Raman spectroscopy of swift heavy ion–irradiated carbon nanomaterials – from sample preparation to spectrum analysis

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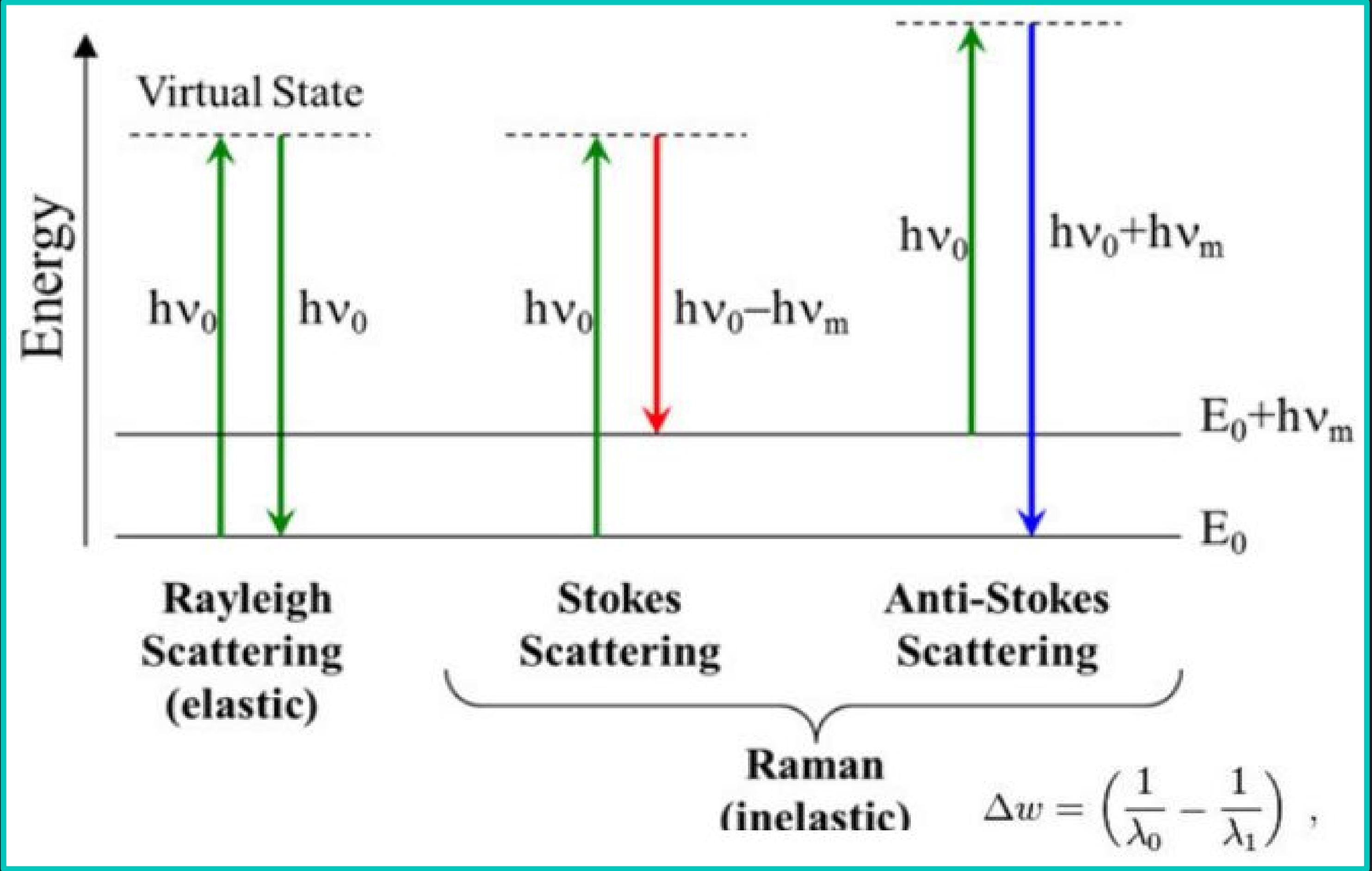
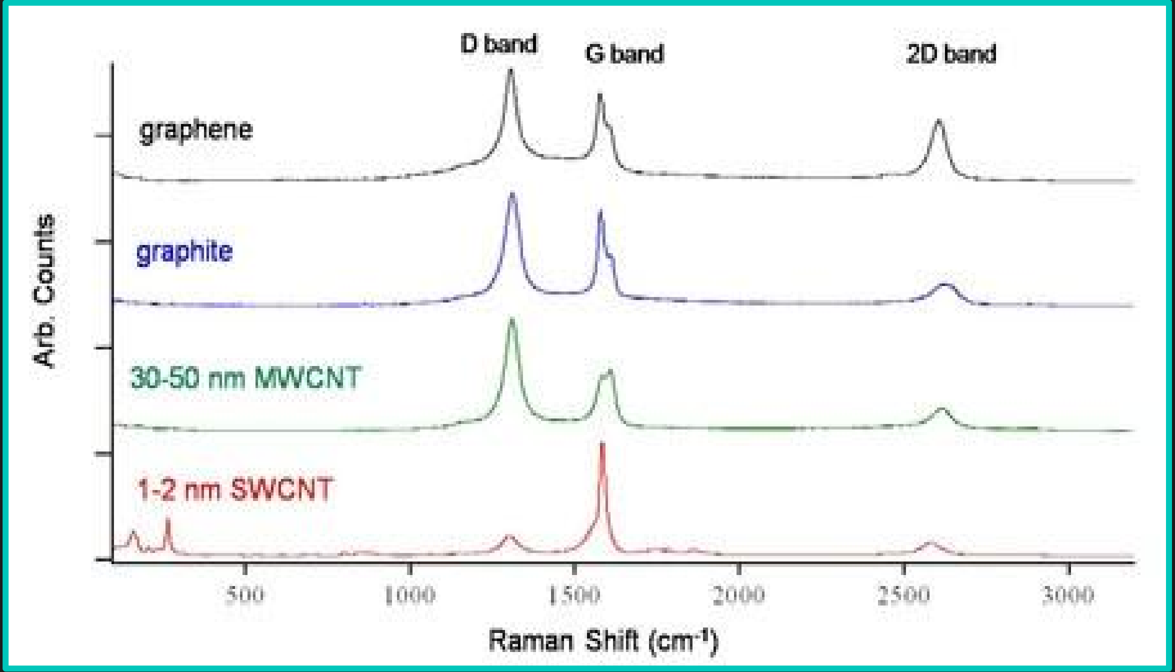
Raman Spectroscopy

Spectroscopic technique used to observe vibrational, rotational, andother low-frequency modes in a system. Its based on inealistic scattering (called also Raman scattering) of monochromatic light, typically from a laser.

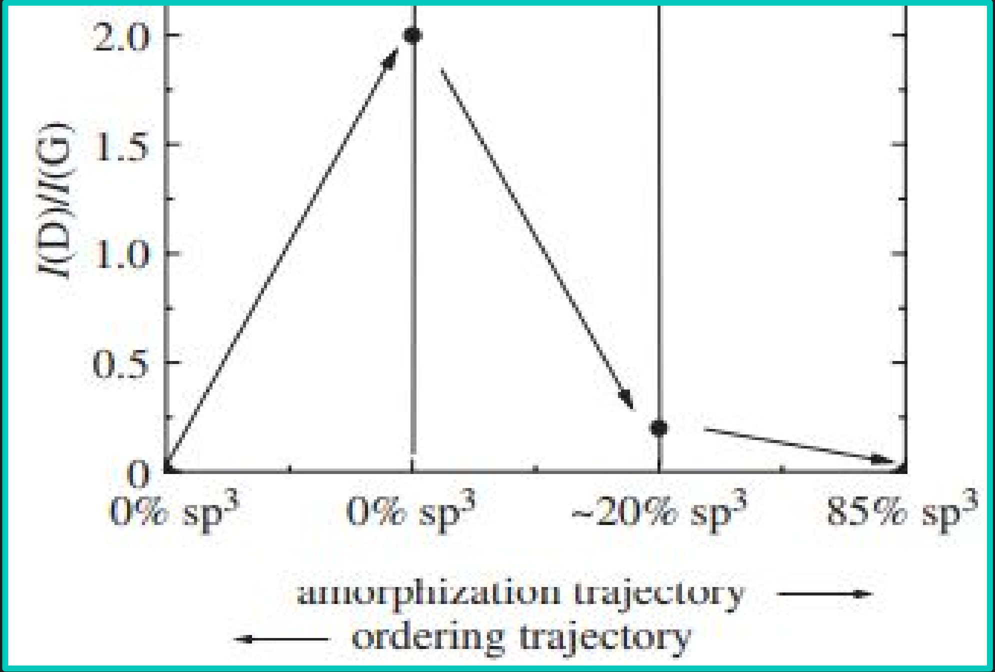


http://bwtek.com/raman-theory-of-raman-scattering/

Raman spectra of carbon materials



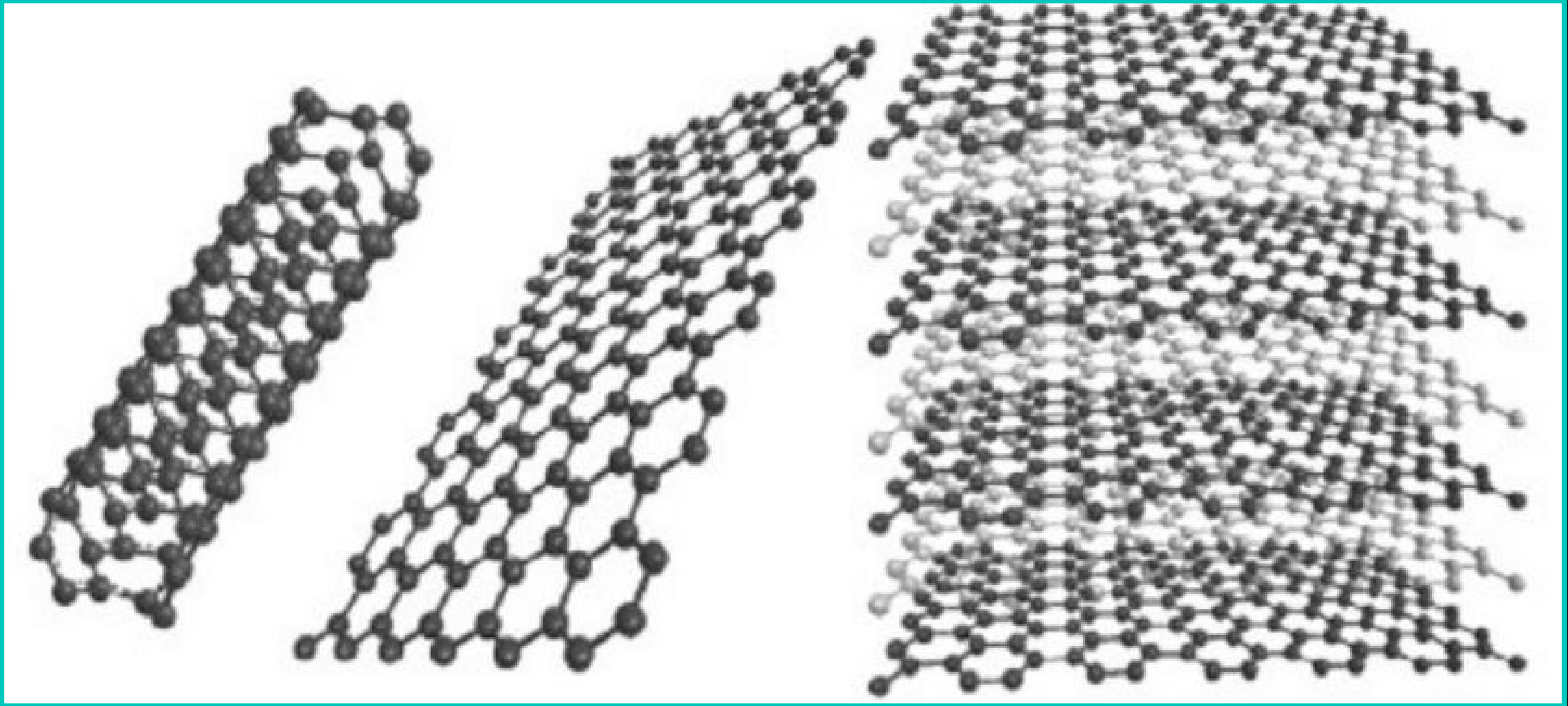
http://www.ceriumlabs.com/104/Q2_2010_Newsletter.htm



http://www-g.eng.cam.ac.uk/nms/publications/pdf/FerrariPTRS2004.pdf

Carbon nanostructures

- Graphene is made of a single layer of carbon atoms that are bonded together in a repeating pattern of hexagons. Graphene is so thin that it is actually considered two dimensional. These single layers of carbon atoms provide the foundation for other important materials.
- Graphite is formed when you stack graphene.
- Carbon nanotubes, which are another emerging material, are made of rolled graphene.



Chao Zhou et al., Graphene's cousin: The present and future of graphane, Nanoscale Research Letters 9(1):26, DOI: 10.1186/1556-276X-9-26

Irradiation of samples – IC100 cyclotron

Raman spectrometer

Table 1: Ions accelerated at IC100

Element	Ion	A/Z	Current, μA
Neon	$^{22}Ne^{4+}$	5,5	0,7
Argon	$^{40}Ar^{7+}$	5,714	2,5
Iron	$^{56}Fe^{10+}$	5,6	0,5
Krypton	$^{86}Kr^{15+}$	5,733	2
Iodine	$^{127}I^{22+}$	5,773	0,25
Xenon	$^{132}Xe^{23+}$	5,739	1,2
Xenon	$^{132}Xe^{24+}$	5,5	0,6
Tungsten	$^{182}W^{32+}$	5,6875	0,015
Tungsten	$^{184}W^{31+}$	5,9355	0,035
Tungsten	$^{184}W^{32+}$	5,75	0,017

Xe – 167 MeV
Kr – 107 MeV

Laser wavelength – 473 nm

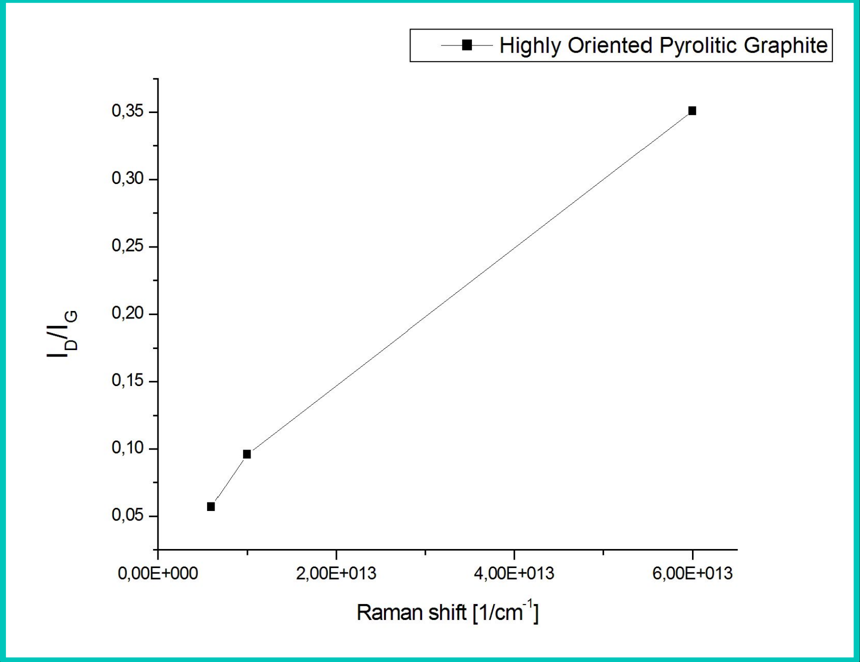
Laser Power – 50 mW

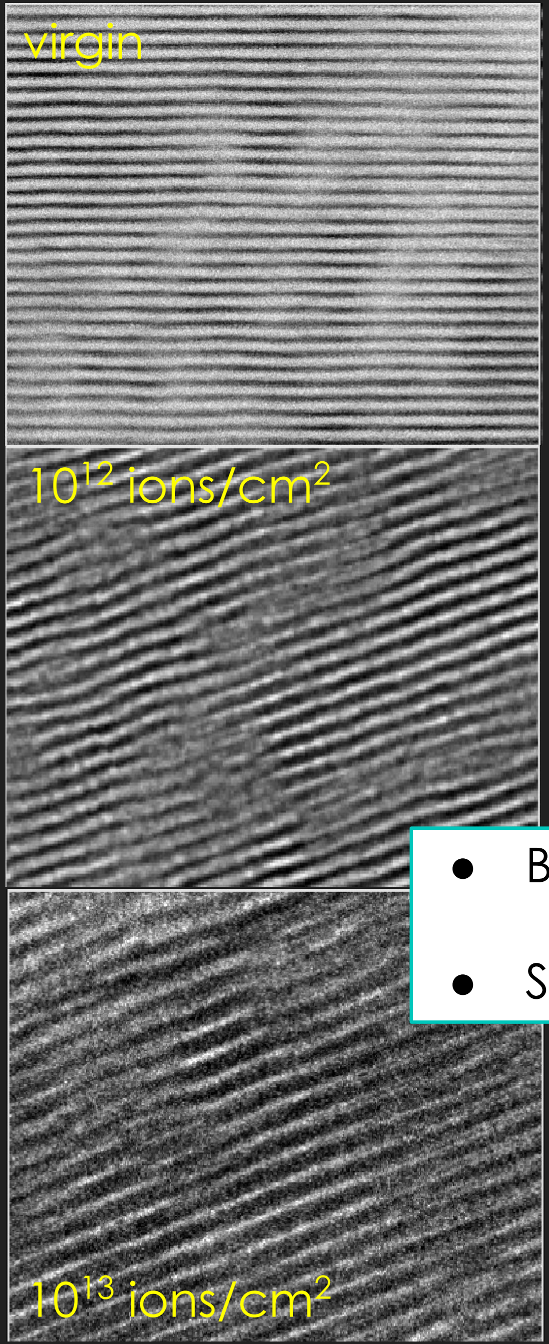
Diffraction grid – 600/600

Central wavelength – 515 nm

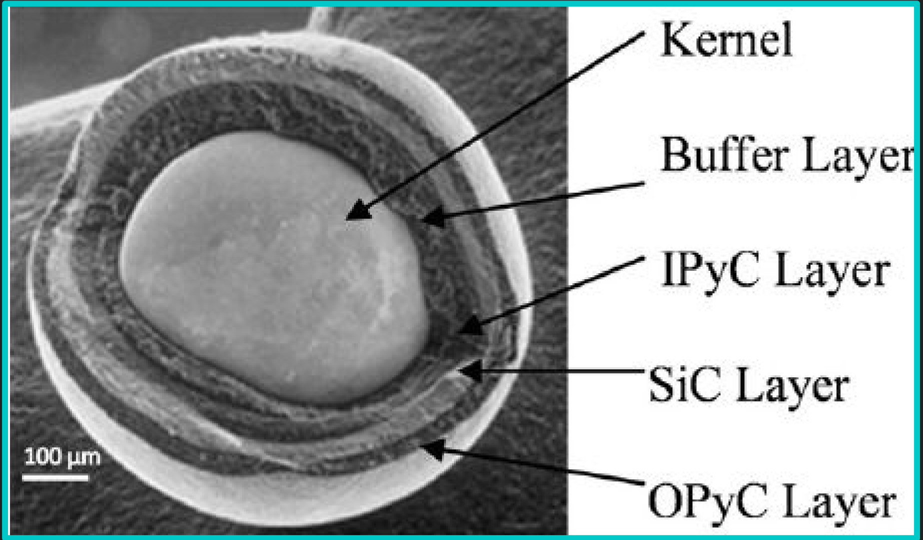
Graphite

Cross-section, electron microscopy images of virgin and Xe Ion-irradiated HOPG

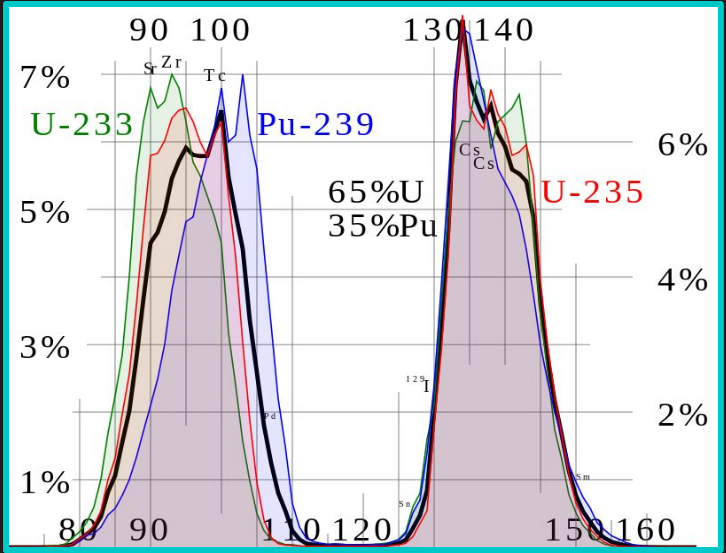




- Ba, Kr
- Sr, Xe



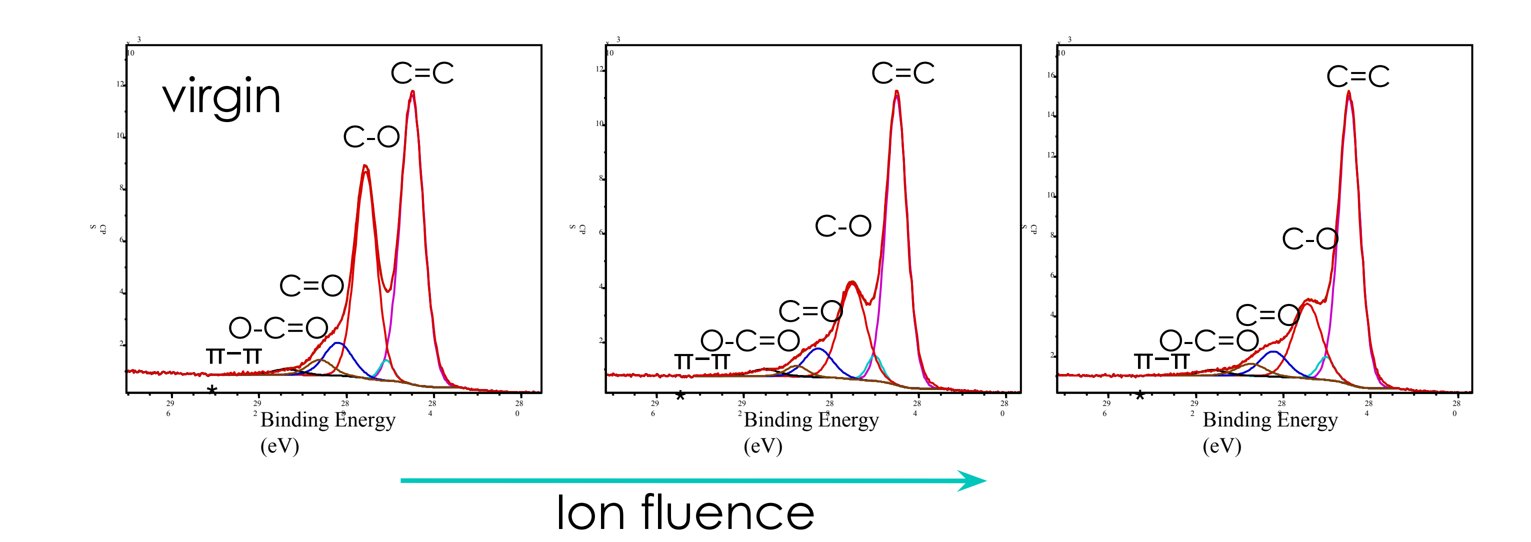
https://www.researchgate.net/figure/251511168_fig1_Fig-1-Cross-section-of-TRISO-fuel-coating-layers-DOE-2002

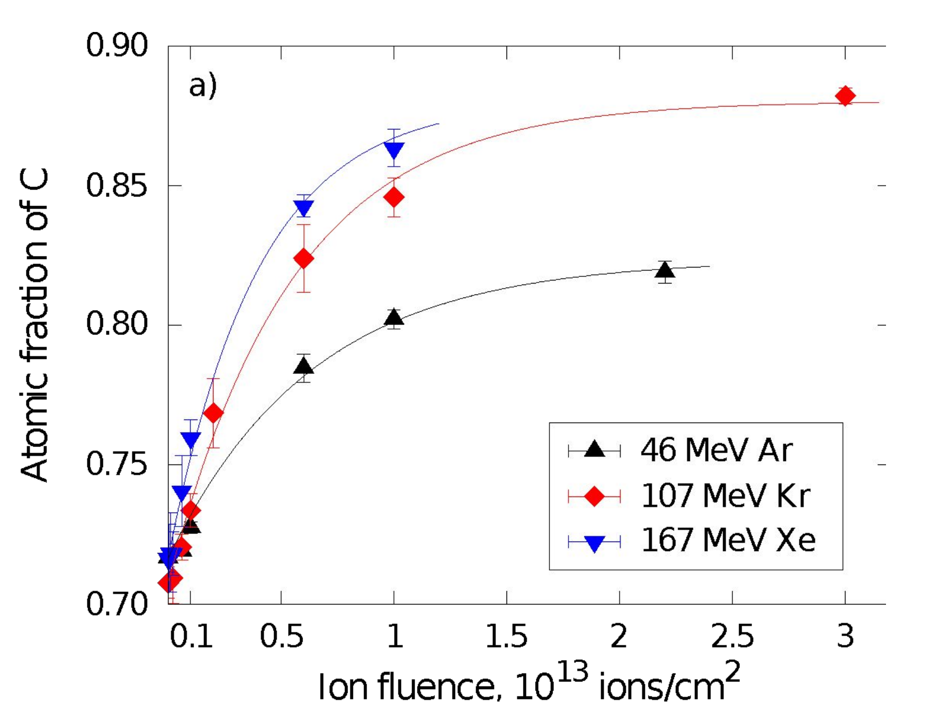


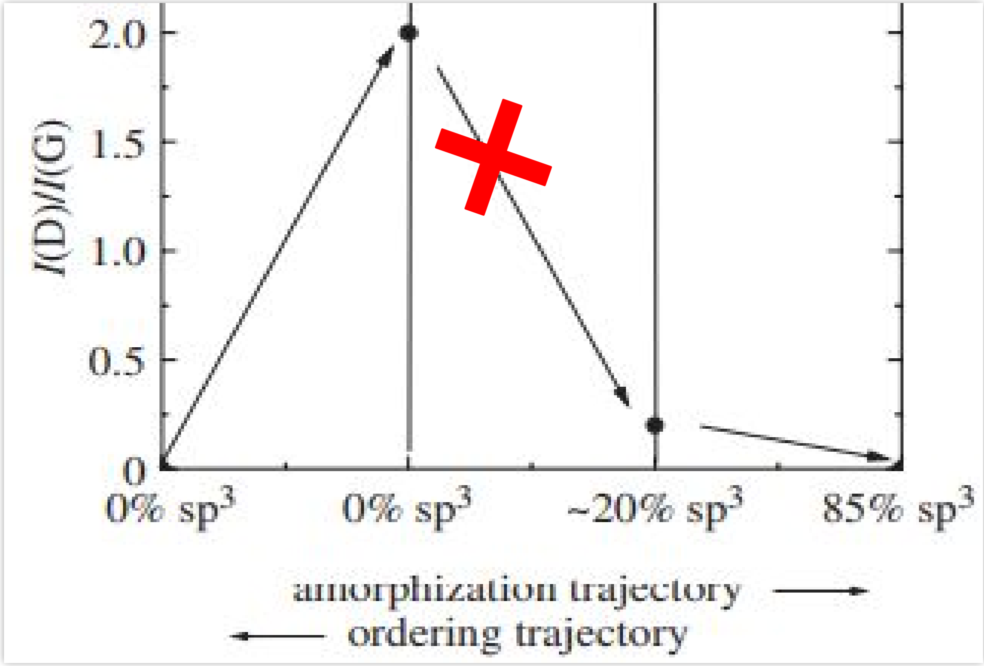
https://en.wikipedia.org/wiki/Fission_products_(by_element)#/media/File:ThermalFissionYield.svg

Graphene oxide

Prepared by the oxidation of graphite using strong oxidizing agents.







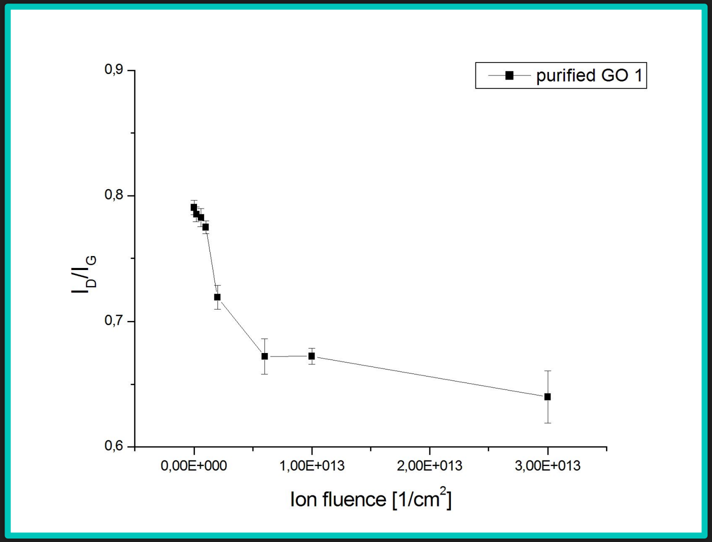
Graphene oxide

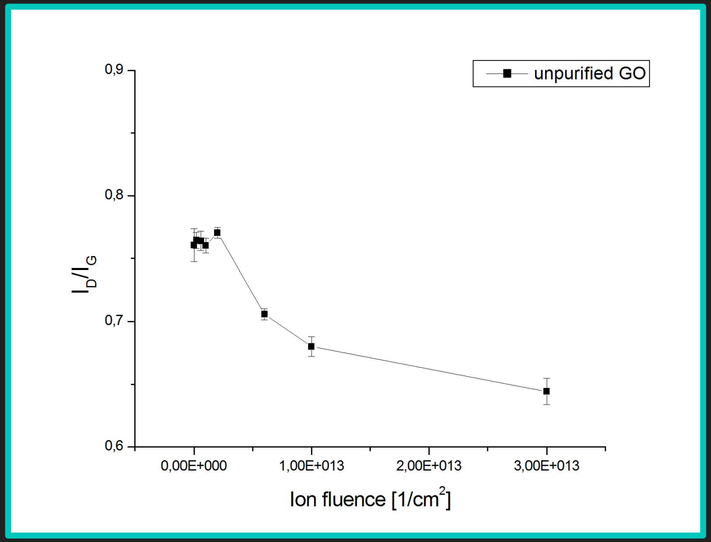
Prepared by the oxidation of graphite using strong oxidizing agents:

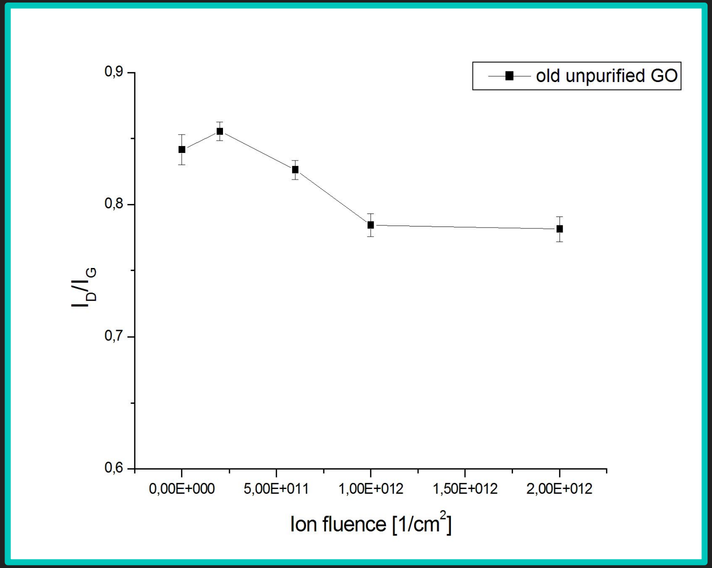
- oxygen - containing functional groups are introduced in the graphite structure
- increase in the layer separation
- the material becomes hydrophilic (can be dispersed in water and other polar solvents).

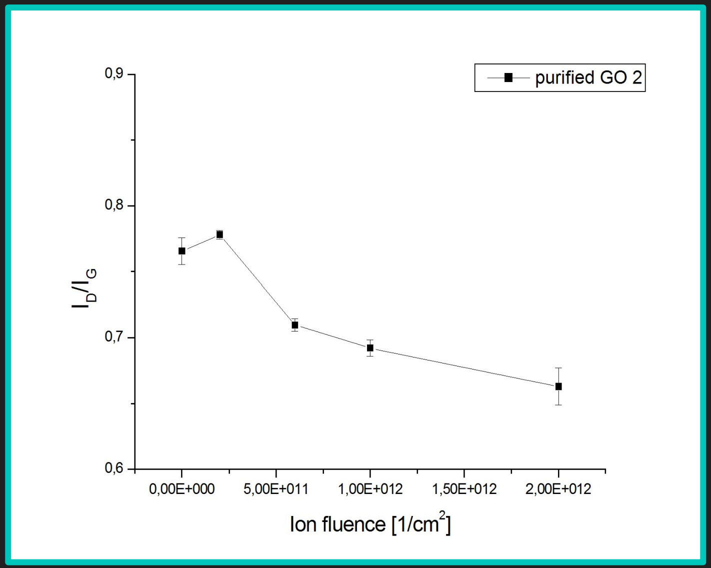
This property allows one to exfoliate the graphite oxide using sonication

- single or few layer flakes, called graphene oxide (GO) can be obtained.



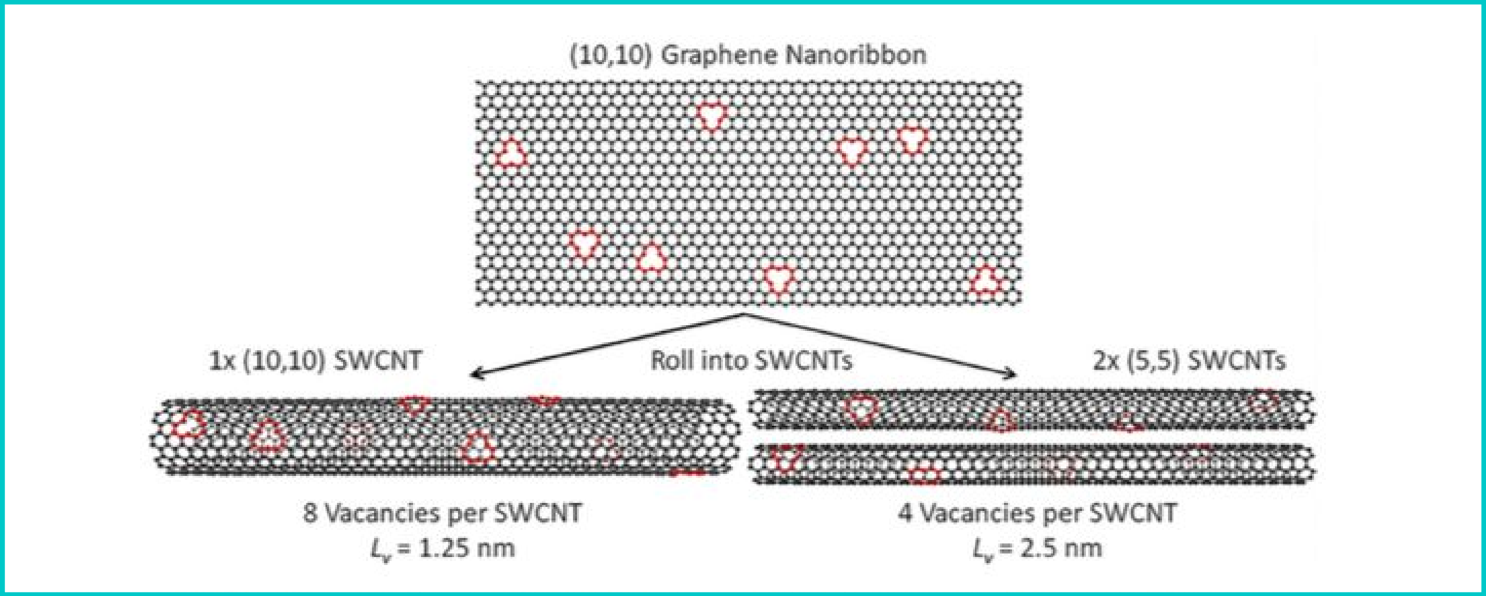


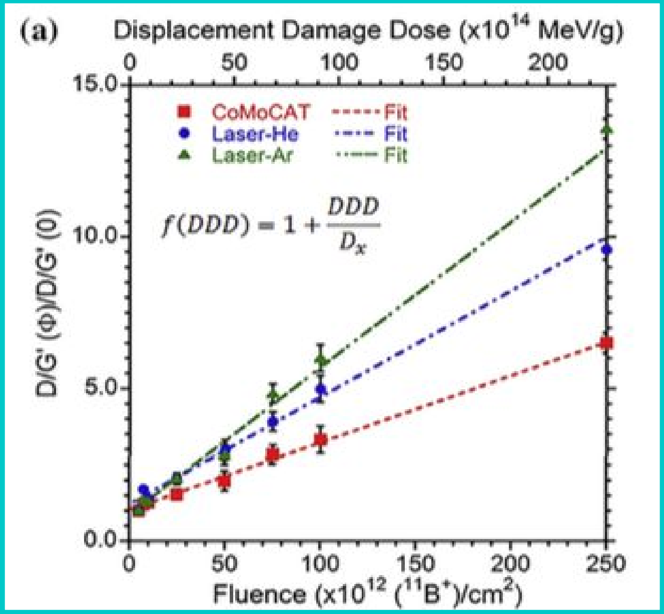


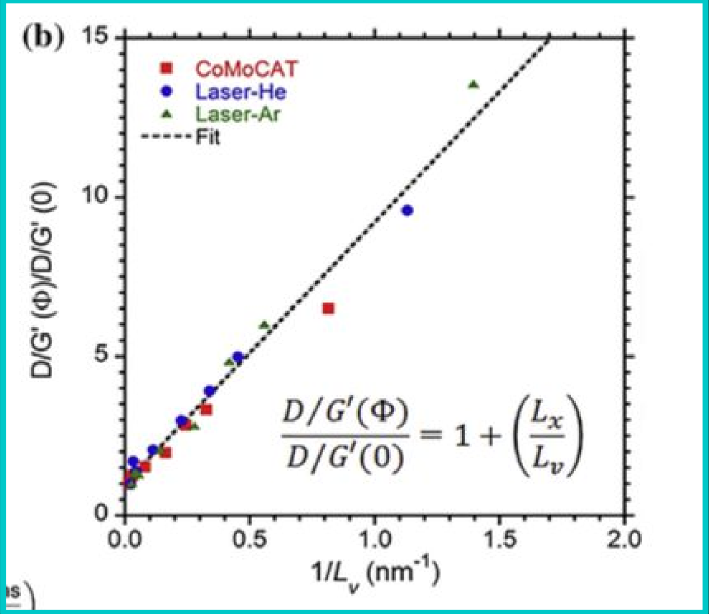


SWNTs - low energy ion irradiation

- Increase in the disorder parameter was more pronounced (by a factor of 2.5) for large-diameter nanotubes than for small-diameter ones.
- Defect density introduced upon irradiation was independent on tube diameter -> the inter-defect length, being inversely proportional to the diameter, was larger for small-diameter tubes, thus causing their lower Raman D-band response (and higher stability) at equivalent doses.





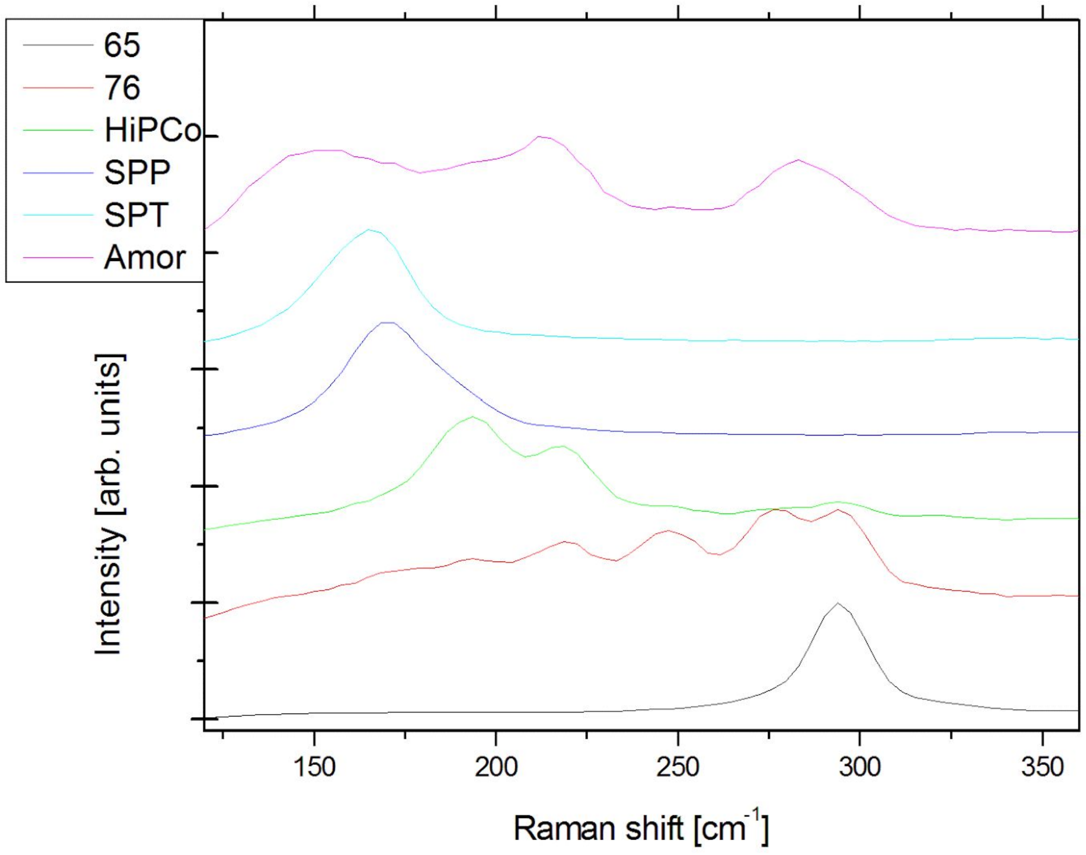


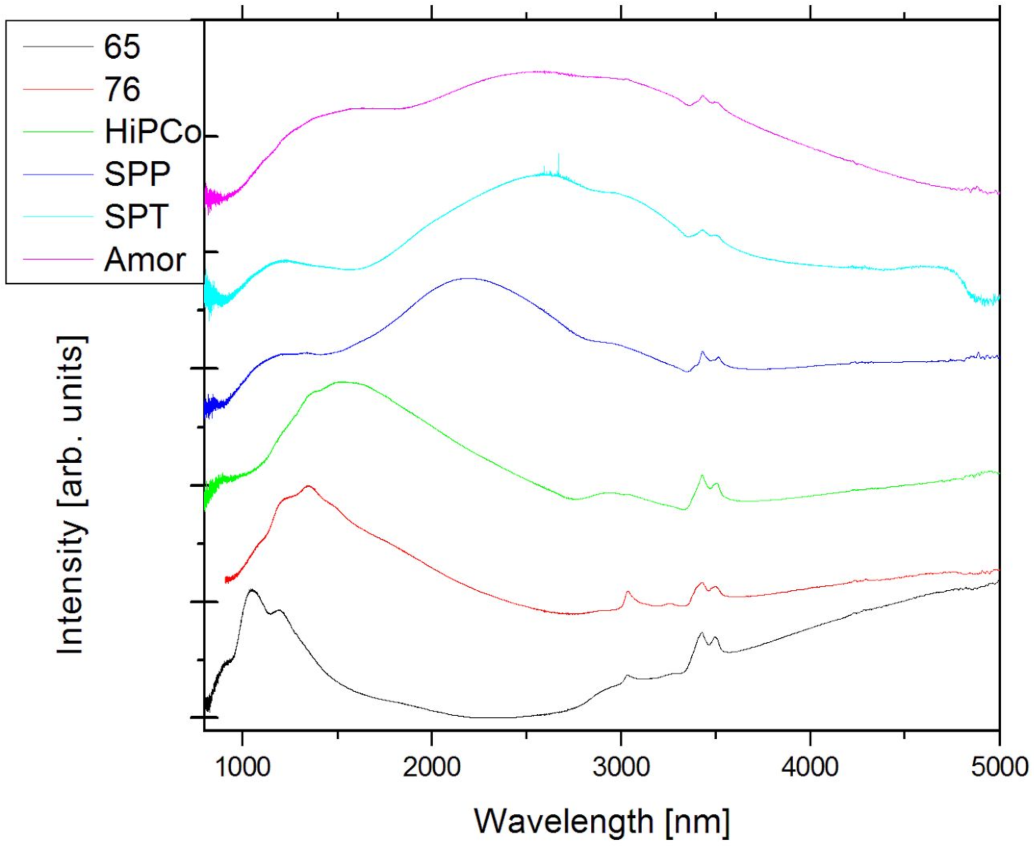
Intrinsic diameter dependent degradation of single-wall carbon nanotubes from ion irradiation,

Jamie E. Rossi, Cory D. Cress, Andrew Merrill, Karen J. Soule, Nathanael D. Cox, Brian J. Landi

The diameter distribution of our SWNT samples: Raman and NIR-MIR analysis

DIAMETER INCREASE

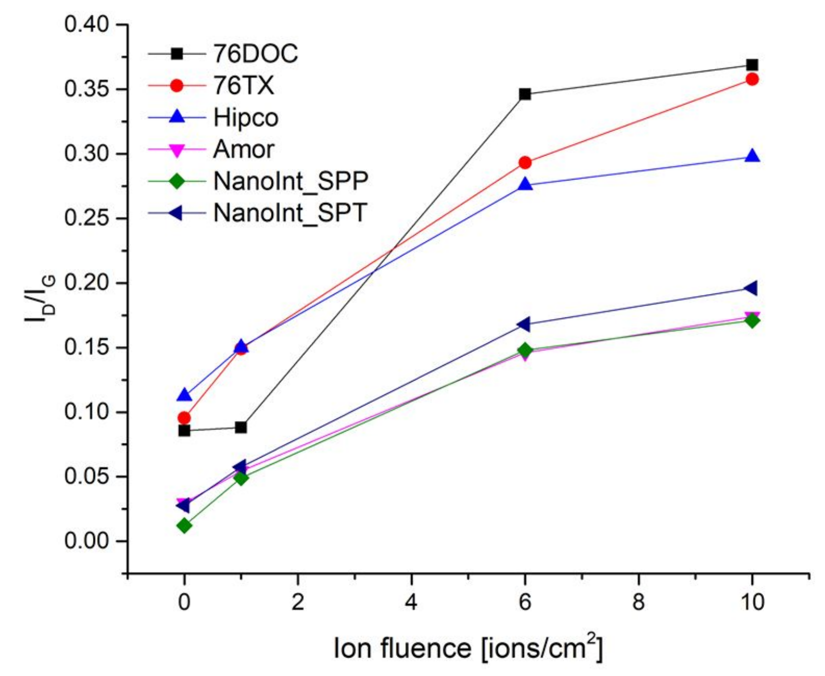


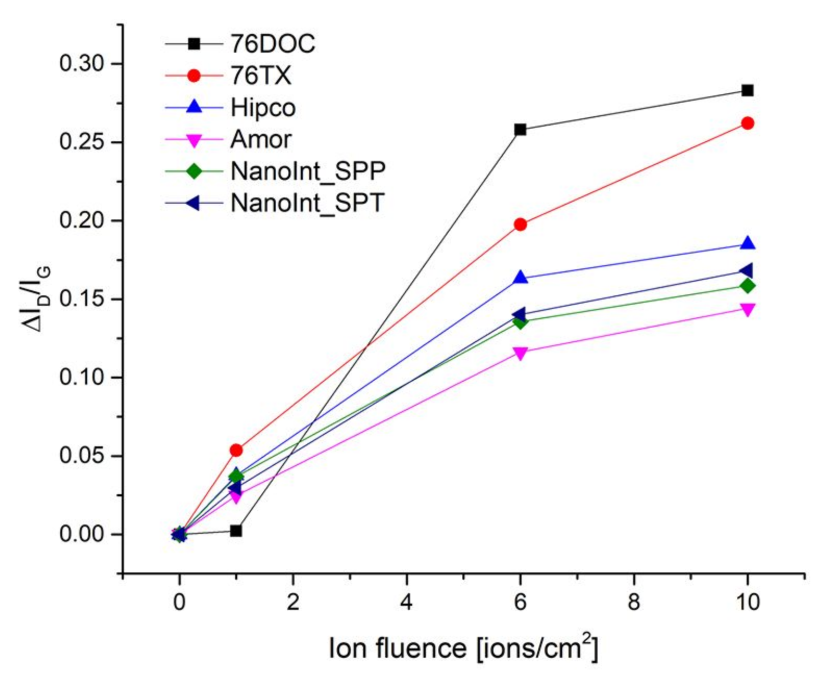


Experimental Raman spectra in the RBM region.

Absorption spectra of various SWNTs specimens in the near-infrared (NIR) to mid-infrared (MID) wavelength range.

Results of our measurements: SWNTs – swift heavy ion irradiation





In conclusion

- Raman spectroscopy is a useful technique to investigate heavy ion irradiation damage
- The irradiation of graphite in the cyclotron and Raman spectroscopy of the samples can be helpful to investigate the damages similar to nuclear reactor conditions
- Generally, nanotubes with bigger diameter are more resistant to swift heavy ion irradiation
- During the irradiation of a nanotube using high energy ions beyond the knockout of the atom a large amount of energy will be transferred which heats up the sample & increases the damage

Thank you for your attention