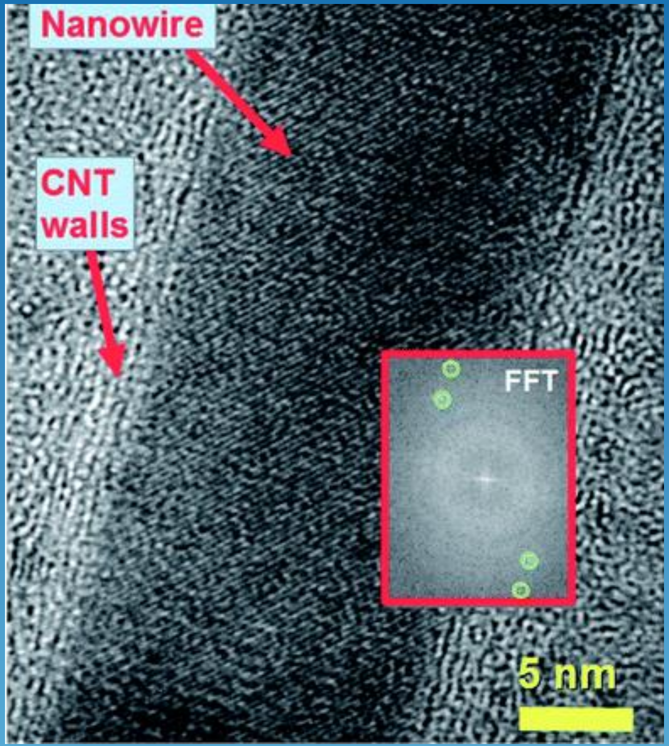




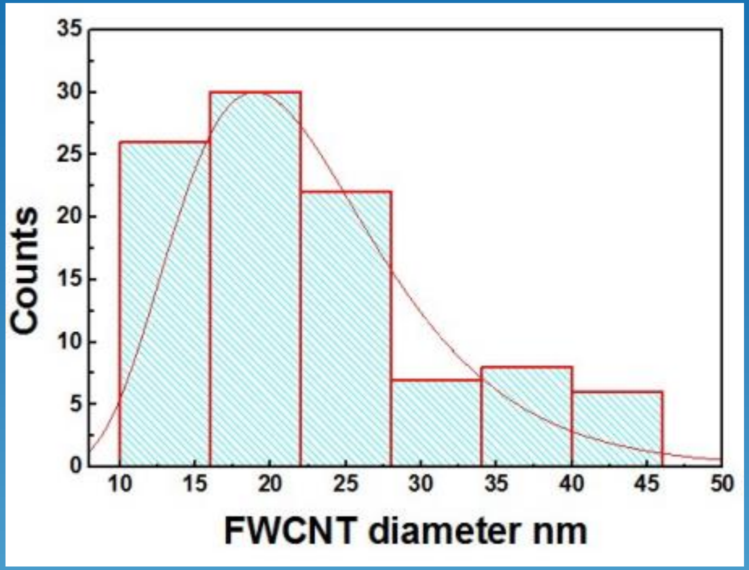
Boi et al.

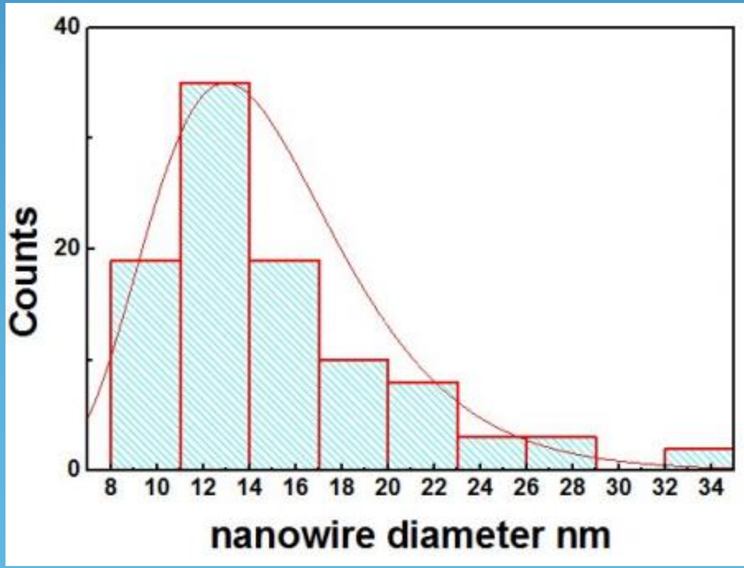
DOI: 10.1039/C7RA00240H (Paper) *RSC Adv.*, 2017, **7**, 13272-13280

Sulfur has important effects on the number of CNT walls!!

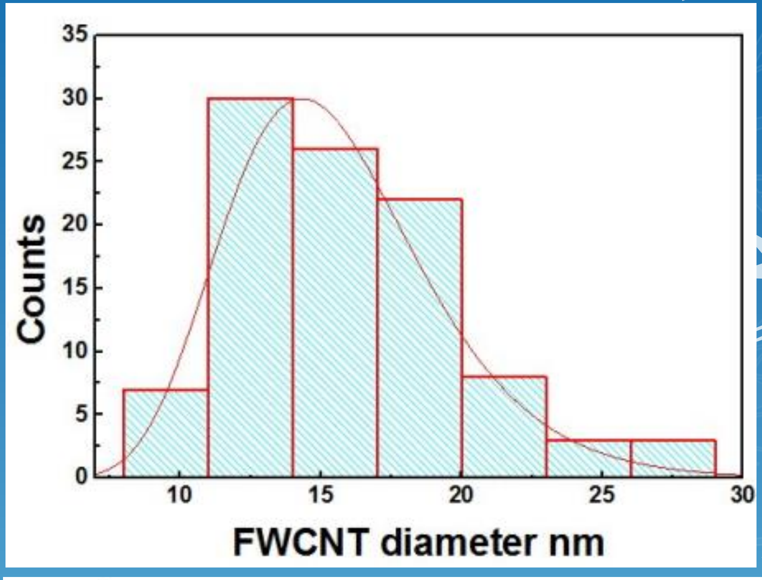


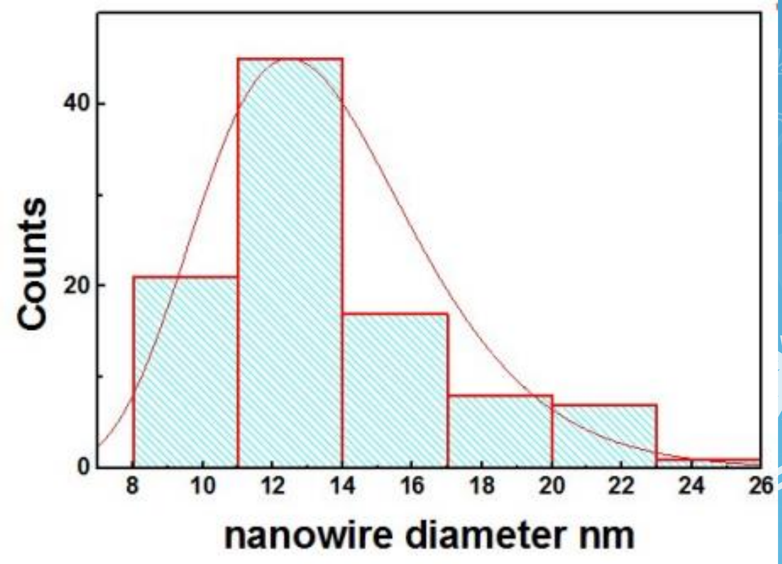
4 mg sulfur





2.5 mg sulfur





Boi et al.

DOI: 10.1039/C7RA00240H (Paper) *RSC Adv.*, 2017, **7**, 13272-13280

These results are in agreement with Wei et al.

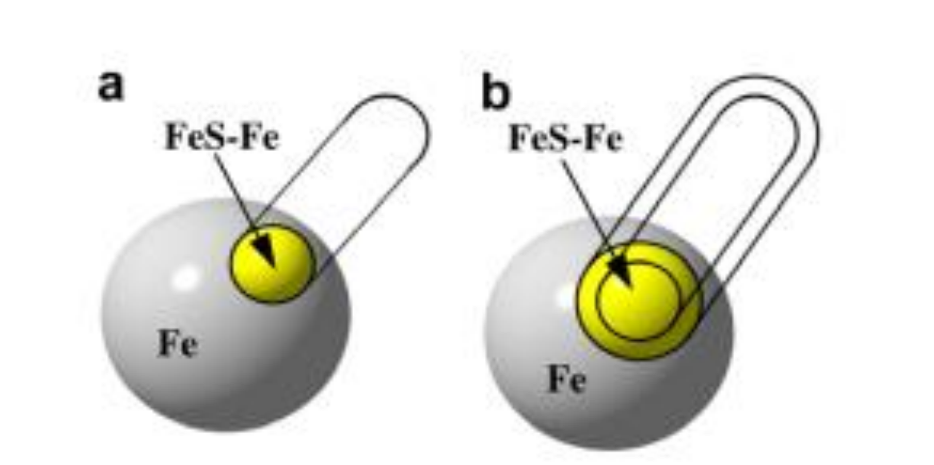
ELSEVIER

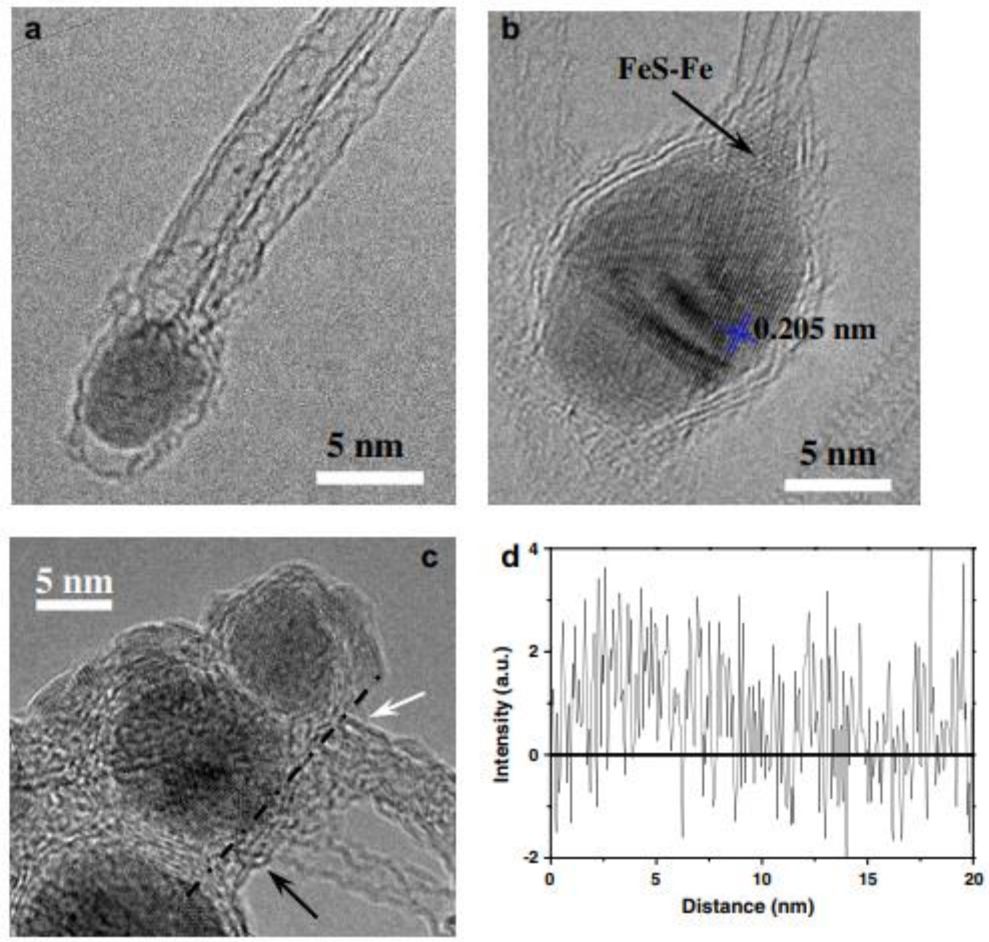
Carbon

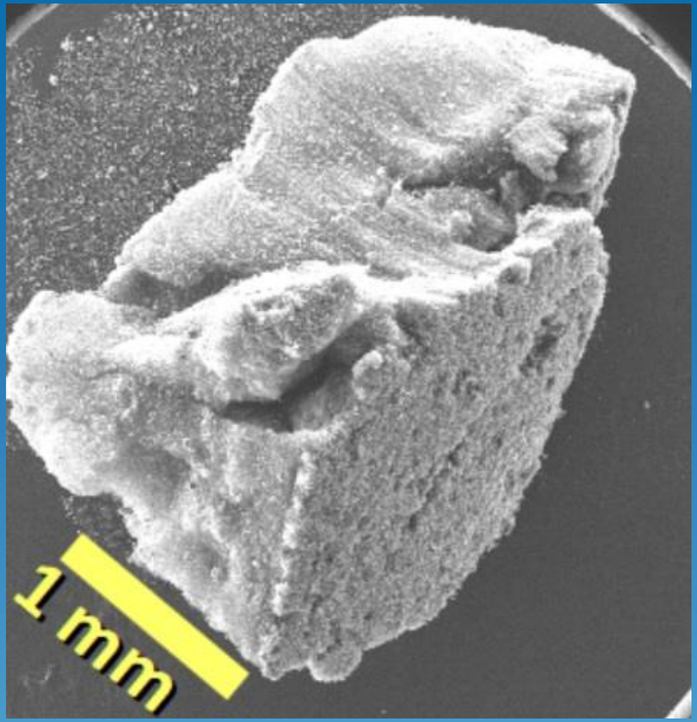
Volume 45, Issue 11, October 2007, Pages 2152-2158

The effect of sulfur on the number of layers in a carbon nanotube

Jinquan Wei [a], Hongwei Zhu [b], Yi Jia [a], Qinke Shu [a], Chuangang Li [a], Kunlin Wang [a], Bingqing Wei [b], Yanqiu Zhu [c], Zhicheng Wang [a], Jianbin Luo [a], Wenjin Liu [a], Dehai Wu [a]

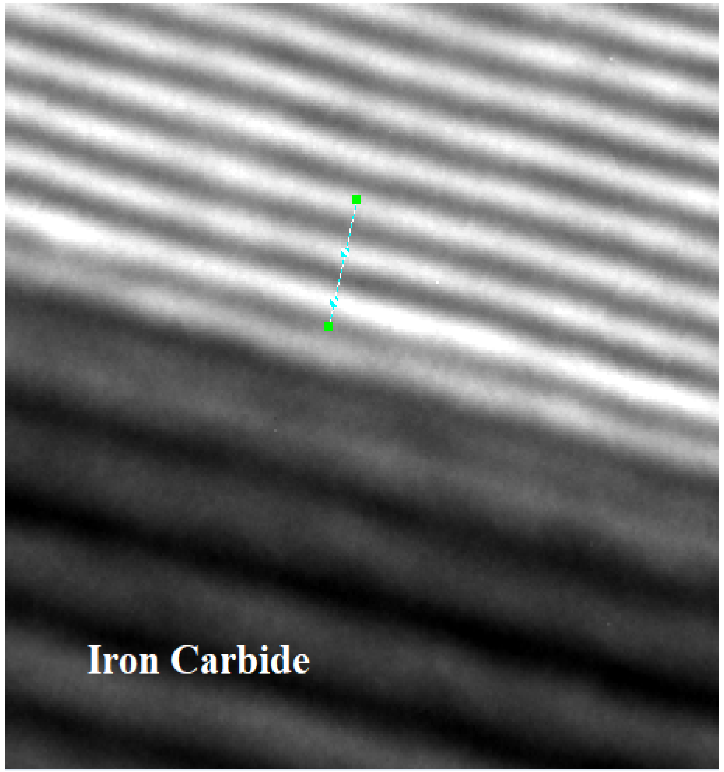


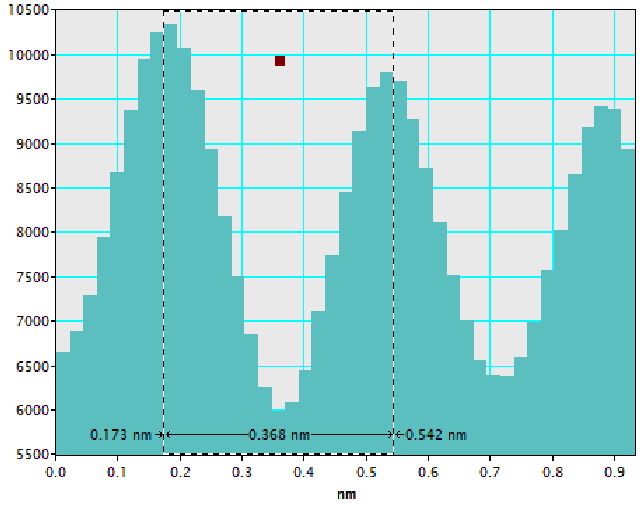




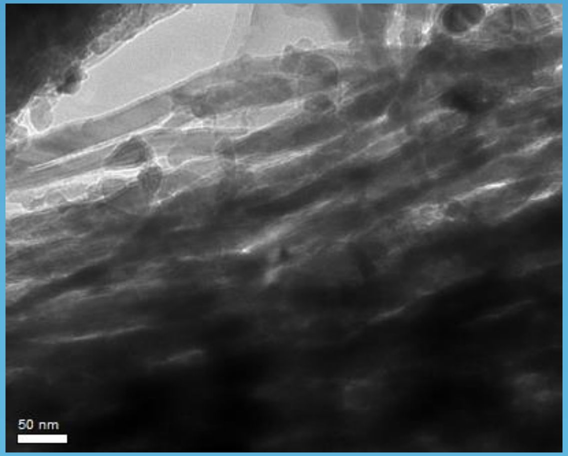
Using Chlorine and Sulfur as combined promoters!!

The employed quantity of sulfur was found to have an important impact on the structural properties of the as grown CNTs, yielding either few wall CNTs (FWCNTs) or multiwall CNTs (MWCNTs) in presence of 0.4 mg or 1.2 mg of sulfur





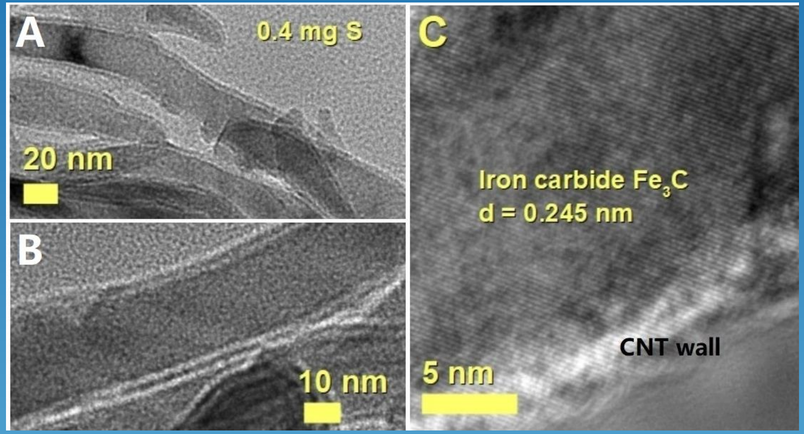
Filled CNTs



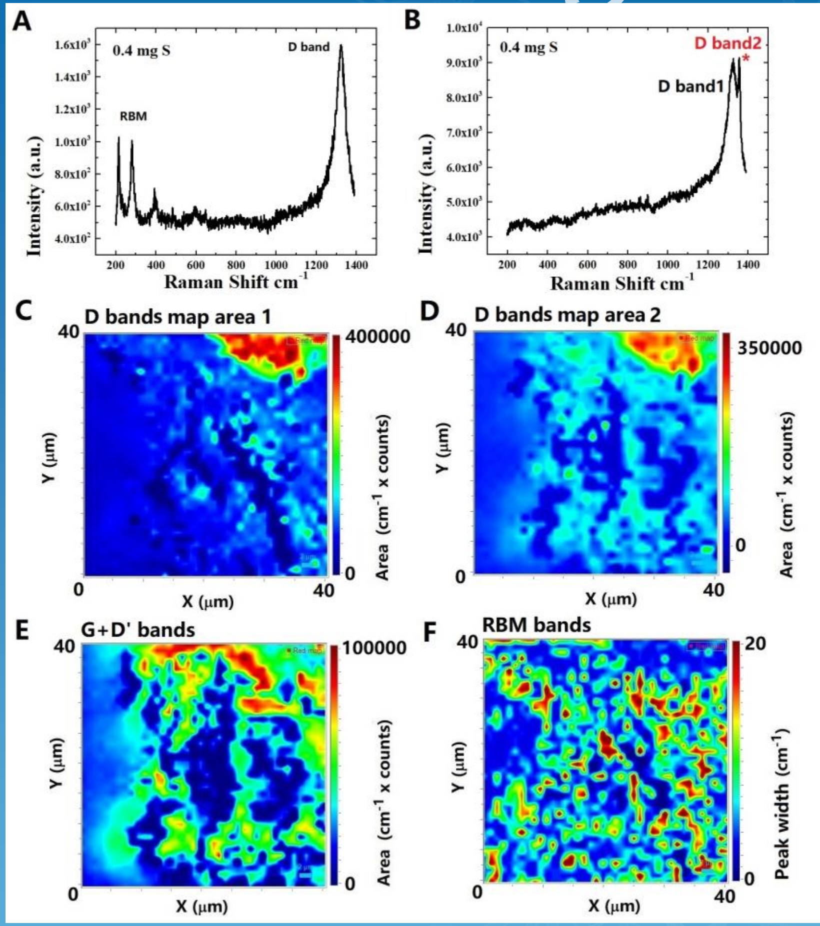
Omololu Odunmbaku+, Jiaxin Song+, Shanling Wang, Ayoub Taallah, Yixin Dai, Wenkang Li, Wenxue Li, Yi He, Jian Guo, Hong Zhang* and **Filippo S.Boi***.Nucleation of Carbon-Sulfur phases by manipulation of vertically-aligned mm-long films of iron-filled few-wall/multiwall carbon nanotubes https://doi.org/10.1016/j.cartre.2021.100102 Carbon Trends 2021 (published)

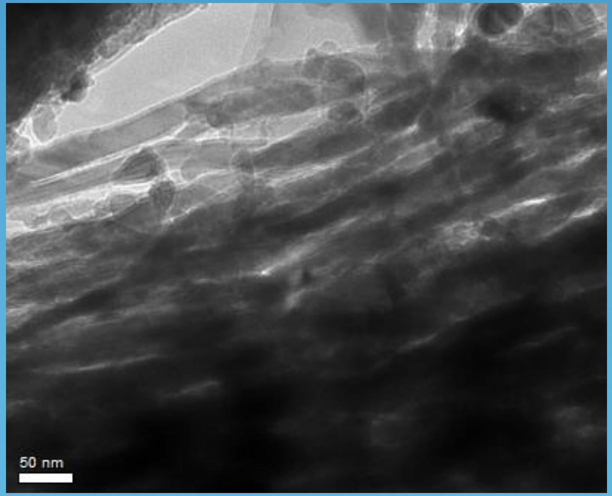
Using Chlorine and Sulfur as combined promoters!!

0.4 mg (S)+ Chlorine (dichlorobenzene)



Single wall CNT features





Omololu Odunmbaku+, Jiaxin Song+, Shanling Wang, Ayoub Taallah, Yixin Dai, Wenkang Li, Wenxue Li, Yi He, Jian Guo, Hong Zhang* and **Filippo S.Boi*.**Nucleation of Carbon-Sulfur phases by manipulation of vertically-aligned mm-long films of iron-filled few-wall/multiwall carbon nanotubes https://doi.org/10.1016/j.cartre.2021.100102 Carbon Trends 2021 (published)

Carbon Shell Manipulation: The role of Sulfur

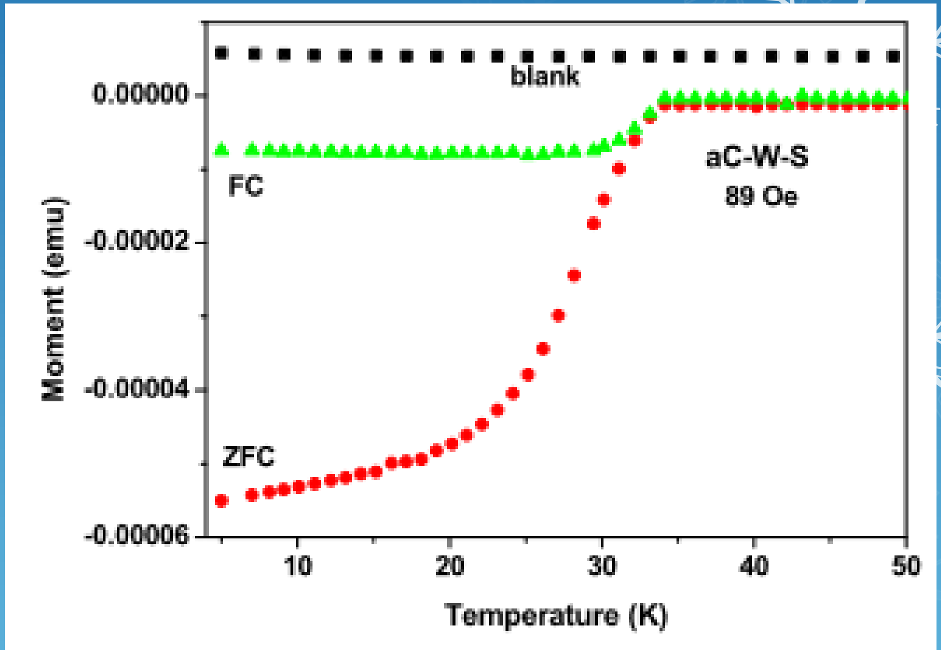
Letter | Published: 26 January 2013

Superconductivity in Sulfur-Doped Amorphous Carbon Films

I. Felner, O. Wolf & O. Millo

Journal of Superconductivity and Novel Magnetism **26**, 511–514 (2013) | Cite this article

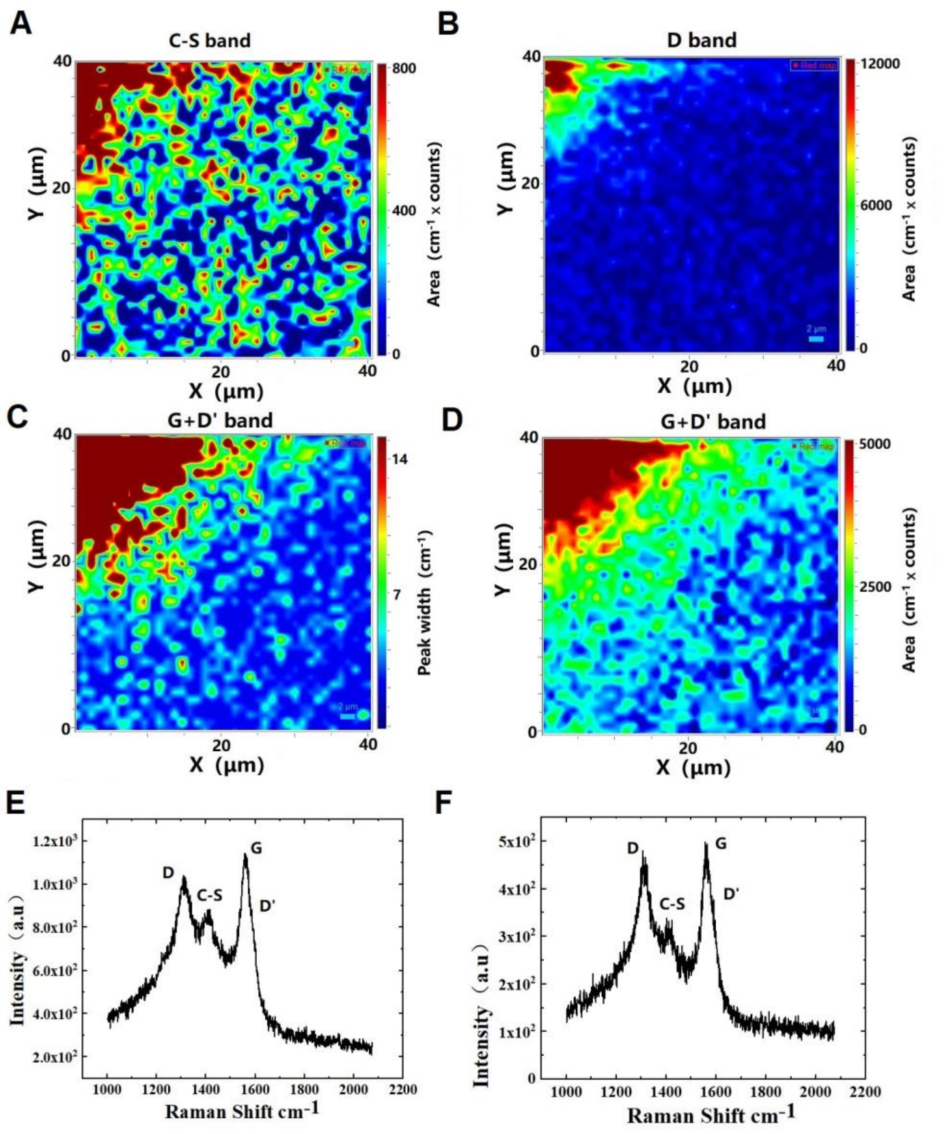
Prepared by heating the blank film with sulfur powder (Aldrich Chemical Company, Inc.) in evacuated quartz tube at 250 ◦C for 24 h before cooling down to room temperature

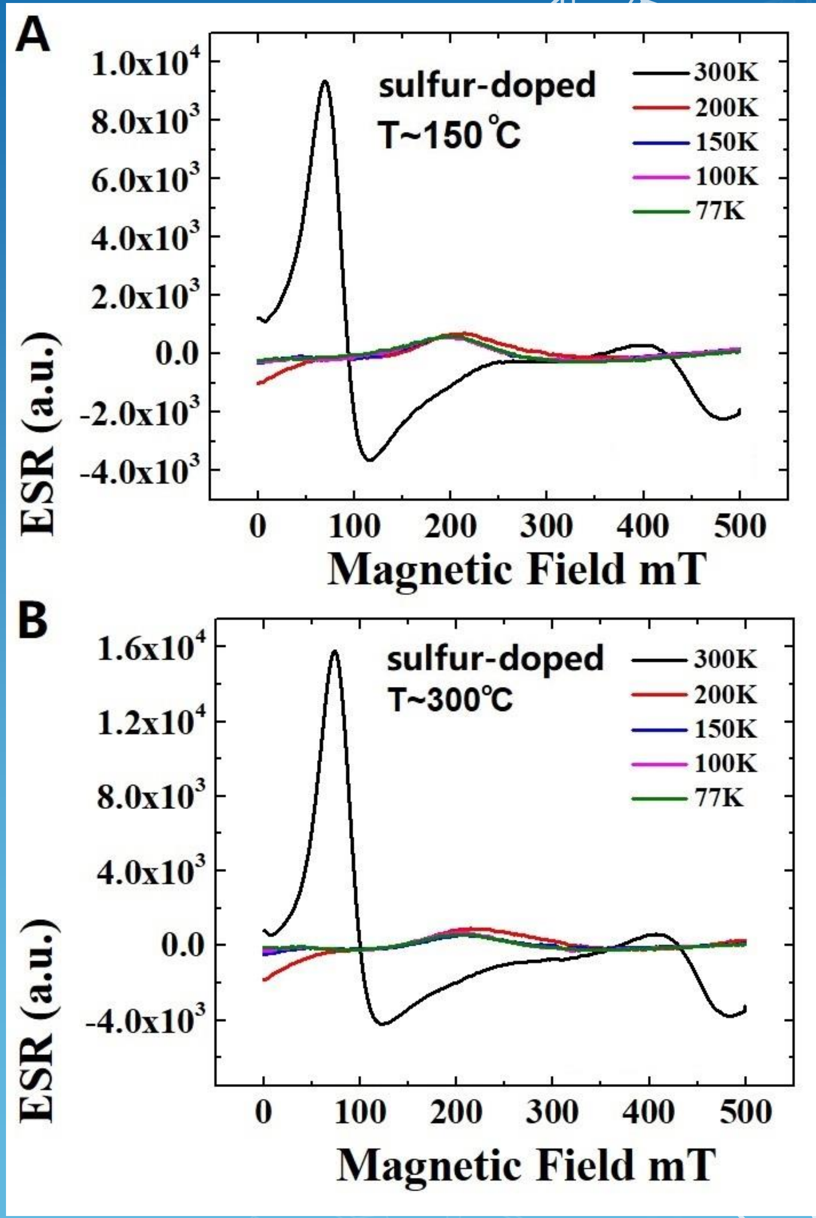


Manipulating the CNTs shell with sulfur may open-up new application perspectives

Annealing 150 °C

Carbon-Sulfur phases (cofirmed by EDS)

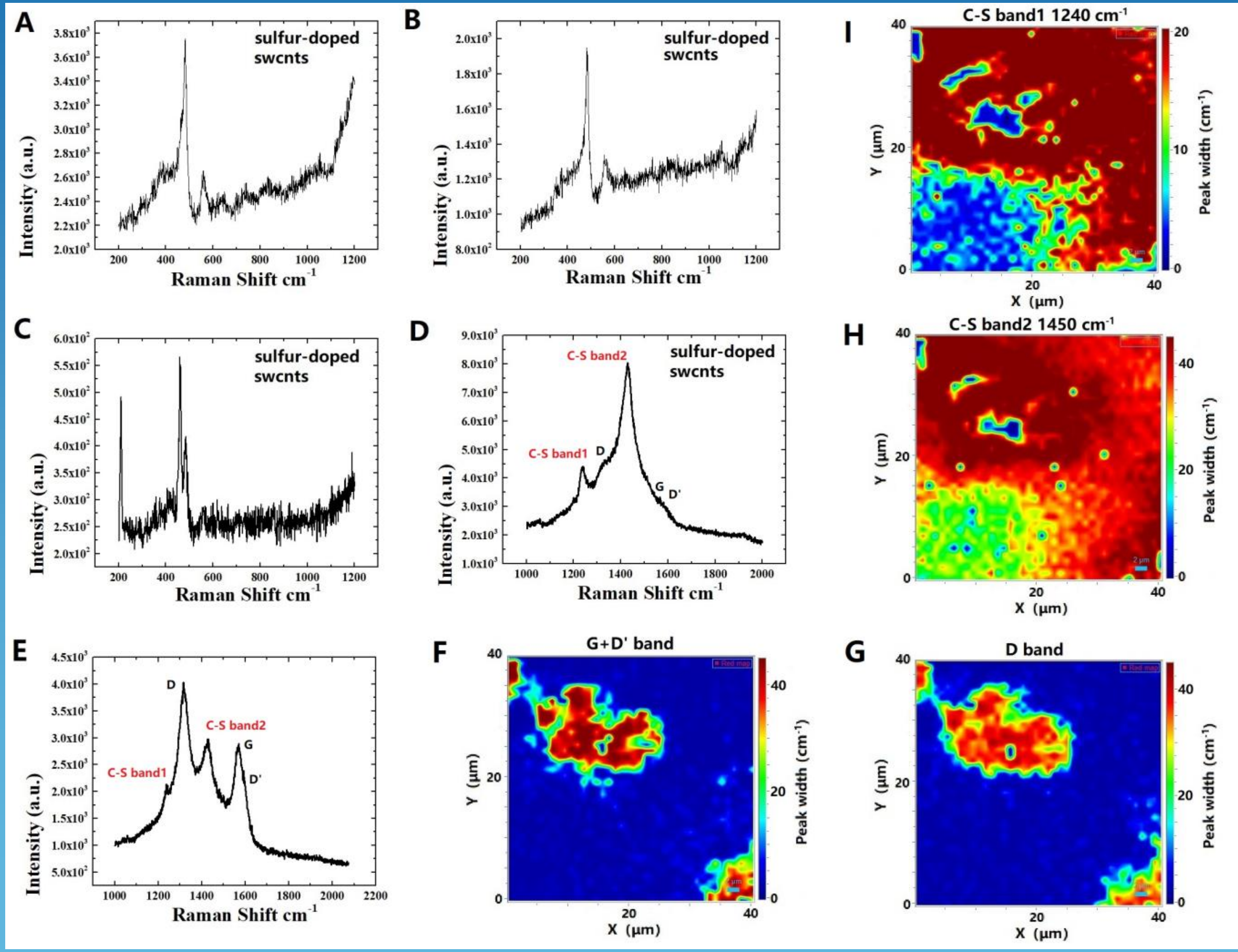


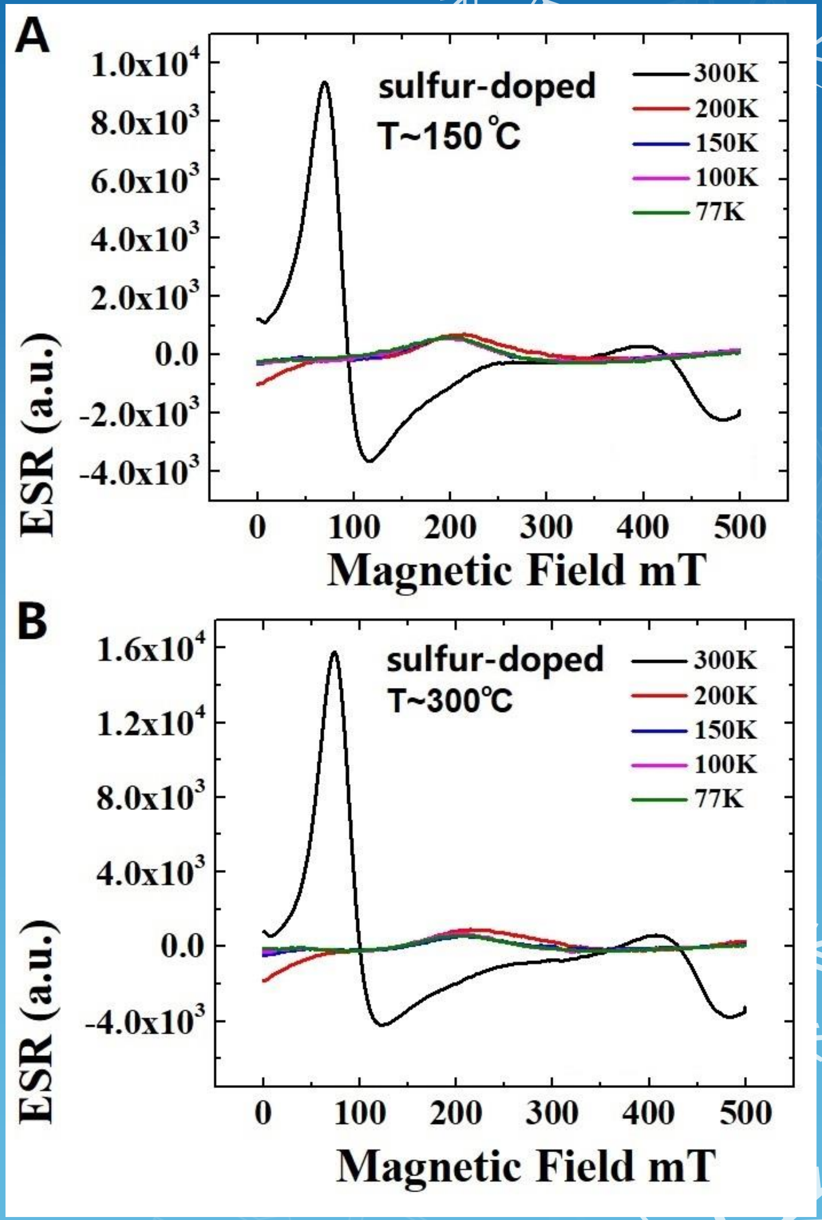


Manipulating the CNTs shell with sulfur may open-up new application perspectives

Annealing 300 °C

Carbon-Sulfur phases (cofirmed by EDS)





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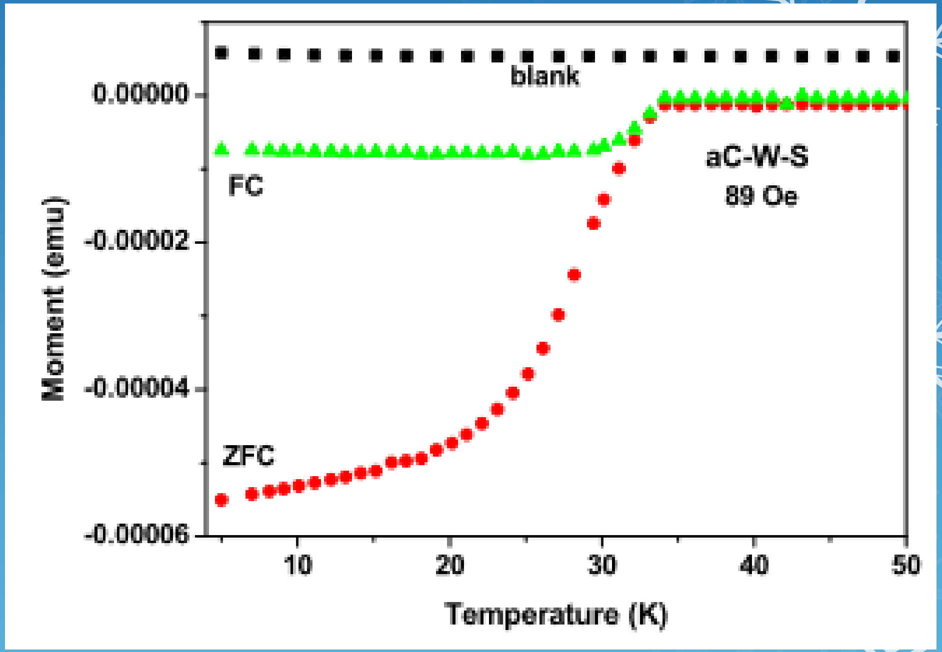
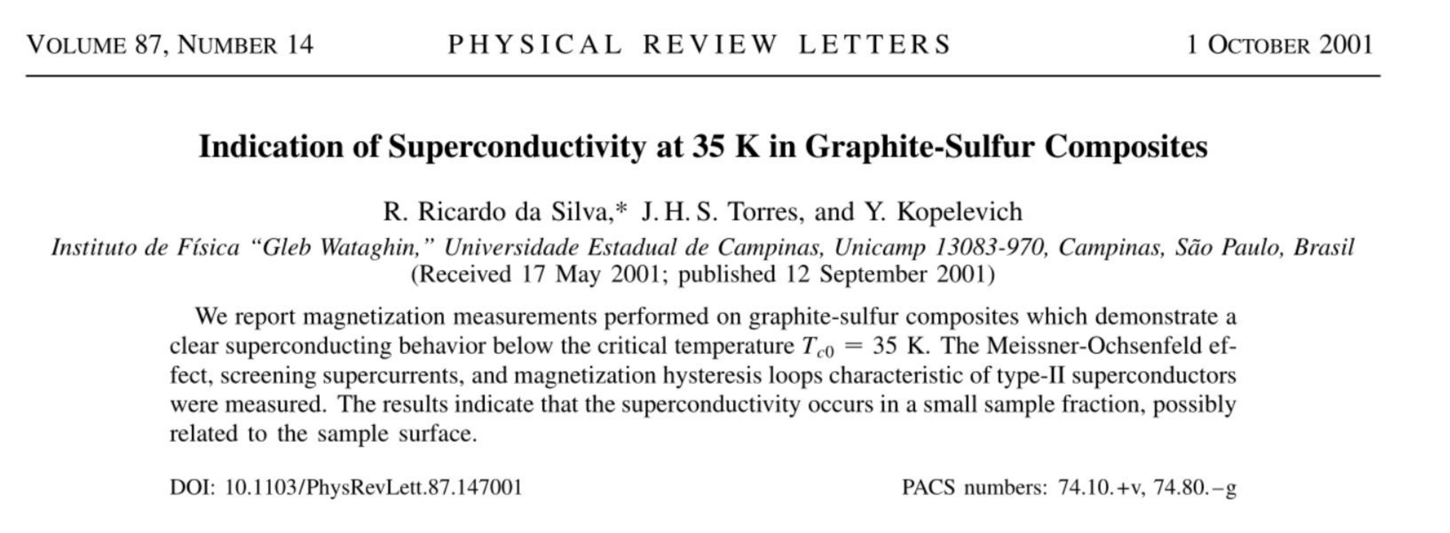


Carbon Shell Manipulation: The role of Sulfur

In 2001, Silva et al. reported the superconductivity occurrence in graphite-sulfur (C-S) composite samples below 35 K.

Sulfur may therefore play a particular role towards modification of the CNT-shell properties



VOLUME 87, NUMBER 14 PHYSICAL REVIEW LETTERS 1 OCTOBER 2001

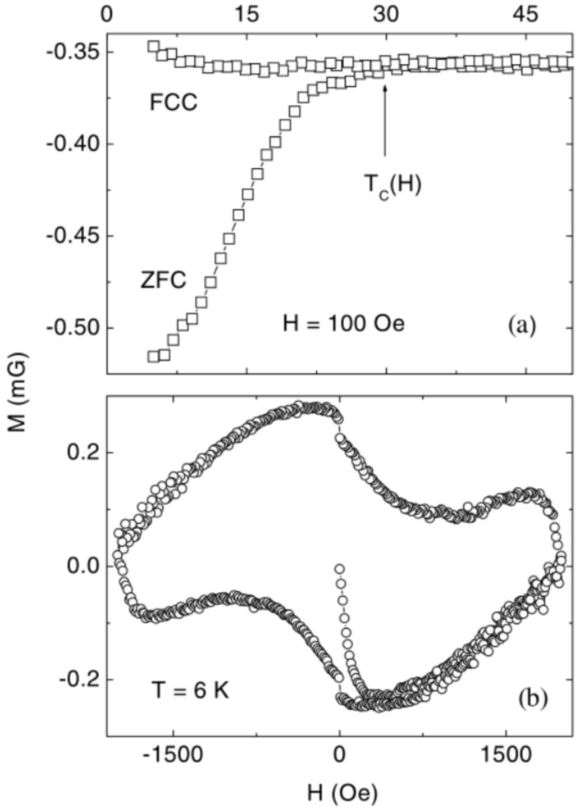
Indication of Superconductivity at 35 K in Graphite-Sulfur Composites

R. Ricardo da Silva,* J. H. S. Torres, and Y. Kopelevich
Instituto de Física "Gleb Wataghin," Universidade Estadual de Campinas, Unicamp 13083-970, Campinas, São Paulo, Brasil

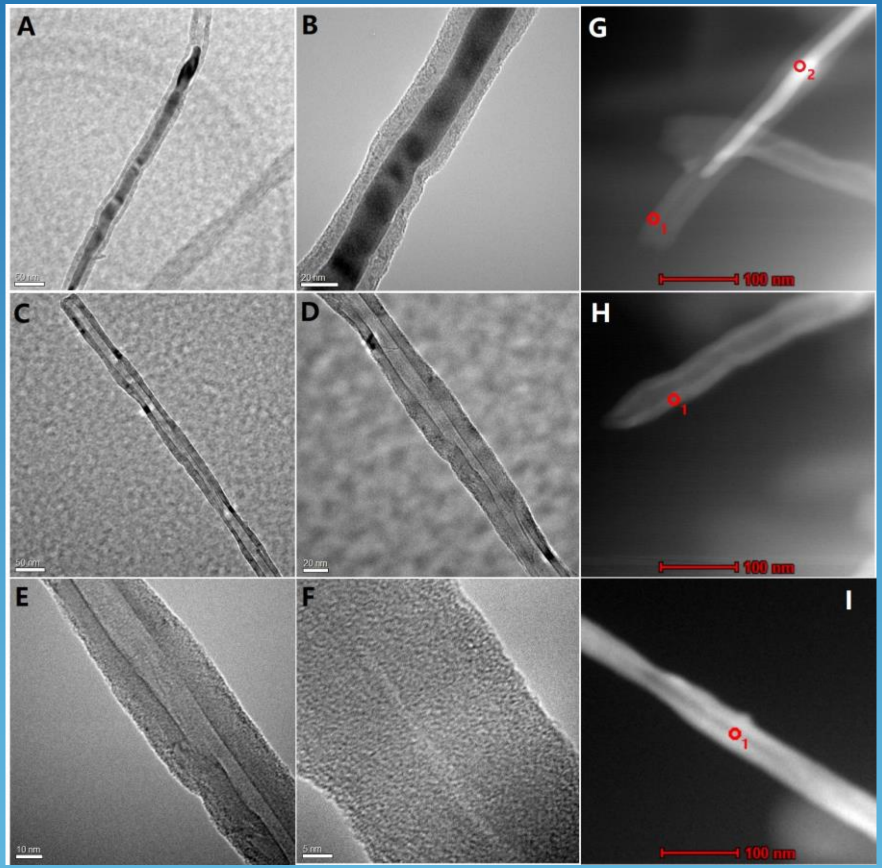
(Received 17 May 2001; published 12 September 2001)

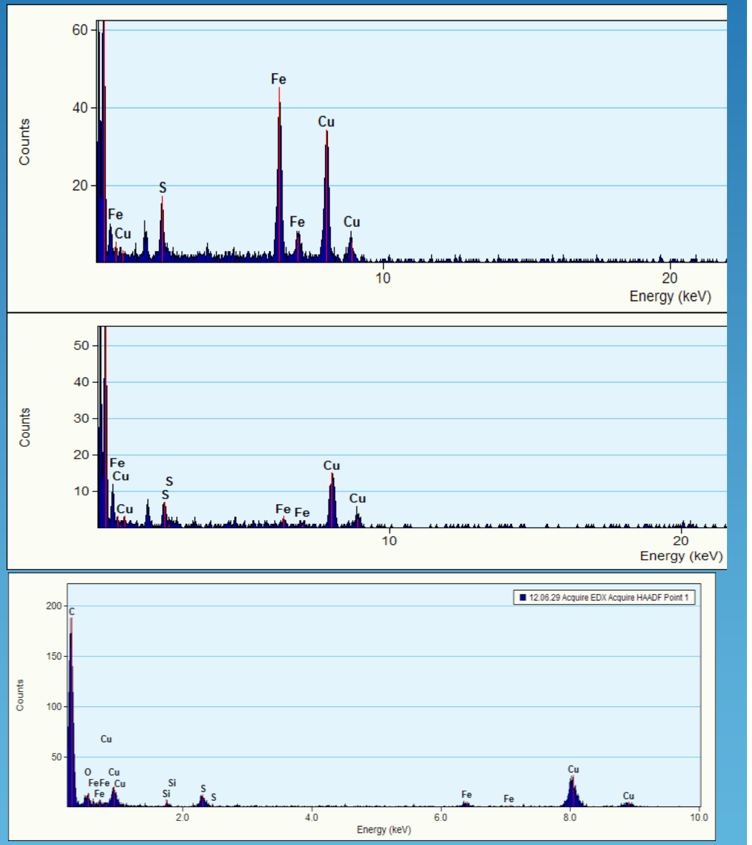
We report magnetization measurements performed on graphite-sulfur composites which demonstrate a clear superconducting behavior below the critical temperature $T_{c0} = 35$ K. The Meissner-Ochsenfeld effect, screening supercurrents, and magnetization hysteresis loops characteristic of type-II superconductors were measured. The results indicate that the superconductivity occurs in a small sample fraction, possibly related to the sample surface.

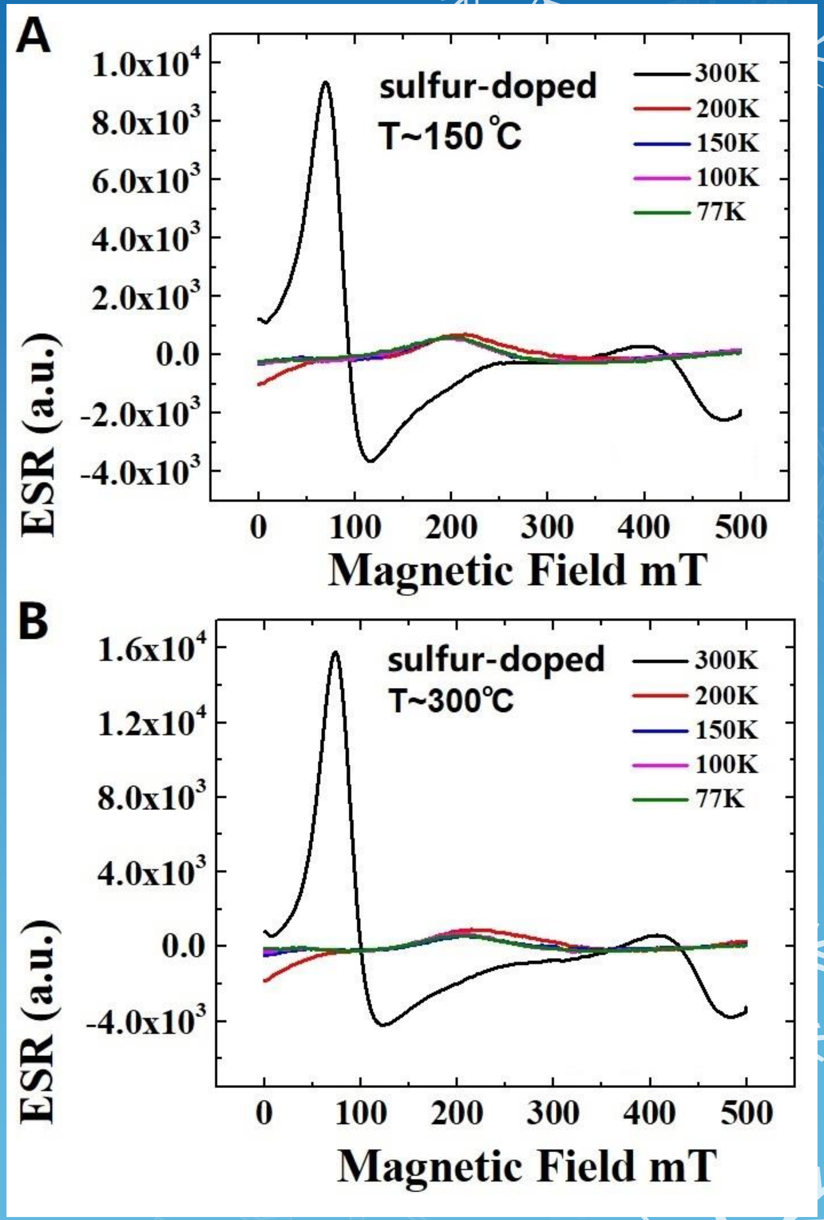
DOI: 10.1103/PhysRevLett.87.147001 PACS numbers: 74.10.+v, 74.80.–g



Manipulating the CNTs shell with sulfur may open-up new application perspectives

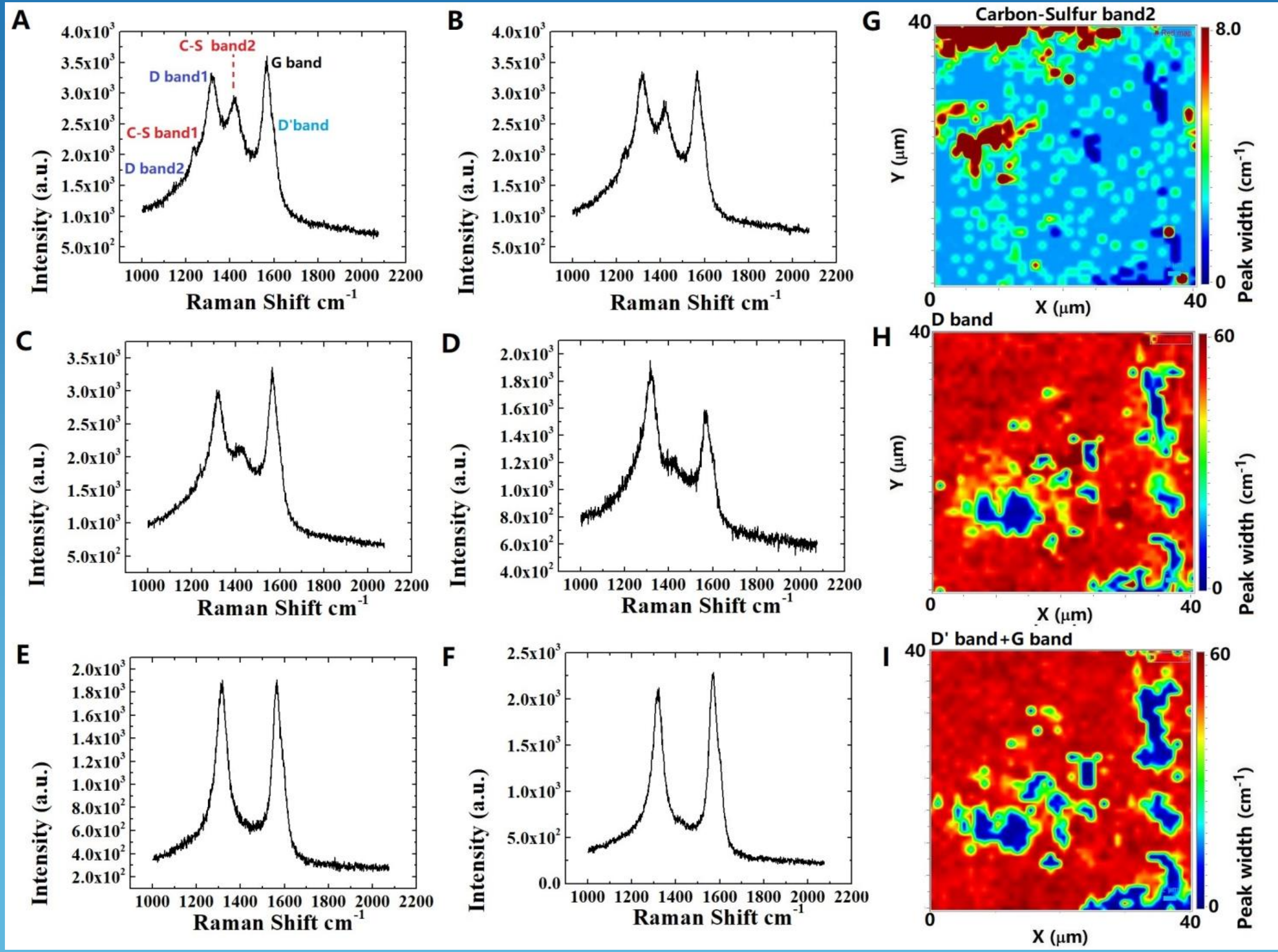


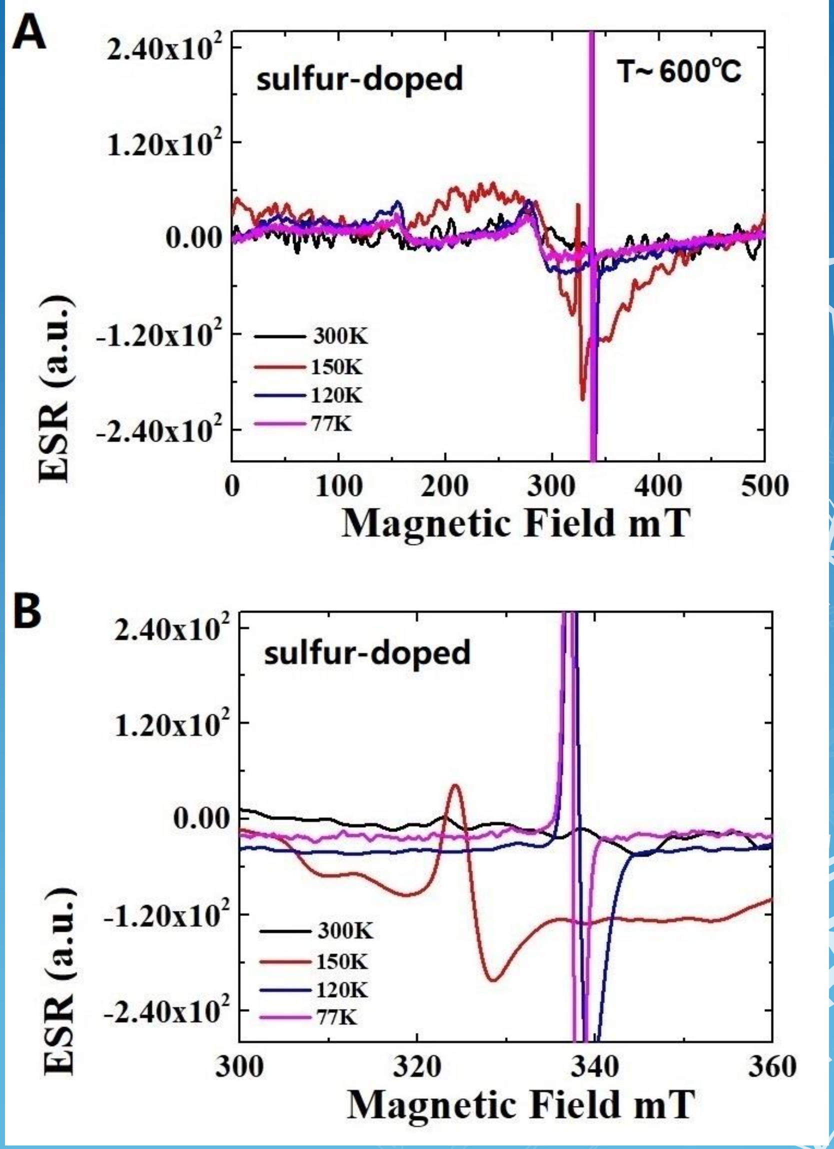




Manipulating the CNTs shell with sulfur may open-up new application perspectives

Annealing 600 °C



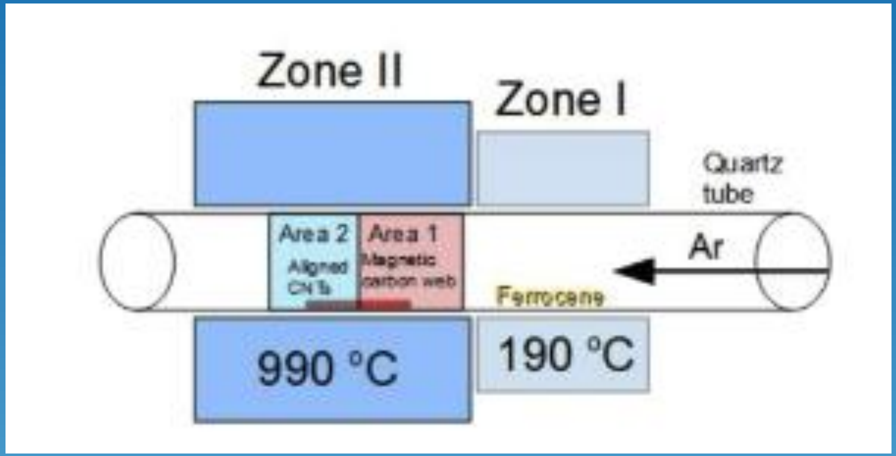


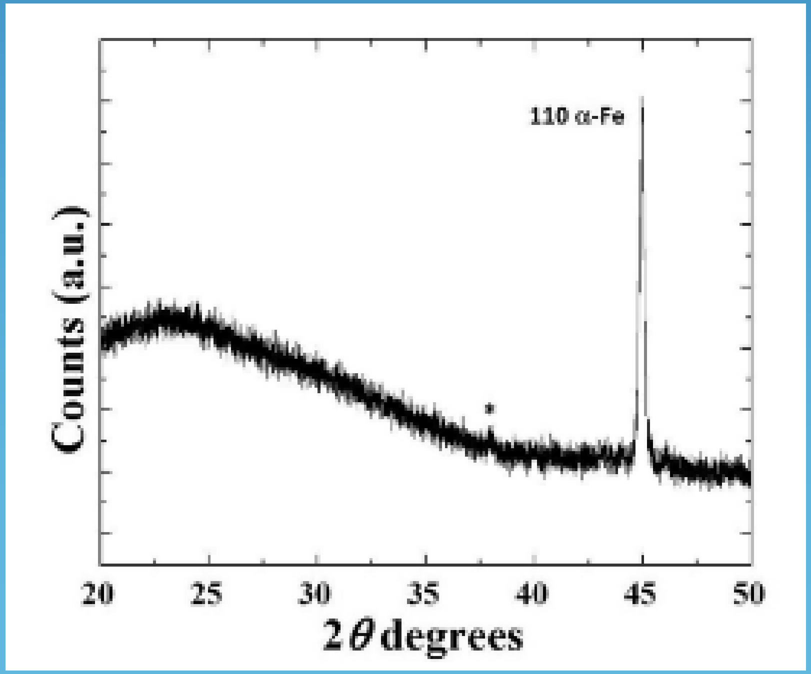
Ferromagnetically filled Carbon Foams

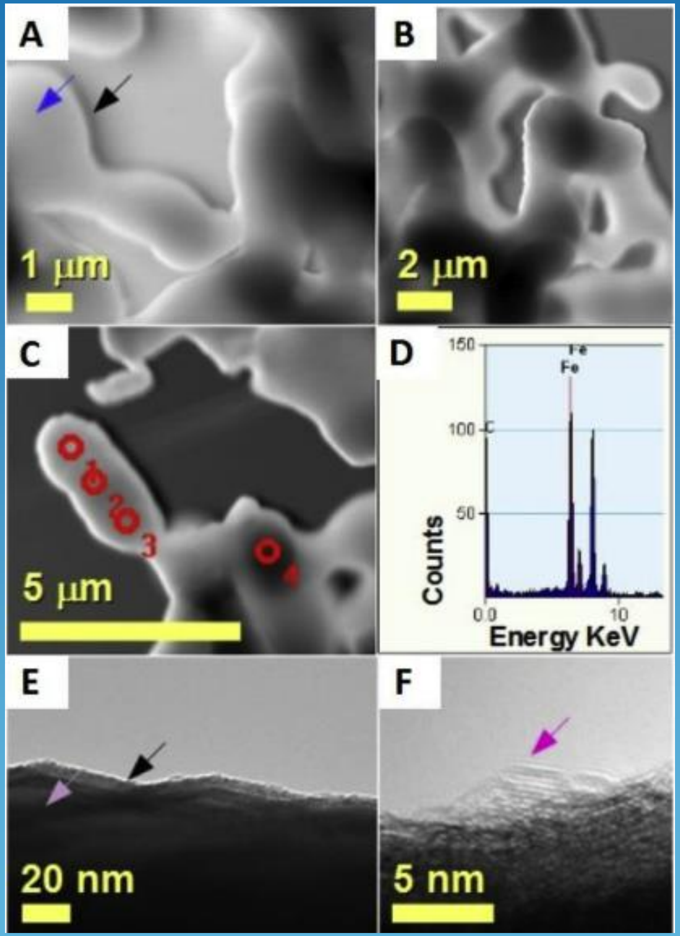
Ferromagnetically filled graphitic-carbon foams are a new class of foam-materials characterized by an open-cell structure continuously filled with ferromagnetic crystals.

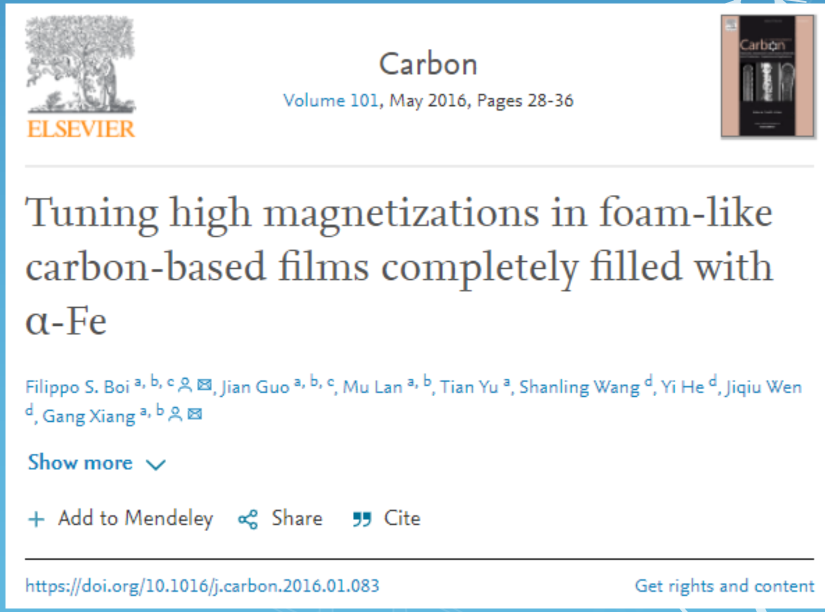
Ferromagnetically filled Carbon Foams

2016 Early Observation








ELSEVIER
Carbon
Volume 101, May 2016, Pages 28-36

Tuning high magnetizations in foam-like carbon-based films completely filled with α-Fe

Filippo S. Boi [a, b, c], Jian Guo [a, b, c], Mu Lan [a, b], Tian Yu [a], Shanling Wang [d], Yi He [d], Jiqiu Wen [d], Gang Xiang [a, b]

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https://doi.org/10.1016/j.carbon.2016.01.083

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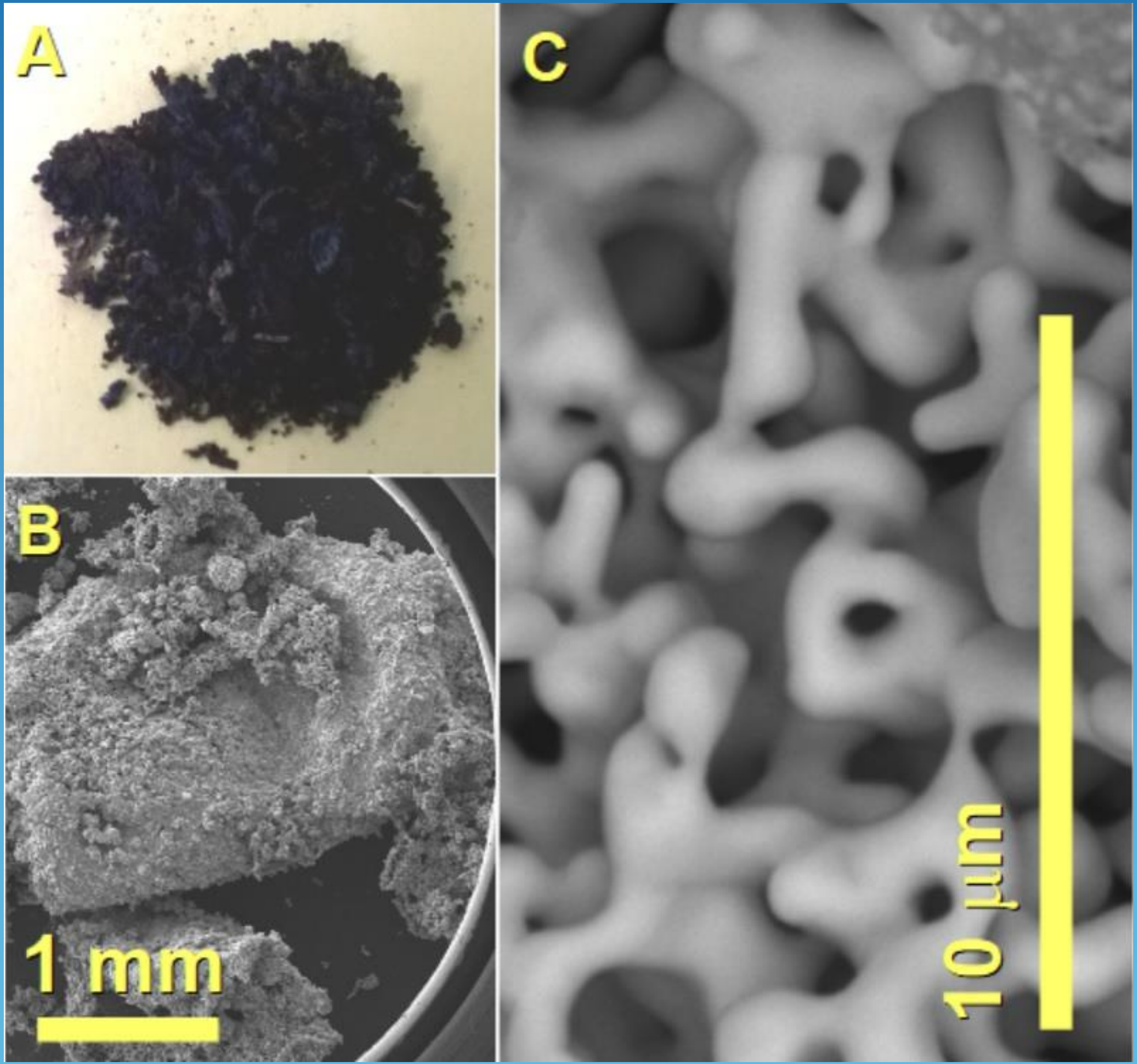
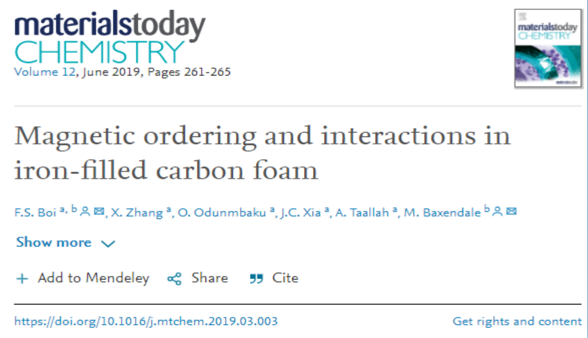
- Fe@CFM by fusion of Fe@CNOs

Optical and SEM micrographs of a Fe-filled CFM





materialstoday CHEMISTRY
Volume 12, June 2019, Pages 261-265

Magnetic ordering and interactions in iron-filled carbon foam

F.S. Boi [a, b], X. Zhang [a], O. Odunmbaku [a], J.C. Xia [a], A. Taallah [a], M. Baxendale [b]

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https://doi.org/10.1016/j.mtchem.2019.03.003

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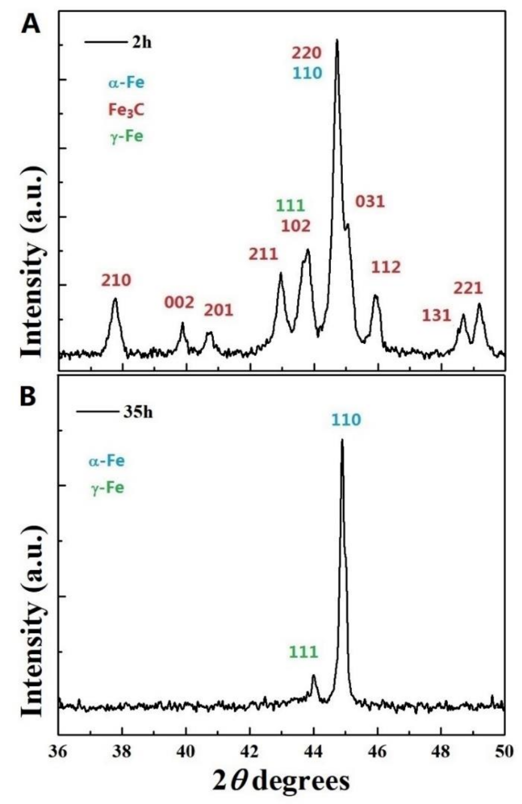
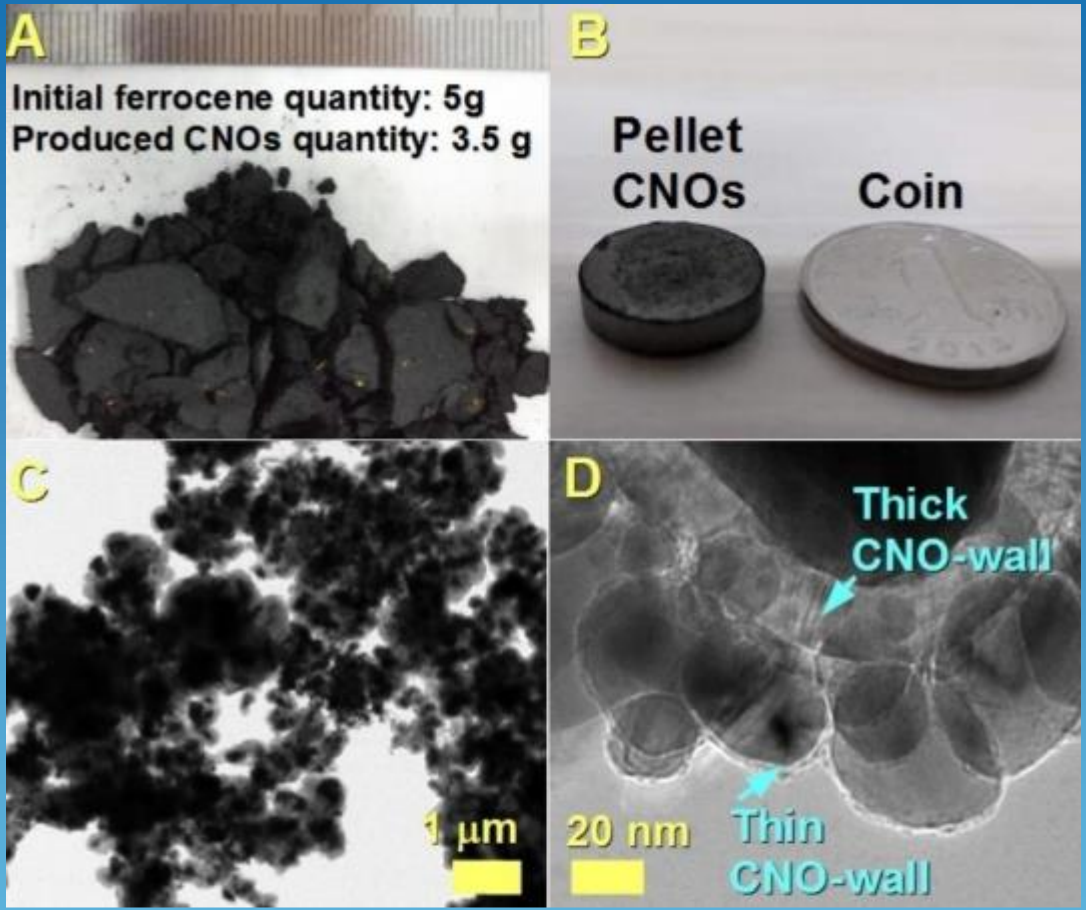
XRD measurements of α Fe-filled CFM



Mass production through fusion of CNOs

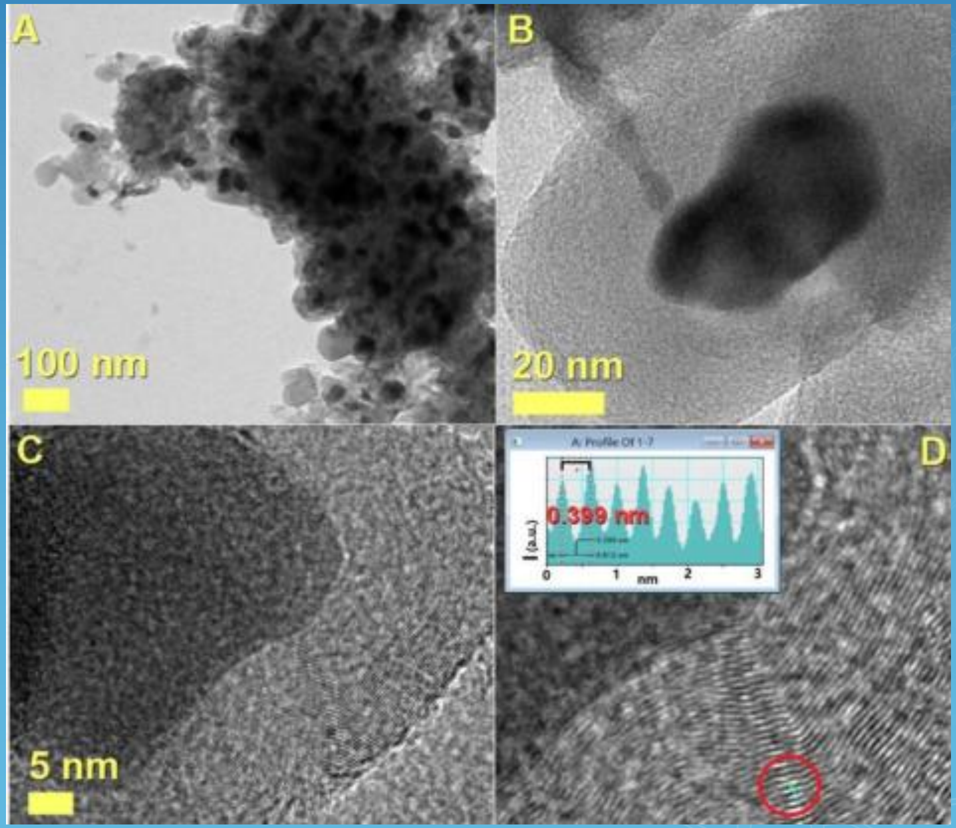
5 g of ferrocene were evaporated at 110 °C and pyrolysed at 990 °C under an Ar flow rate of 5 ml min−1. The duration of the growth experiment at the pyrolysis temperature was of 1 h.




PAPER • OPEN ACCESS
Temperature-dependent c-axis lattice-spacing reduction and novel structural recrystallization in carbon nano-onions filled with Fe_3C/α-Fe nanocrystals

Filippo S Boi[4,4,1], Jiaxiang Li[1], Omololu Odunmbaku[1], Mengjiao Liu[2], Daniel Medranda[1], Ayoub Taallah[1], Li Lei[3] and Shanling Wang[4,4,4]

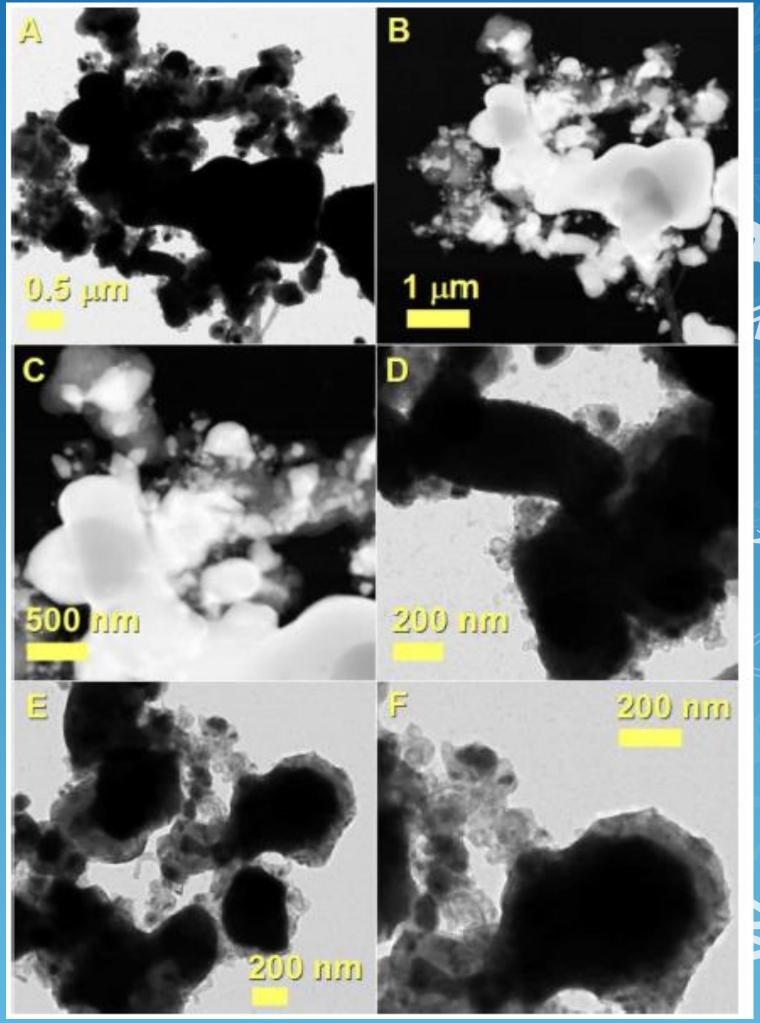
Published 30 July 2020 • © 2020 The Author(s). Published by IOP Publishing Ltd
Nano Express, Volume 1, Number 2



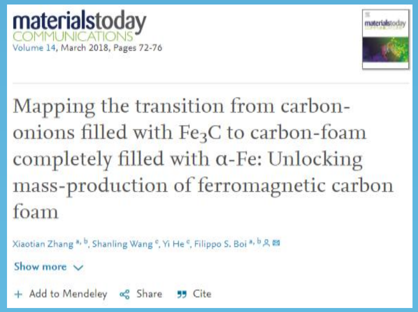
- CFM as capsule for ferromagnetic materials

- - These materials can be obtained through an innovative approach involving fusion of Fe_3C filled CNOs

- Magnetometry together with electron spin resonance, and calorimetric measurements on iron-filled carbon foam reveal ferromagnetic responses at room temperature with a transition to antiferromagnetic behavior in some fraction of the filling at 130 K.



TEM micrographs evidencing the process of CNOs fusion which leads to CFM formation



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Mapping the transition from carbon-onions filled with Fe_3C to carbon-foam completely filled with α-Fe: Unlocking mass-production of ferromagnetic carbon foam

Xiaotian Zhang [a, b], Shanling Wang [c], Yi He [c], Filippo S. Boi [a, b]

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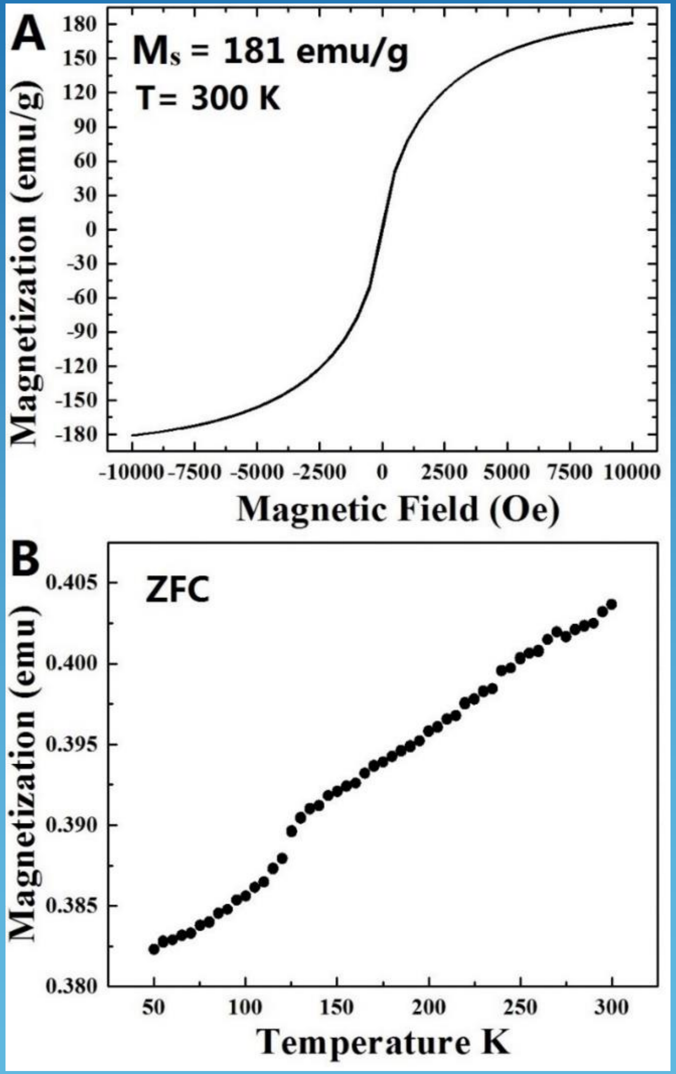
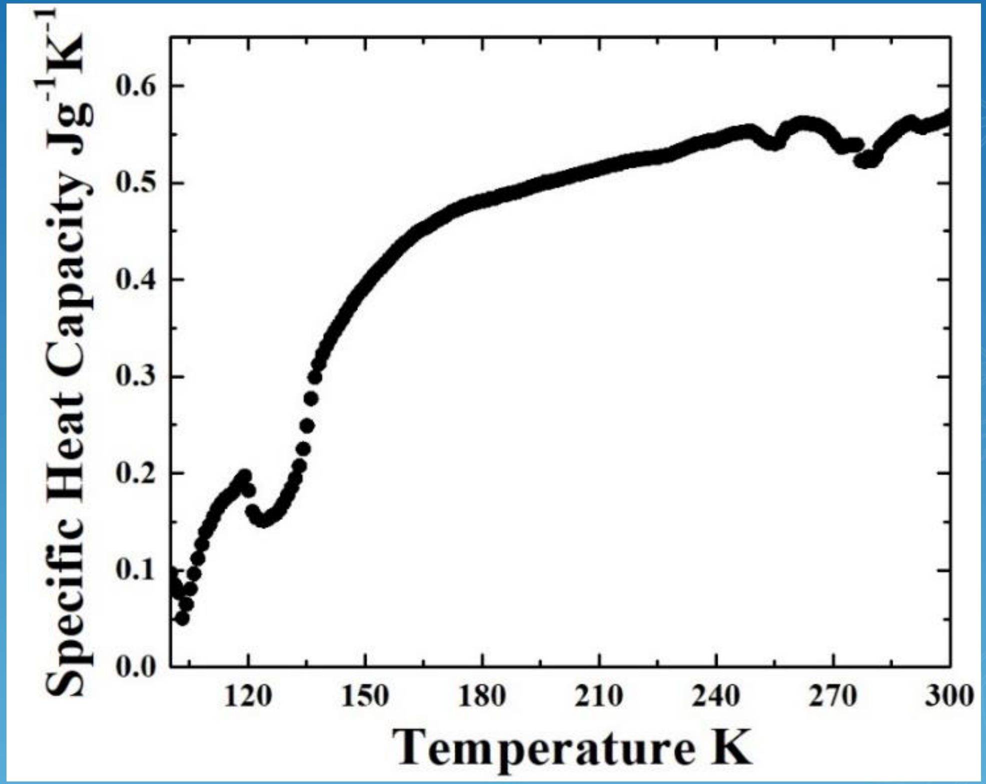
CFM as capsule for ferromagnetic materials

Magnetization measurement evidence high saturation magnetizations and an unusual alpha to gamma Fe transition



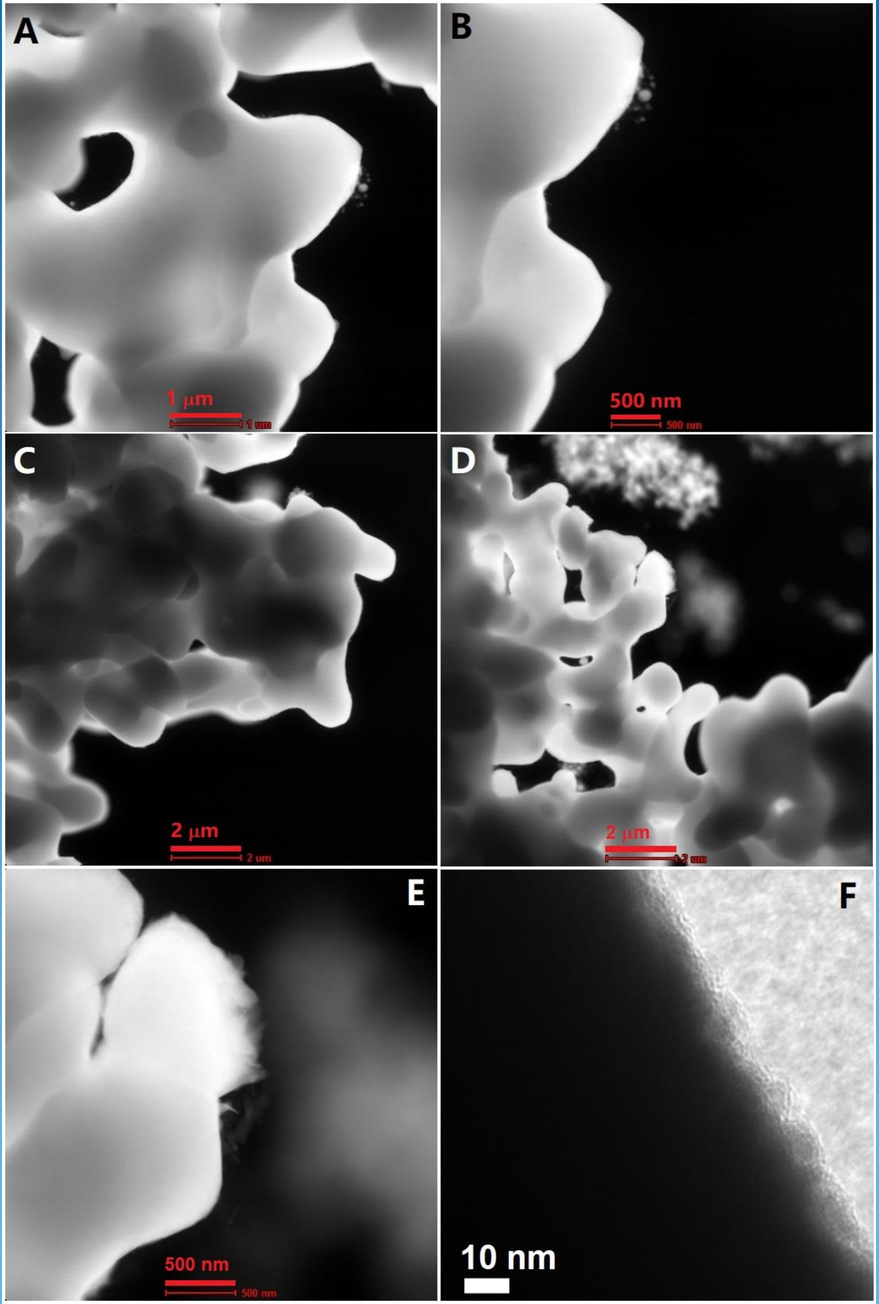
Specific heat capacity vs. temperature obtained from the DSC measurement. DSC, differential scanning calorimetry.

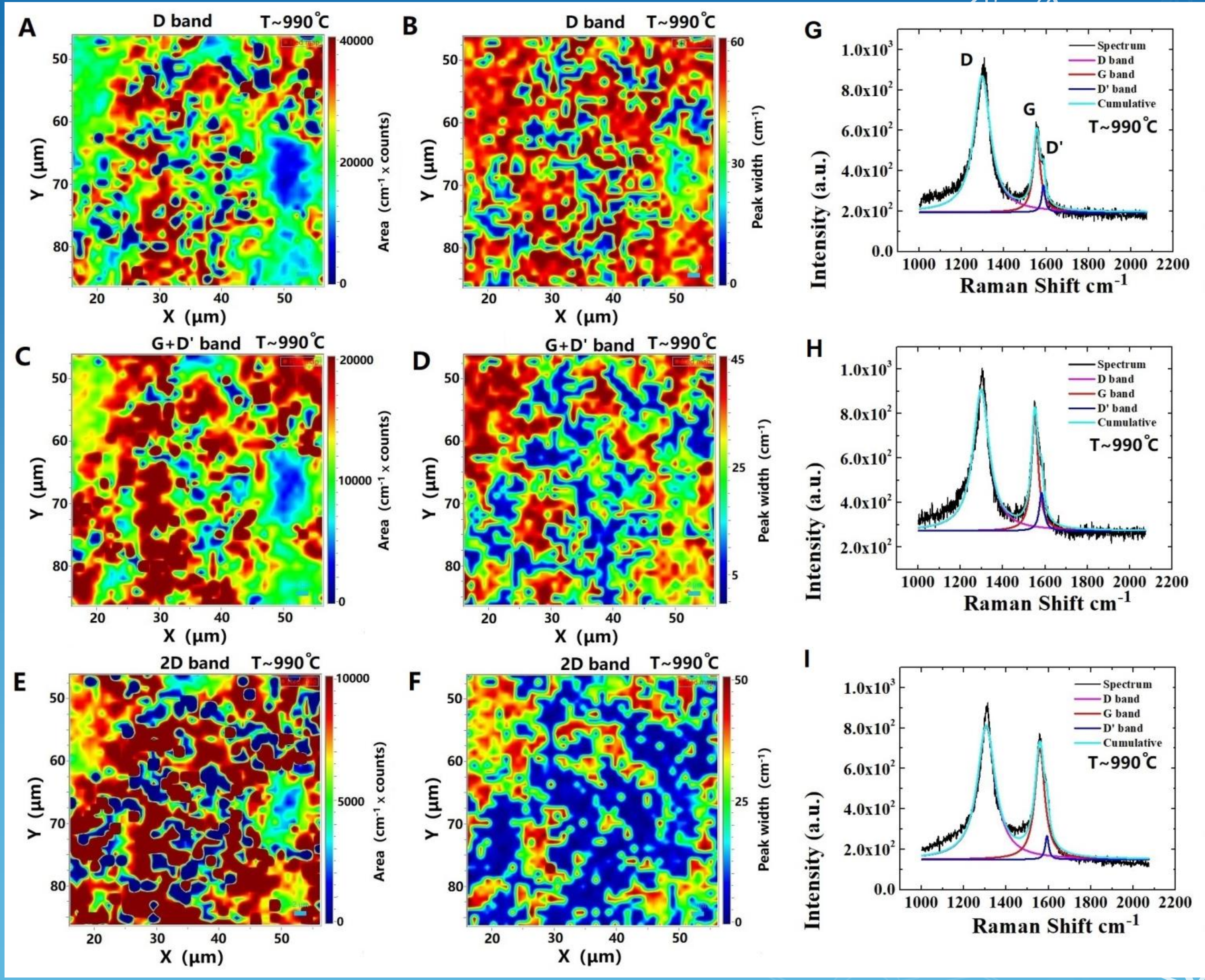


unusual alpha to gamma Fe transition

Manipulating the CFM Properties

(In preparation)

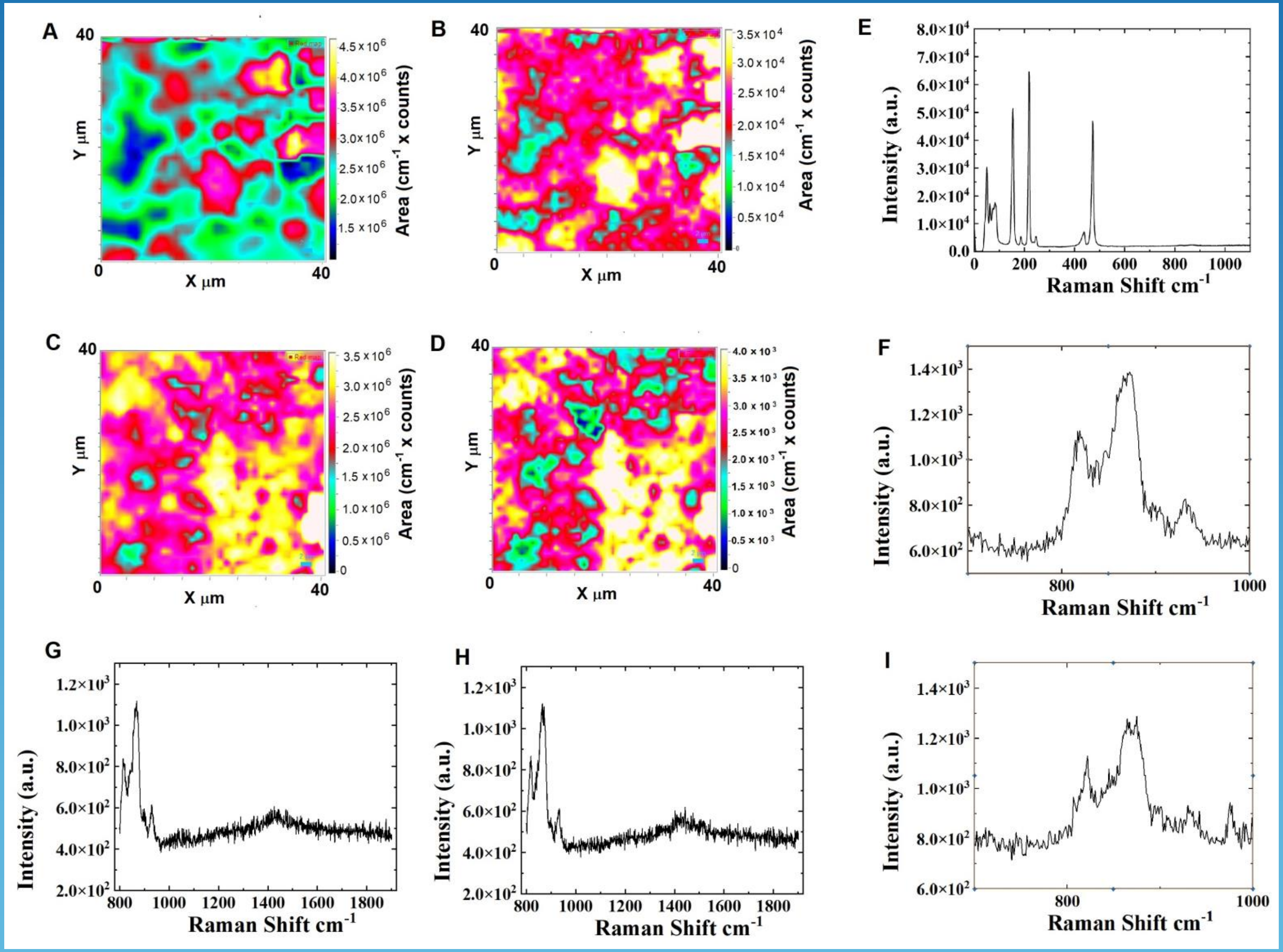




Manipulating the CFM Properties

(In preparation)

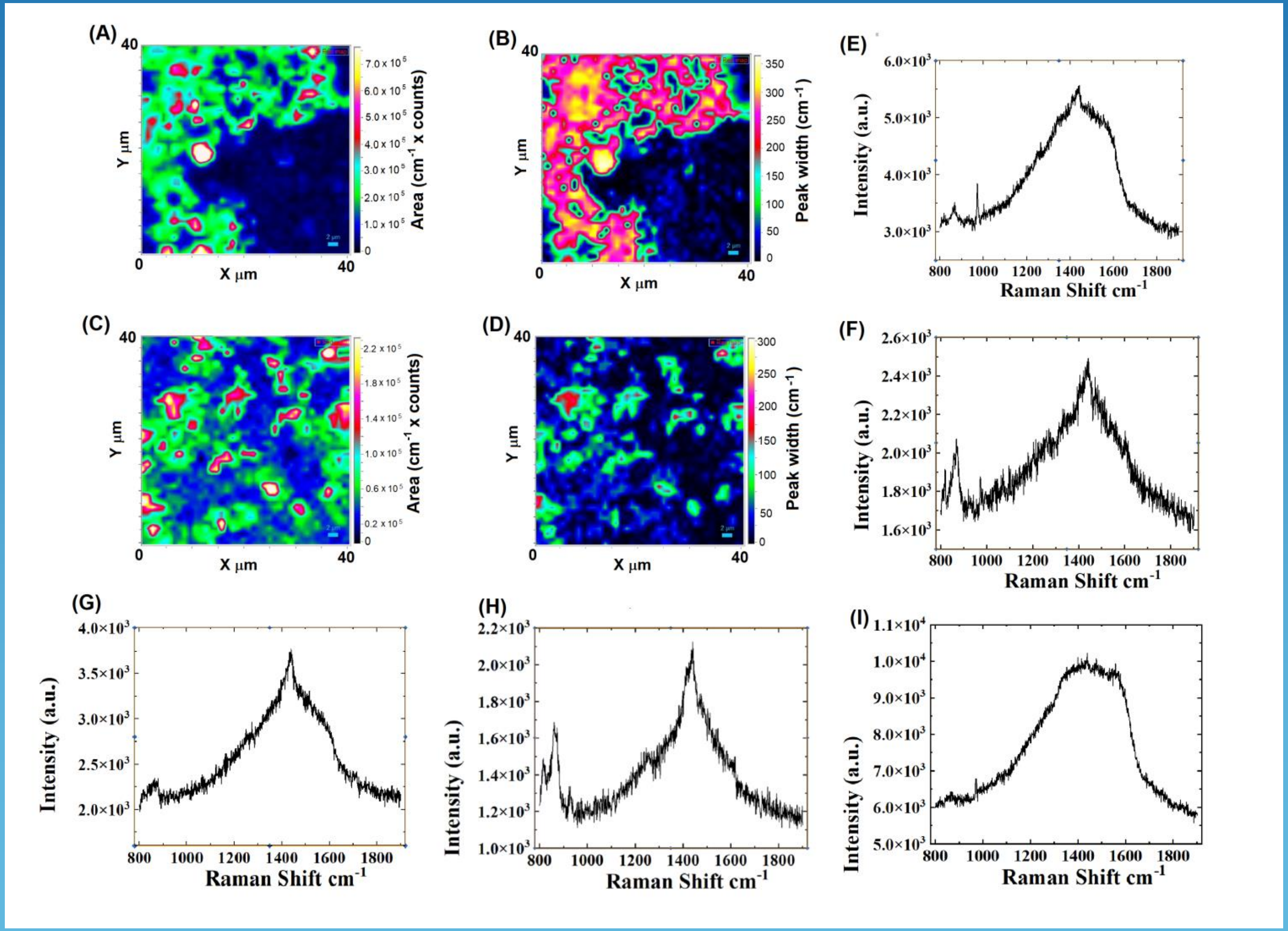
Sulfur-doping 300 degree Celsius = The CFM is fully encapsulated into liquid-sulfur



Manipulating the CFM Properties

(in preparation)

Sulfur-doping 300 degree Celsius, It is noticeable the formation of additional bands ascribed to C-S bonding resulting from sulfur doping



Conclusions

- Perturbed-vapour CVD allows the growth of continuous α-Fe and Fe_3C nanowires.
- In both methods is possible to reach a phase control by employing a quench cooling method.
- The nanowires fill continuously the MWCNT.
- The use of chlorine as a filling-promoter is particularly useful for production of flexible filled CNT-films
- Sulfur instead is found to mostly influence the number of CNT-shells
- When used in large quantities sulfur can allow to significantly manipulate the CNT-shell, with creation of C-S bonds. This latter approach is promising towards possible creation of superconductive buckypapers

Future Work

- We are currently proceeding on the investigation of the low-temperature magnetic properties of these systems

- We will focus on the identification of the critical superconductive temperature and on the optimization of the doping process which will be applied to cm-scale buckypapers

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THANK YOU!