Characterization techniques applied to Carbon Nanowires

Team 3
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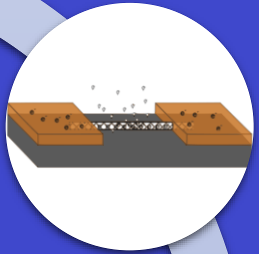
Thursday, 04 June 2020

Agenda

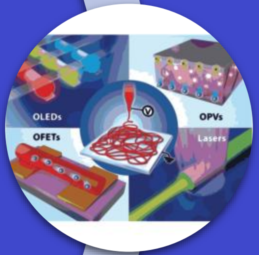
- Project Overview, goals & justification : <2min
- Characterization Techniques : ~10min
    - Rheometry
    - Scanning Electron Microscopy (SEM)
    - Raman Spectroscopy
    - 4-Wire Kelvin
- Conclusions : <1min
- Q&A : <4min

Justification:

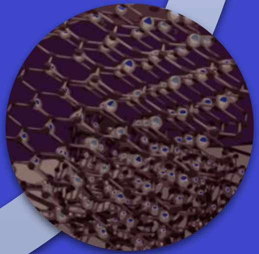
CNWs' Applications



Sensors

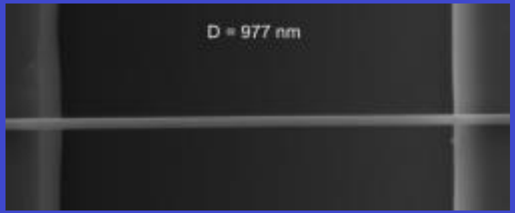


Electronic & Optical Devices



Energy Generation & Storage

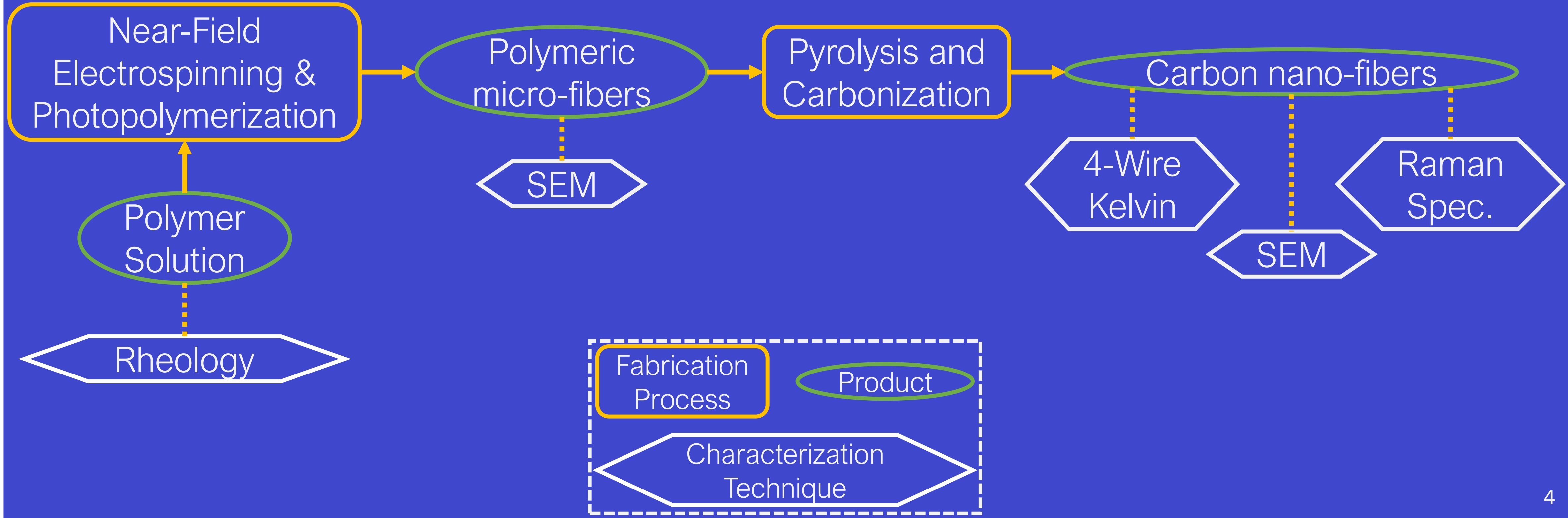
CNWs morphology & conductivity



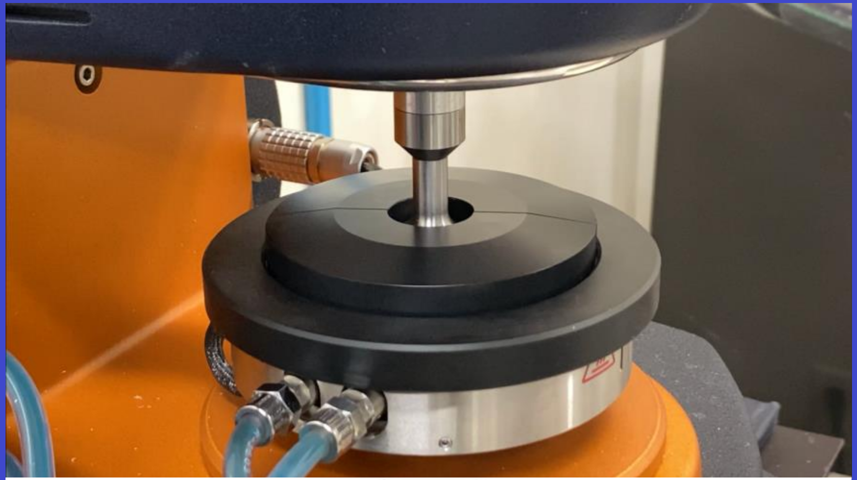
J. Jang, J. Bae, Carbon nanofiber/polypyrrole nanocable as toxic gas sensor, Sensors Actuators B Chem. 122 (2007) 7–13. https://doi.org/10.1016/j.snb.2006.05.002.
X. Shi, W. Zhou, D. Ma, Q. Ma, D. Bridges, Y. Ma, A. Hu, Electrospinning of Nanofibers and Their Applications for Energy Devices, J. Nanomater. 2015 (2015) 1–20. https://doi.org/10.1155/2015/140716.
C. Monterrey, B. Cárdenas Benítez, INSTITUTO TECNOLOGICO Y DE ESTUDIOS SUPERIORES DE MONTERREY Advanced Manufacturing Techniques for the Fabrication and Surface Modification of Carbon Nanowires, (2017) 160.

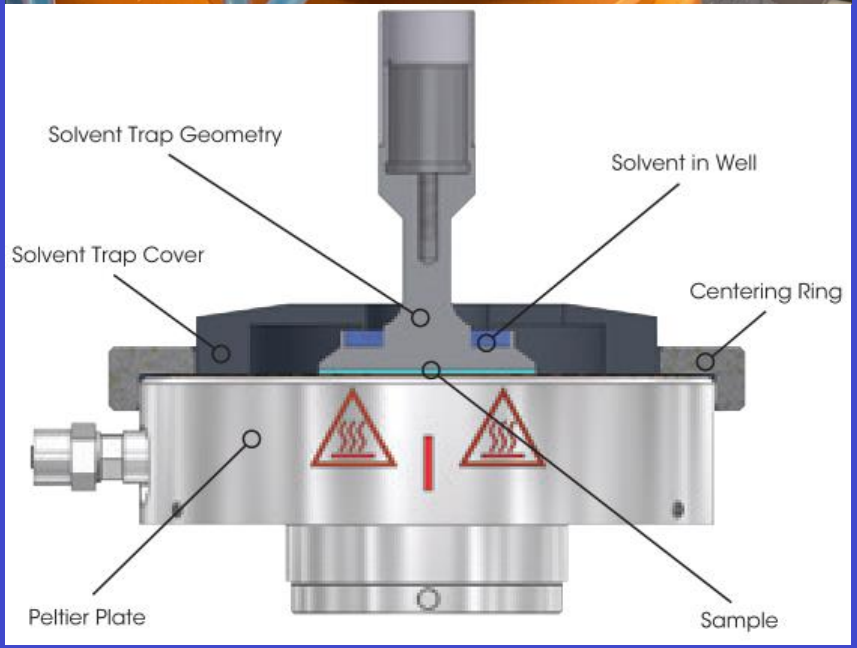
Introduction: Project overview

To design polymer solutions that can be **electrospun** by NFES, **photopolymerized**, and then **pyrolyzed** into conductive carbon nano-wires.



Charc. Techniques: Rheology (instrumentation)





Measured Parameter	Calculated Parameter
Torque	Stress
Angular displacement	Strain
Angular velocity	Shear rate

$$Stress = Torque[\mathrm{N \cdot m}] \cdot Stress\ constant$$

$$Strain = Angular\ displacement[\mathrm{rad}] \cdot Strain\ constant$$

$$Shear\ rate = Angular\ velocity\left[\frac{\mathrm{rad}}{\mathrm{s}}\right] \cdot Strain\ constant$$

$$Viscosity = \frac{Stress}{Shear\ rate}$$

$$Young's\ Modulus = \frac{Stress}{Strain}$$

Discovery Hybrid Rheometers – TA Instruments, (n.d.). https://www.tainstruments.com/products/rheology/discovery-hybrid-rheometers/ (accessed 18 May 2020).
Malvern Instruments Worldwide, Malvern Instruments White Paper - A Basic Introduction to Rheology Shear Flow, (2016) 1–20. https://www.malvernpanalytical.com/en/learn/knowledge-center/whitepapers/WP160620BasicIntroRheology.html.

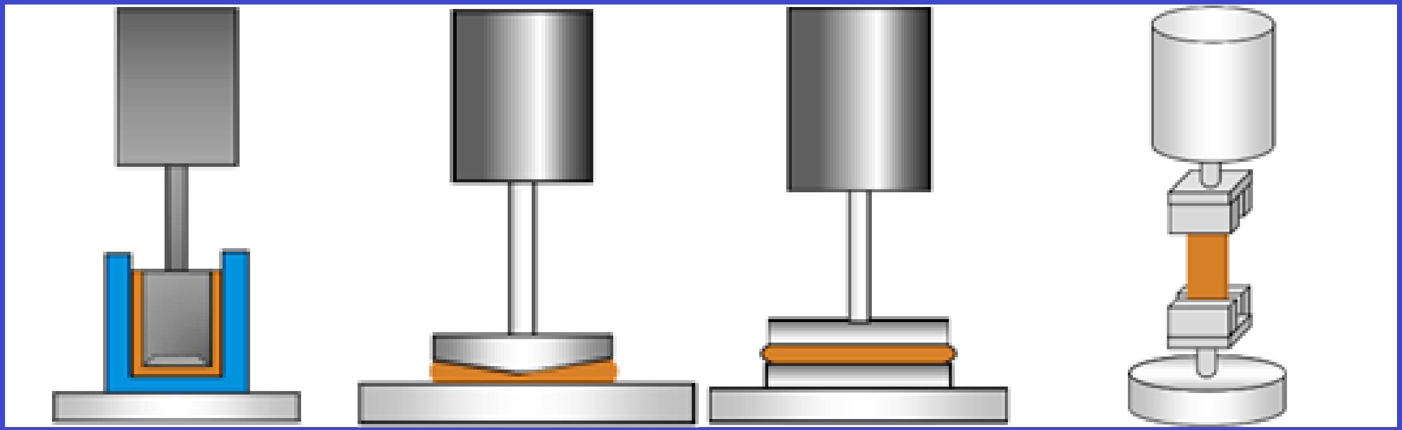
Charc. Techniques: Rheology (process parameters)

Important rheometer parameters:

- Torque Range
- Angular Resolution
- Angular Velocity Range
- Frequency Range

Geometry options:

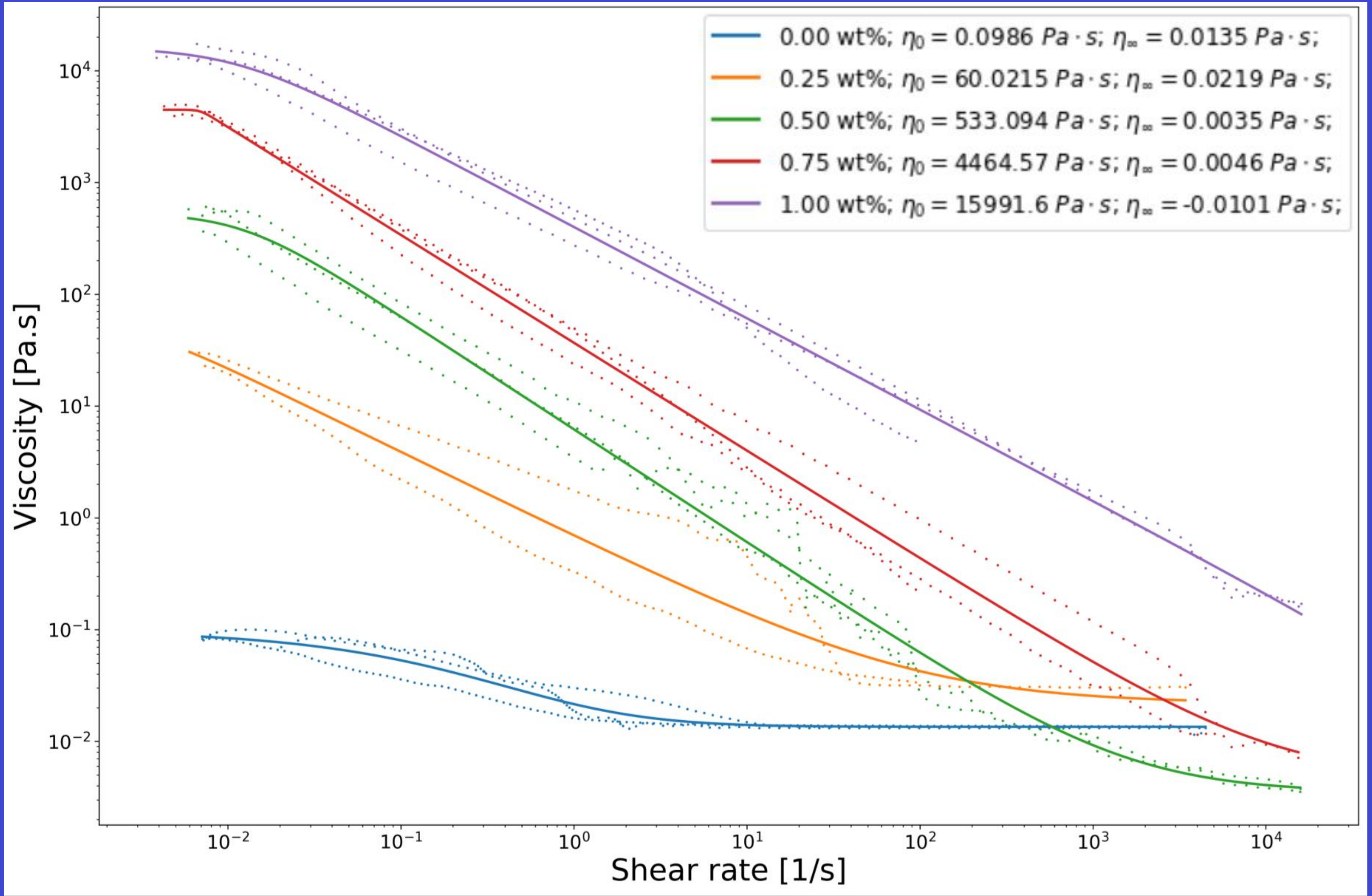
Concentric Cylinders | Cone and Plate | Parallel Plate | Torsion Rectangular



Very Low Viscosity → Solids

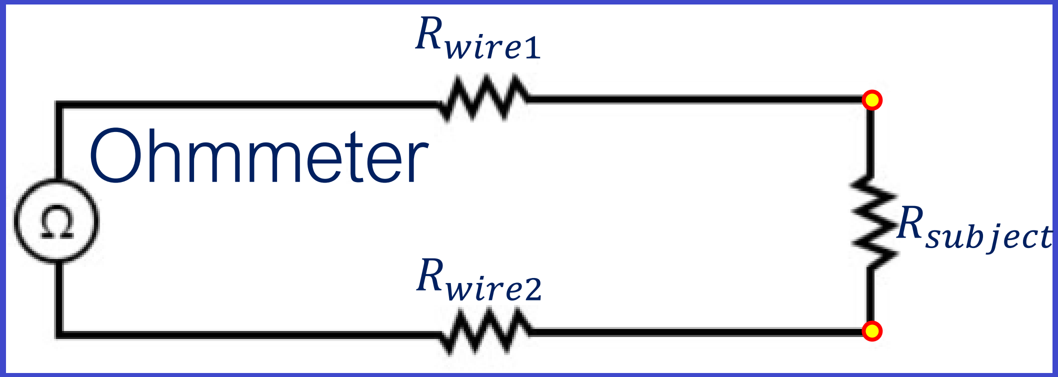
Discovery Hybrid Rheometers – TA Instruments, (n.d.). https://www.tainstruments.com/products/rheology/discovery-hybrid-rheometers/ (accessed 18 May 2020).

Charc. Techniques: Rheometry (Example results)



Charc. Techniques: 4-Wire Kelvin

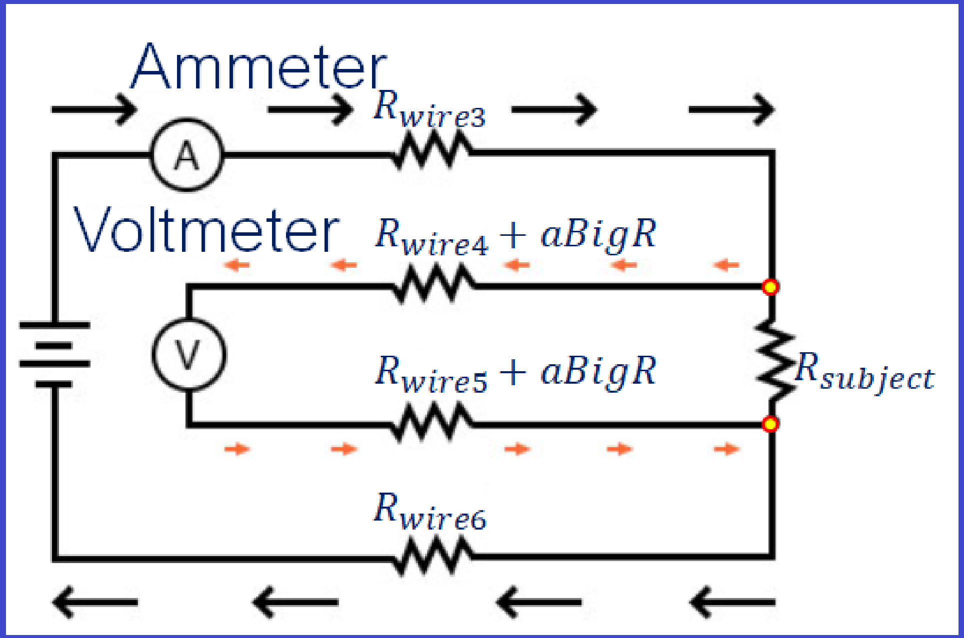
Typical method:



$$Ohmmeter_{indication} = R_{wire1} + R_{subject} + R_{wire2}$$

If the sample has a very low resistance, the measurement error introduced by wire resistance will be substantial.

Kelvin method:



$$R_{subject} = \frac{Voltmeter_{indication}}{Ammeter_{indication}}$$

Electrical conductivity:

$$\sigma = \frac{L}{R_{subject} \cdot A} \left[\frac{S}{m}\right]$$

Keithley Instruments Inc, Series 2600B System SourceMeter ® Instrument Reference Manual, (2019) 1–946.

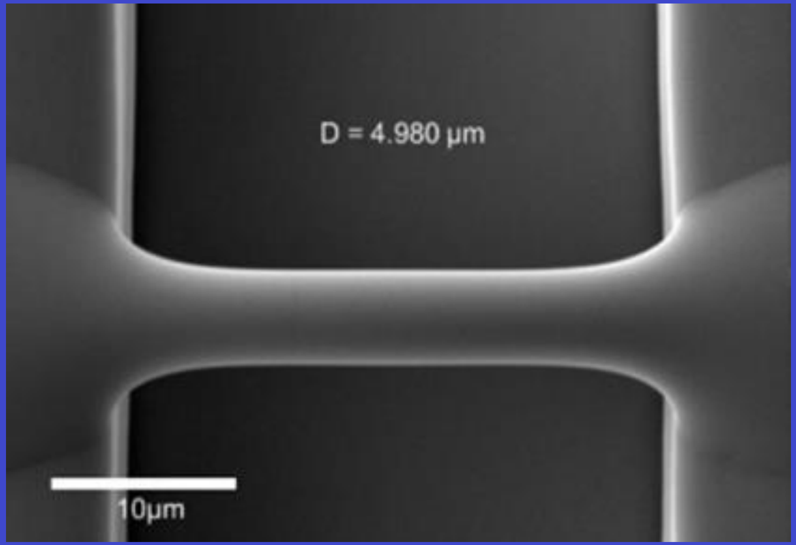
Charc. Techniques: Scanning Electron Microscopy (SEM)

A beam of electrons scans across the material surface, producing high-energy back-scattered electrons and low-energy secondary electrons. The back-scattered electrons provides a physical image of the sample surface.

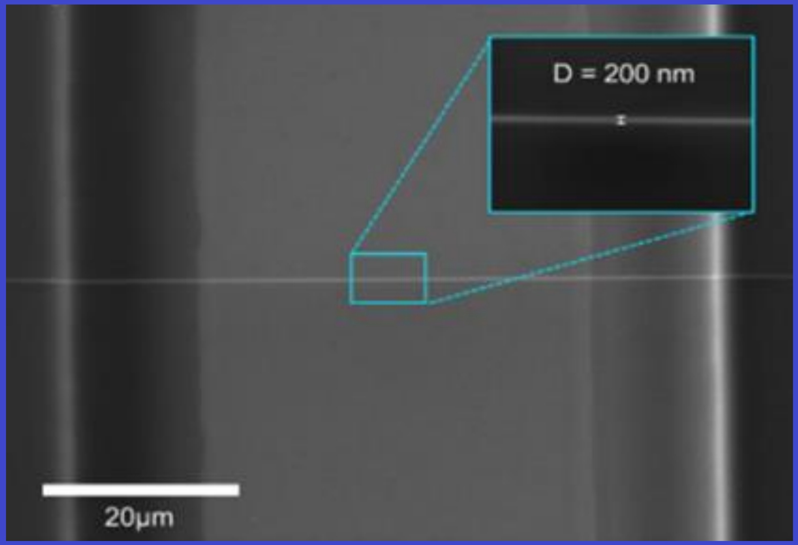
Sample preparation:

After assesing conductivity of carbon nanowires, metal coating is only applied when they don't present conductivity

Fiber diameter before pyrolysis



Fiber diameter after pyrolysis



B. Cárdenas, Advanced Manufacturing Techniques for the Fabrication and Surface Modification of Carbon Nanowires, (2017) 160.

Charc. Techniques: Raman Spectroscopy

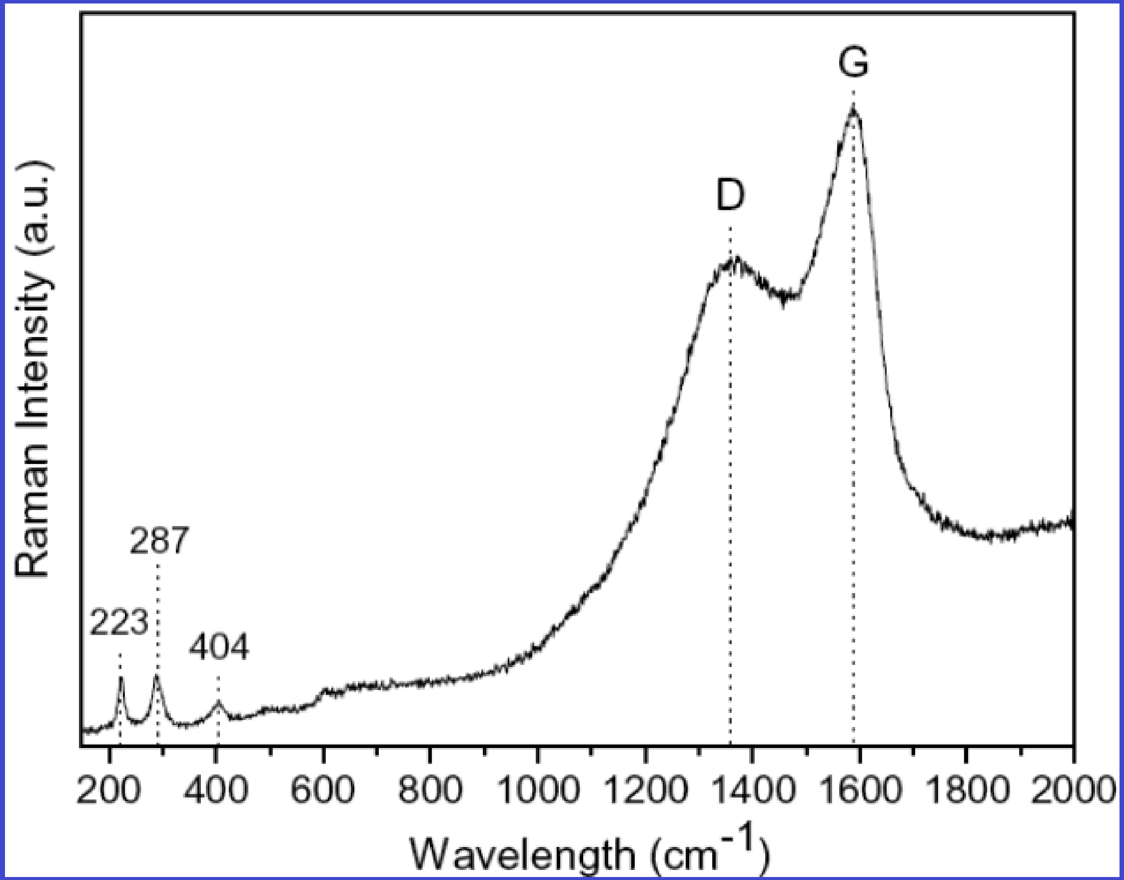
Collect knowledge about molecular information and crystalline structures. A laser irradiate the sample with monochromatic radiation, with excitations in the UV, visible and near-IR spectral region (785-1064nm). The irradiation generates a Raman spectrum of scattered light.

Sample preparation:

The pyrolized sample is placed in a quartz or glass vessel and then analyzed, no more sample preparation is needed for Raman.

Charc. Techniques: Raman Spectroscopy

Expectation is to find two bands (marked D and G in the figure), centered about 1360 and 1594 cm^{-1} to confirm the presence of graphitic carbon in the nanofibers.



C. Eid, A. Brioude, V. Salles, J.-C. Plenet, R. Asmar, Y. Monteil, R. Khoury, A. Khoury, P. Miele, Iron-based 1D nanostructures by electrospinning process, Nanotechnology. 21 (2010) 125701. https://doi.org/10.1088/0957-4484/21/12/125701.

Conclusions:

- The presented techniques provide enough data to satisfy the project goal.

Technique	Data obtained
Rheology	Zero-shear viscosity
4-Wire Kelvin	Electrical resistivity
SEM	Fiber morphology
Raman	Presence of graphitic carbon

- The Rheology ensures electrospunable solutions
- TGA may provide useful information, but is omitted due to time and availability constraints.

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