TEAM 2
EXPERIMENTAL CHARACTERIZATION OF MATERIALS

Synthesis and Characterization of Nanofiber Electrodes with Carbon Nanotubes

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Introduction

Supercapacitors are important elements in the development of technology and in the market; therefore, it is important to develop new materials to make them more efficient.

Carbon nanotubes are excellent alternatives due to their high conductivity and surface area. For this, methods must be developed to characterize their electrical properties under specific conditions.

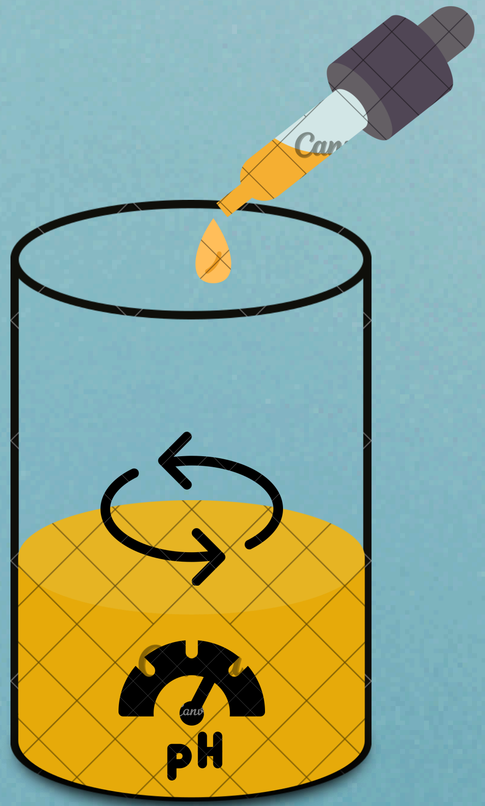
Objectives

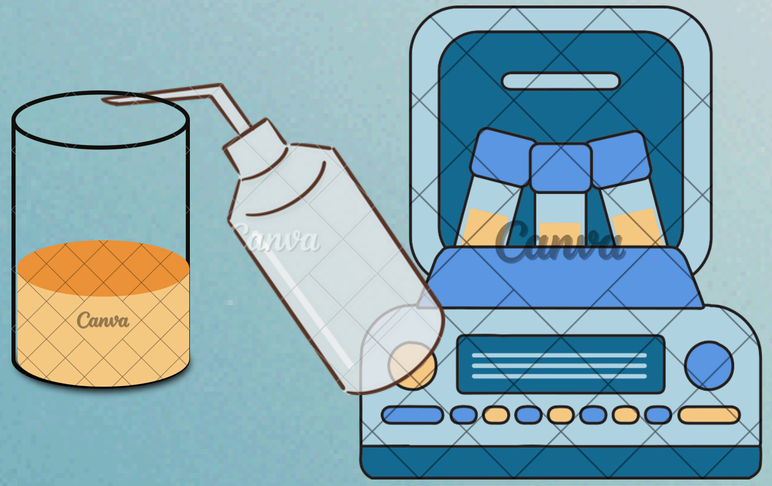
- **Experimental:** To synthesize iron nanoparticles and polymer fiber mats in order to characterize carbon nanotubes using advanced techniques, with the aim of understanding the structural and electrical properties of these materials.

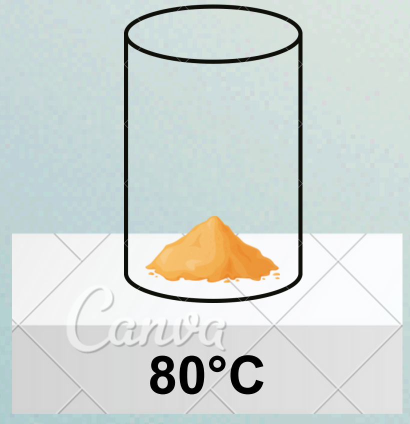
- **Engineering Design:** To design a stainless-steel chamber in order to investigate the electrical transport properties of the samples under variations in the atmosphere.

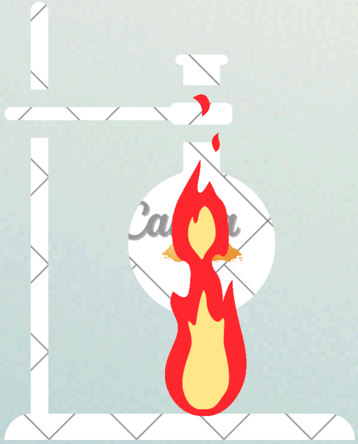
SYNTHESIS OF THE SAMPLES

Nanoparticles synthesis









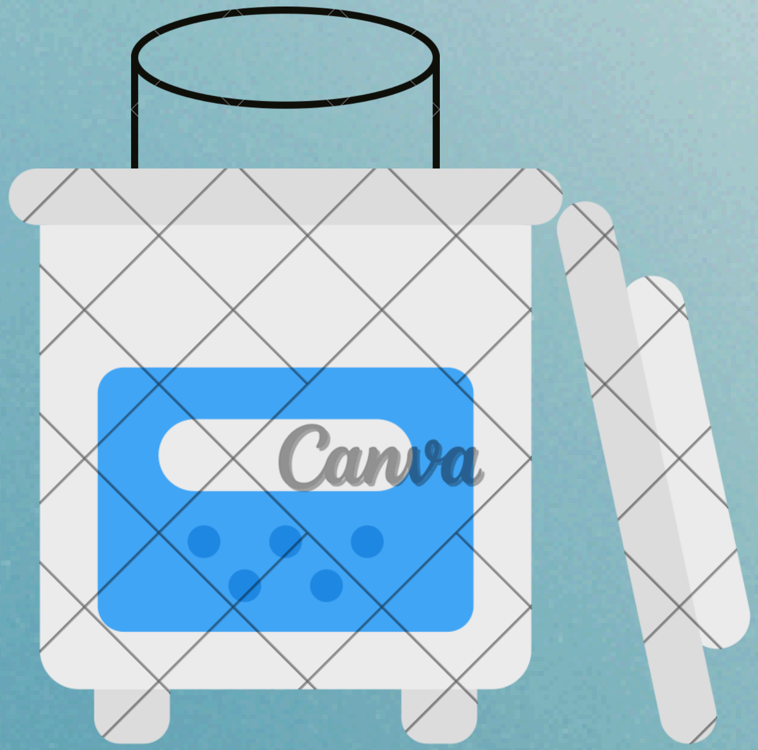
FeCl3+Distilled water
Mixed 30 minutes

NH4OH
PH=11
Mixed 3 hours

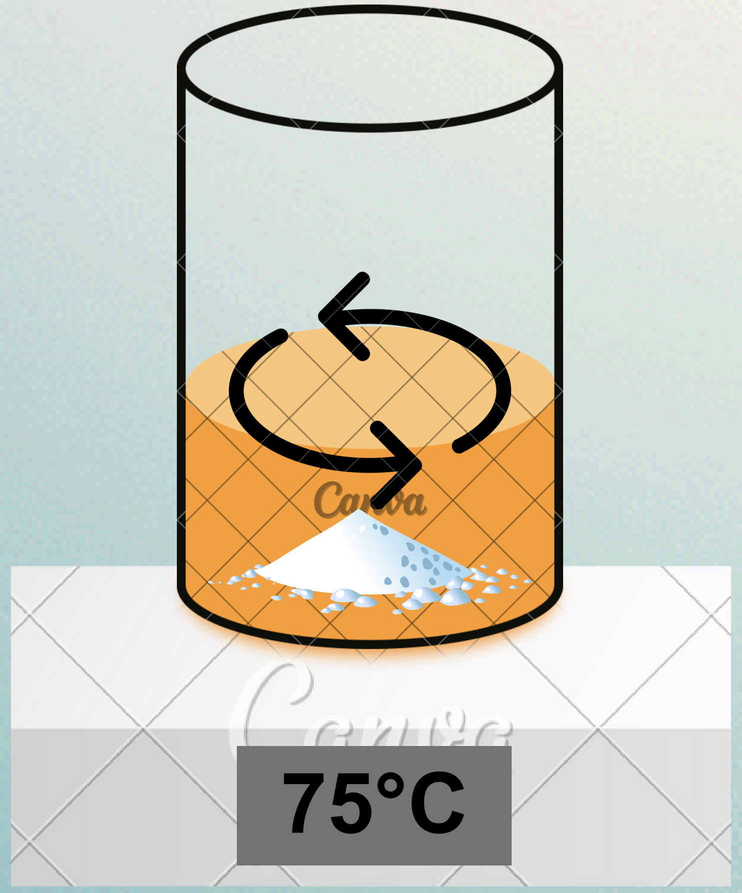
Washed with C_2H_6O y H2O
Centrifuged
Dried at temperature
Calcined

Preparation of the polymer solution

4ml DMF
0.38 g FeNP



20 min



0.38 g PAN
12 hurs spin

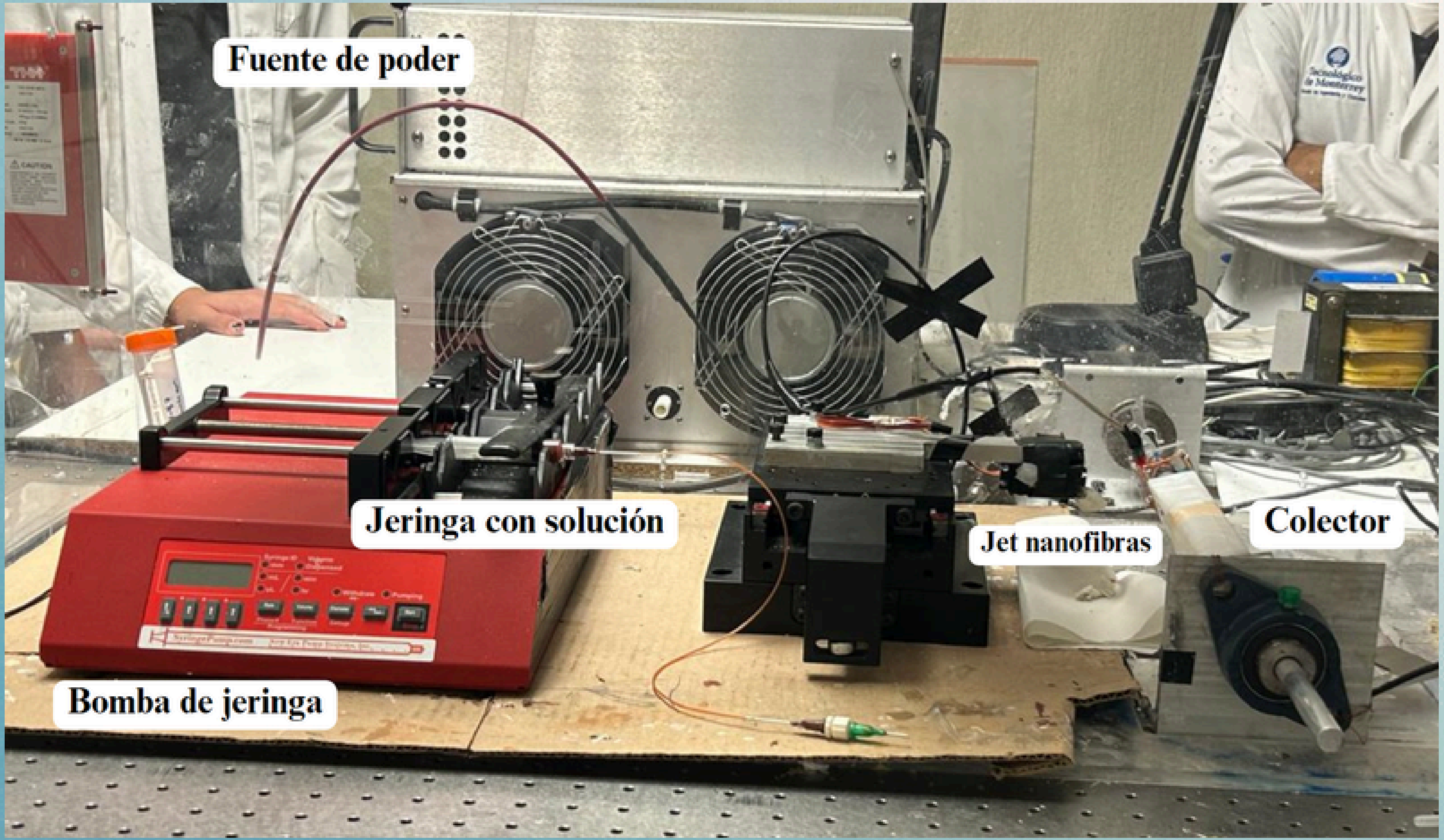
Electrospinning

Technique based on electrostatic interactions.

Important parameters:

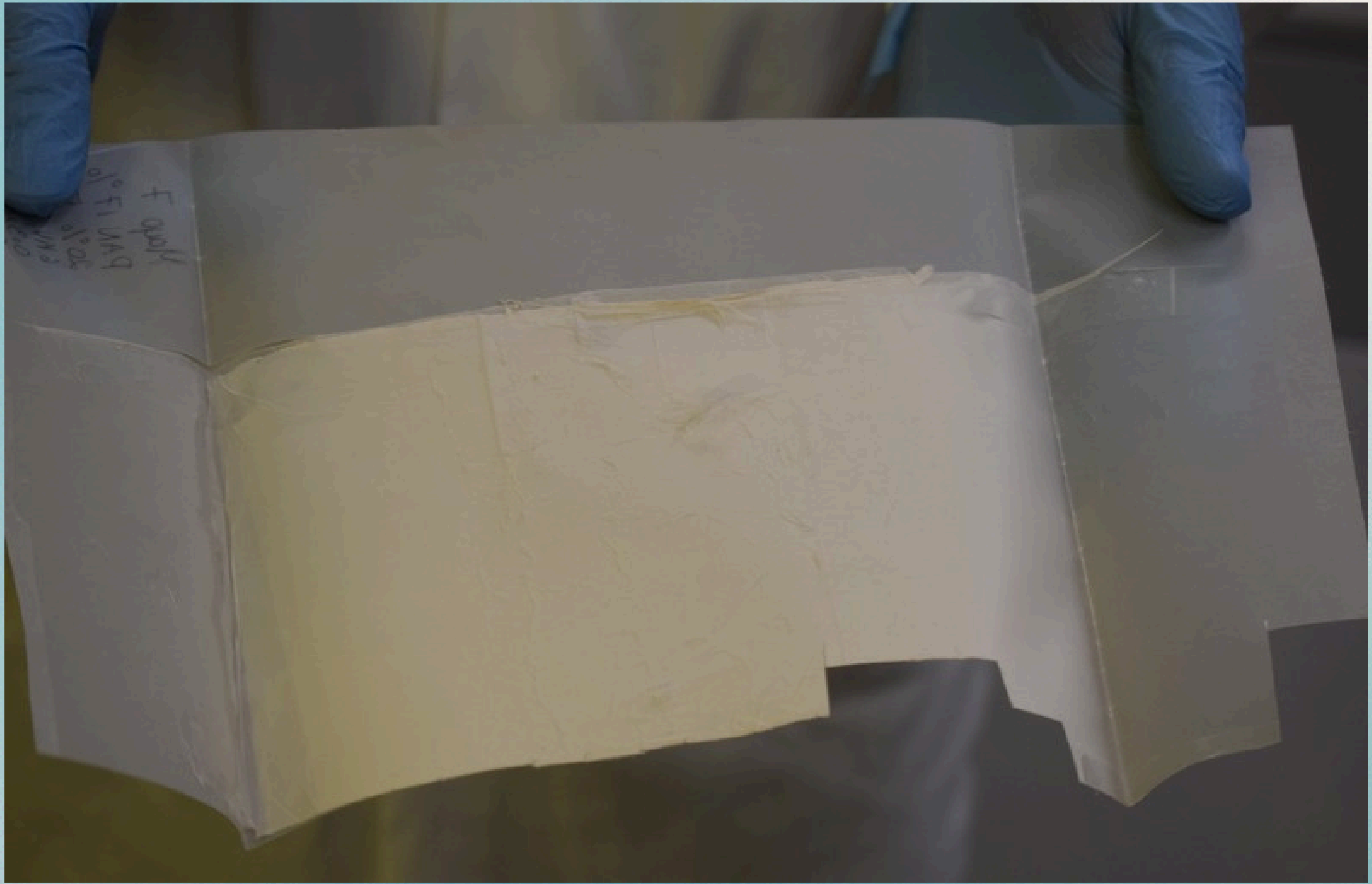
- Constant flux of 1.9 μL/min.
- High Voltage Source 7.2 kV
- Distancia aguja-colector 4 cm
- Collector: 50 rps

A positive charge is induced on the capillary and the collector is grounded.Experimental setup.



Experimental setup

A mat of polymer fibers with iron nanoparticles is obtained.



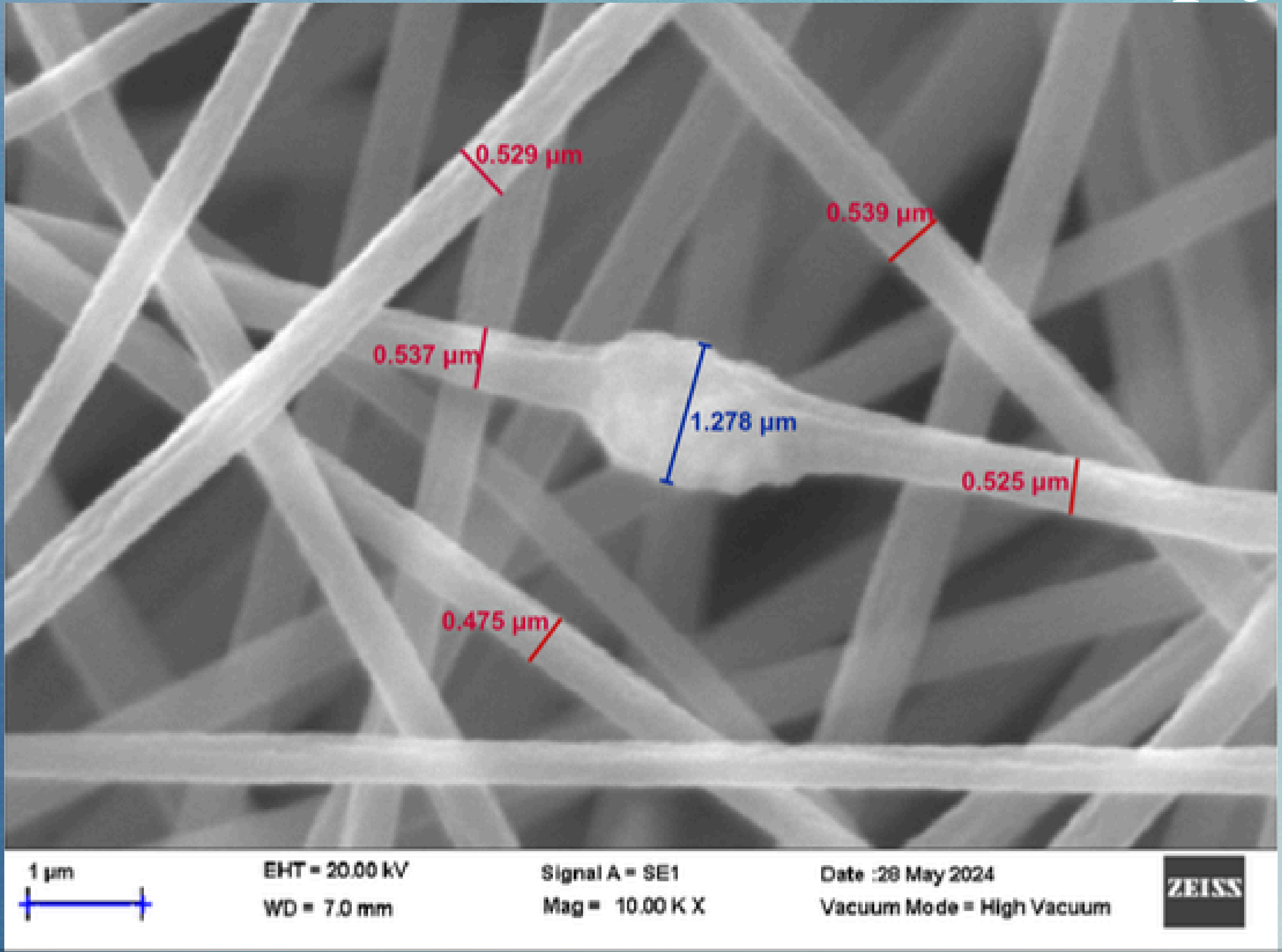
Pyrolysis and CVD

- Precursor: Camphor ($C_{10}H_{16}O$)
- Inert atmosphere of nitrogen
- Catalyst: Fe_2O_3 nanoparticles
- Substrate: PAN polymer fibers
- 90 minutes to reach 900 °C
- 60 minutes of pyrolysis
- 3 hours of cooling

CHARACTERIZATION

Scanning Electron Microscopy (SEM)

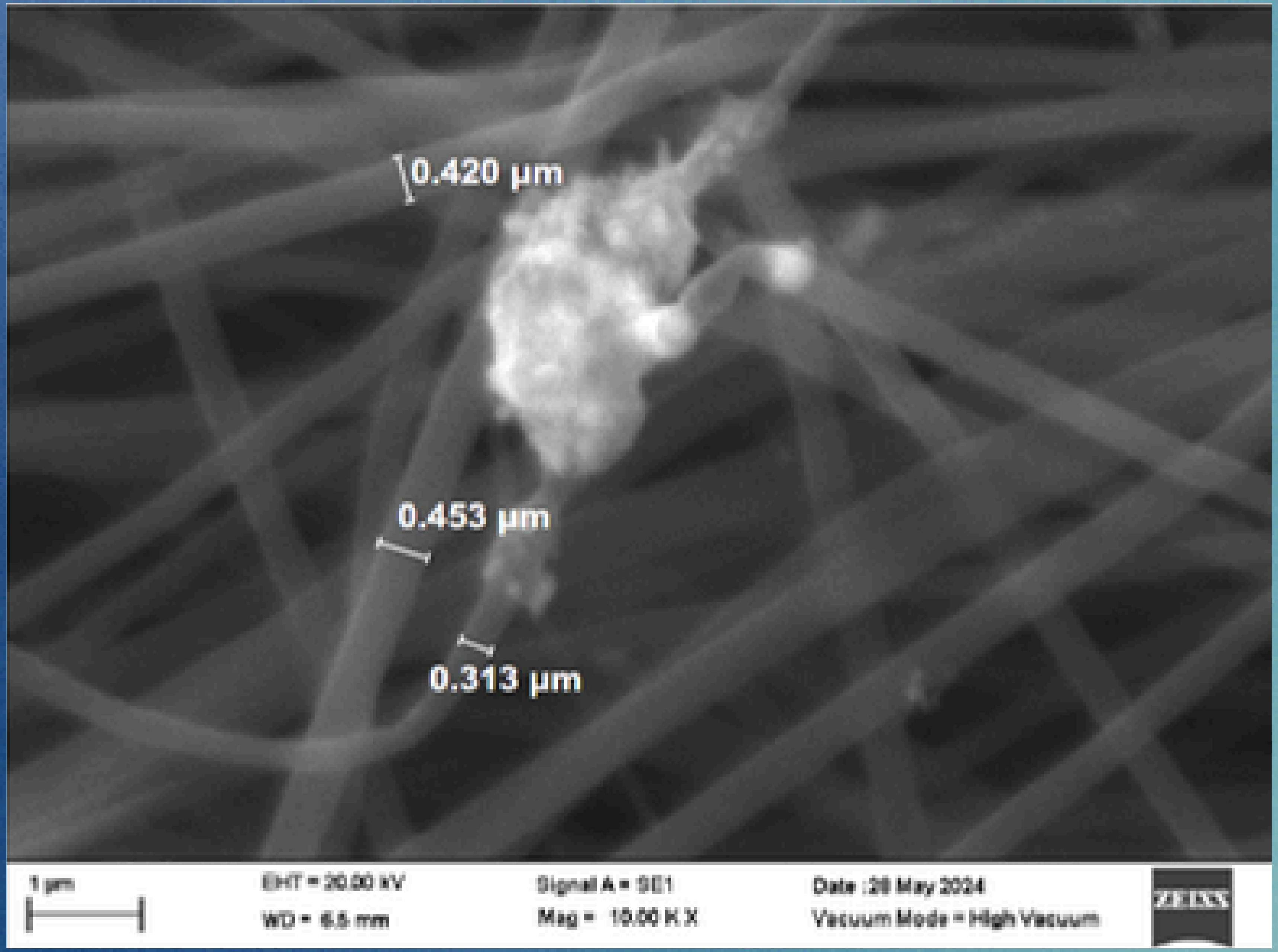
PAN fibers with FeNPs without pyrolysis in SEM:



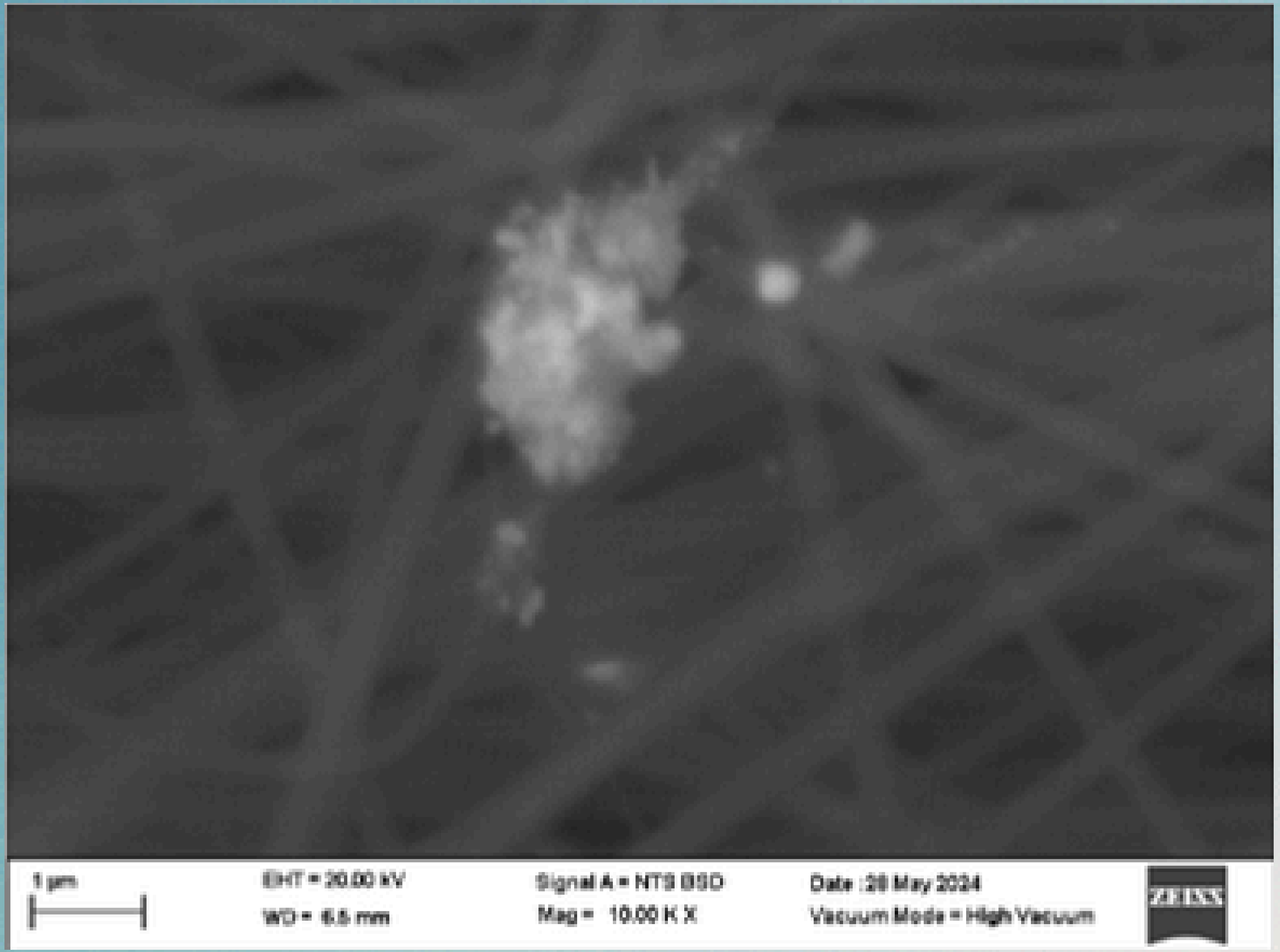
Scanning electron microscopy (SEM) images of PAN fibers with Fe nanoparticles (without pyrolysis) were obtained using the secondary electron signal. The operating conditions were: EHT = 20.00 kV, WD = 7.00 mm, and magnification ×10,000.

PAN fibers with FeNPs after pyrolysis in SEM:

a)



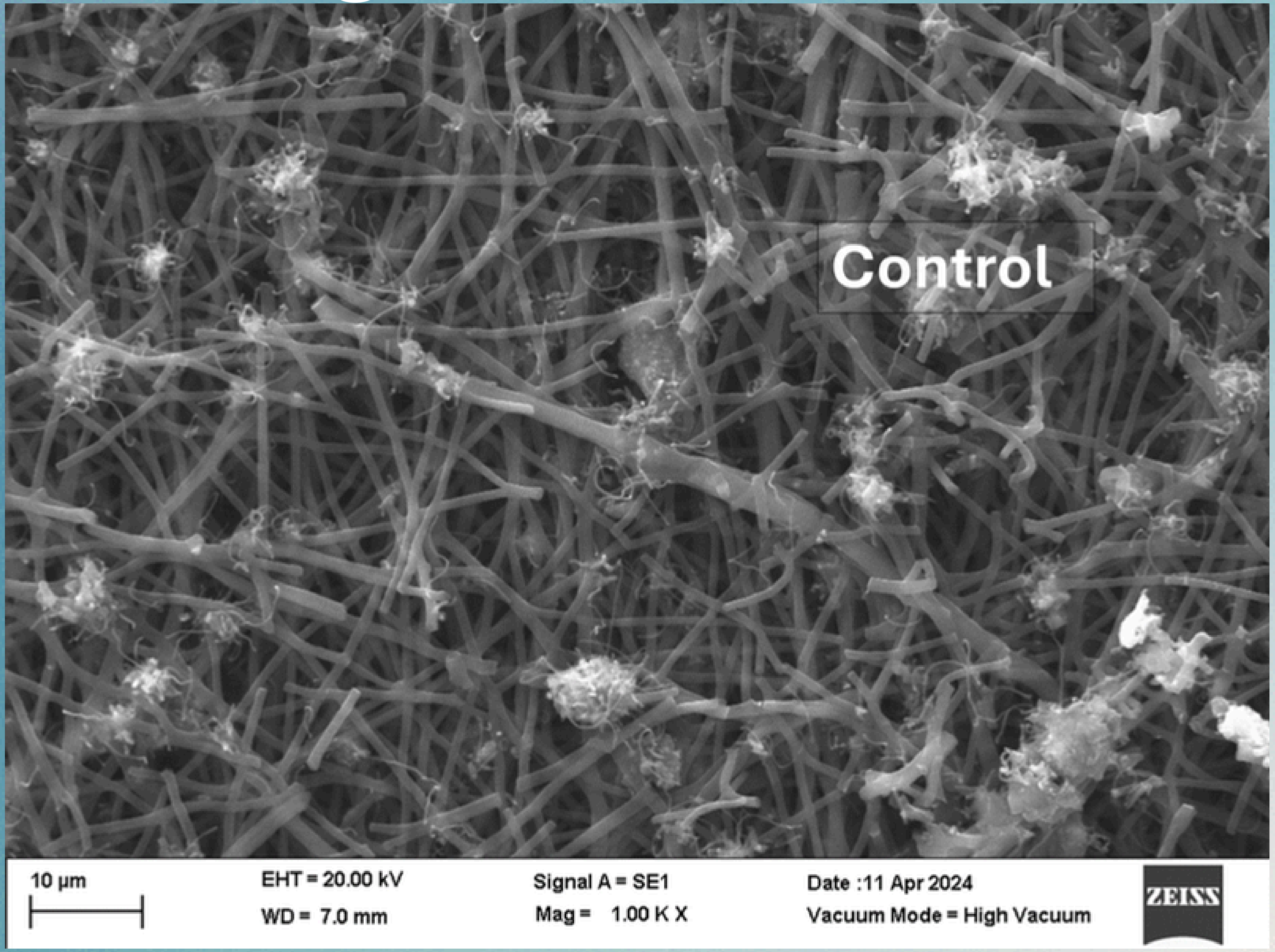
b)



a) Secondary electron signal. b) Backscattered electron signal. EHT = 20.00 kV, WD = 7.00 mm, Mag = ×10.00K.

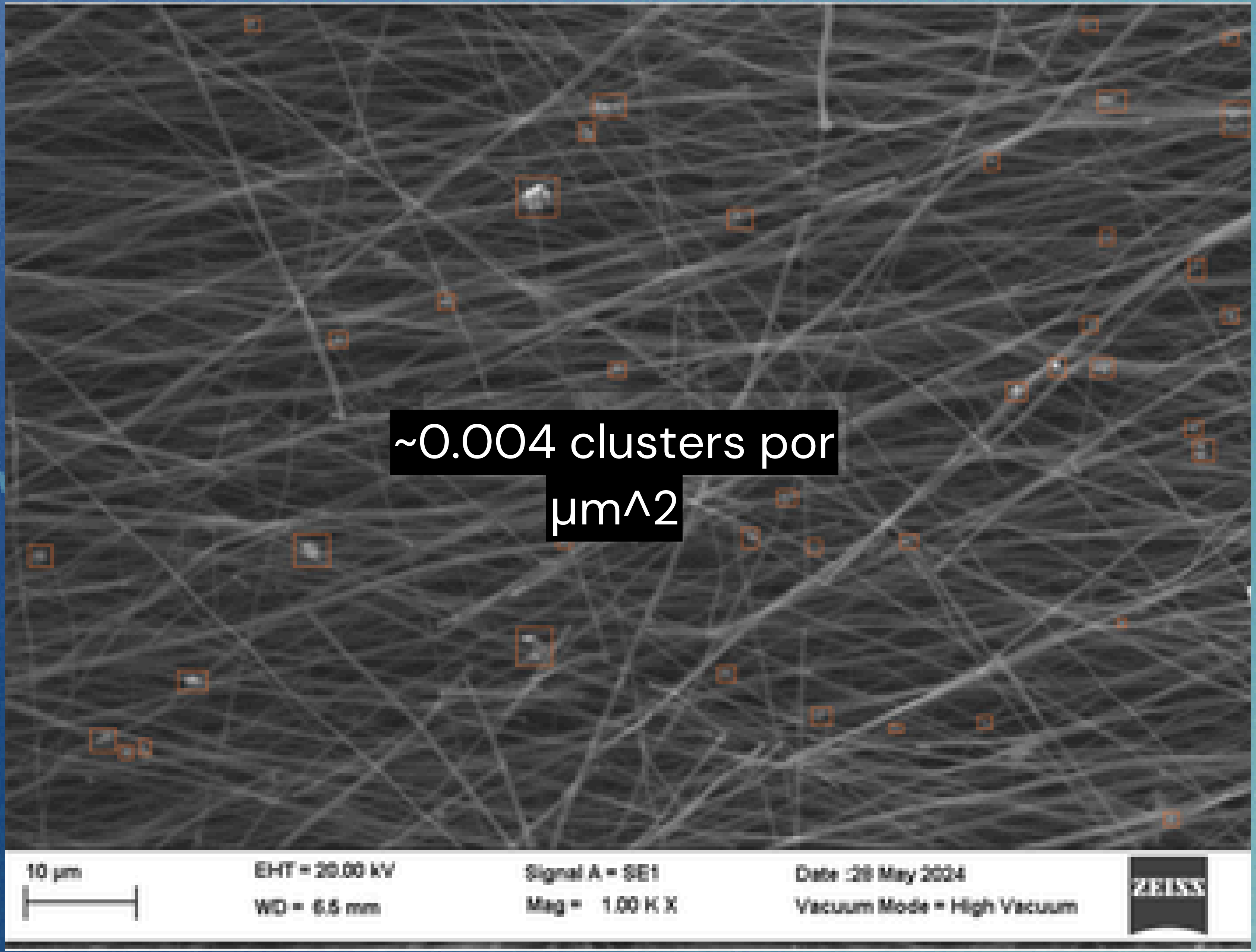
Why was there no growth of carbon nanotubes?

- Distribution of FeNPs in the polymer matrix and cluster size
- Pyrolysis conditions
- Distribution of the polymer matrix
- Contaminants

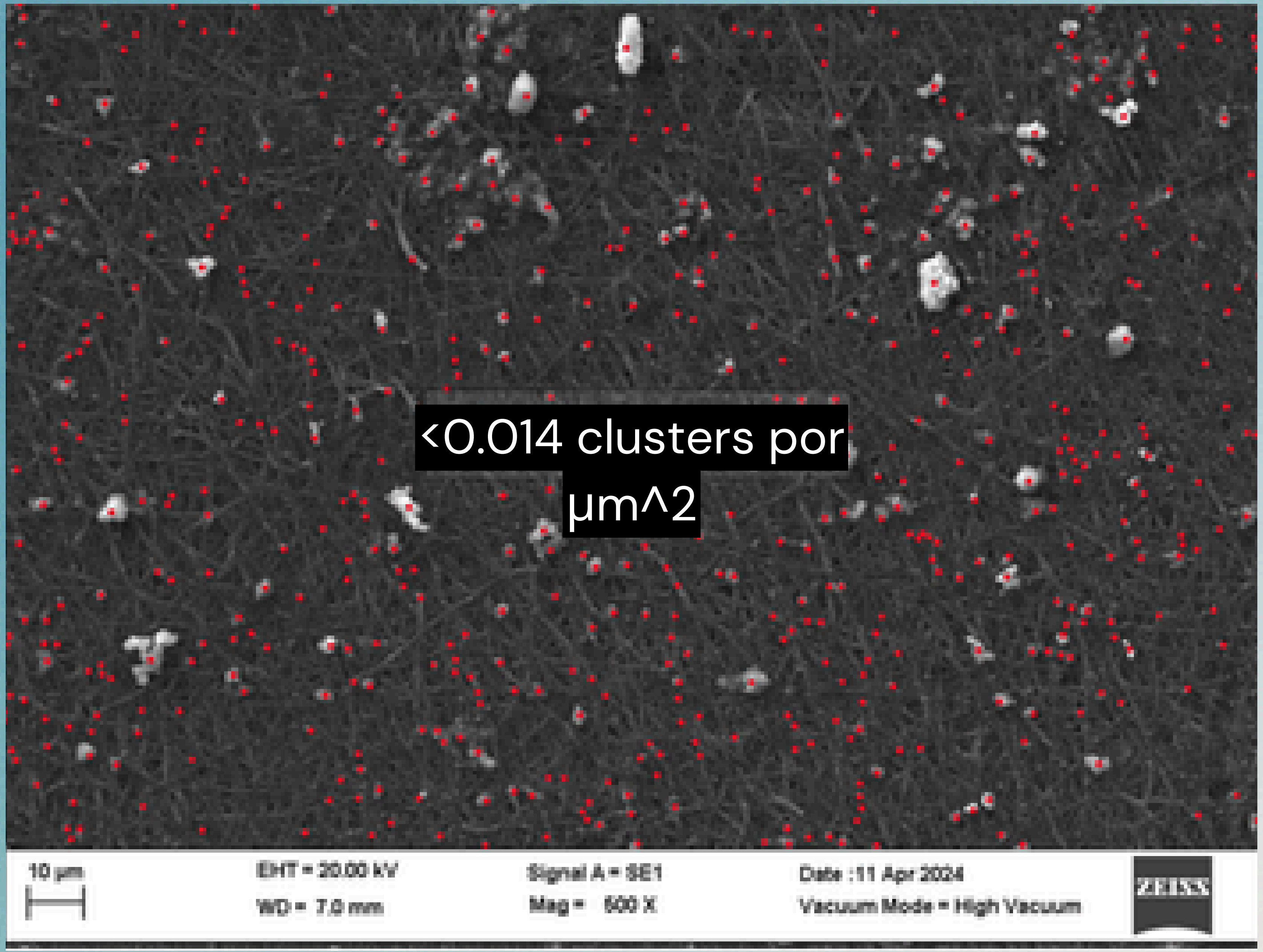


Control sample: PAN with pyrolysis and carbon nanotube growth.

Distribution of FeNPs in the polymer matrix and cluster size



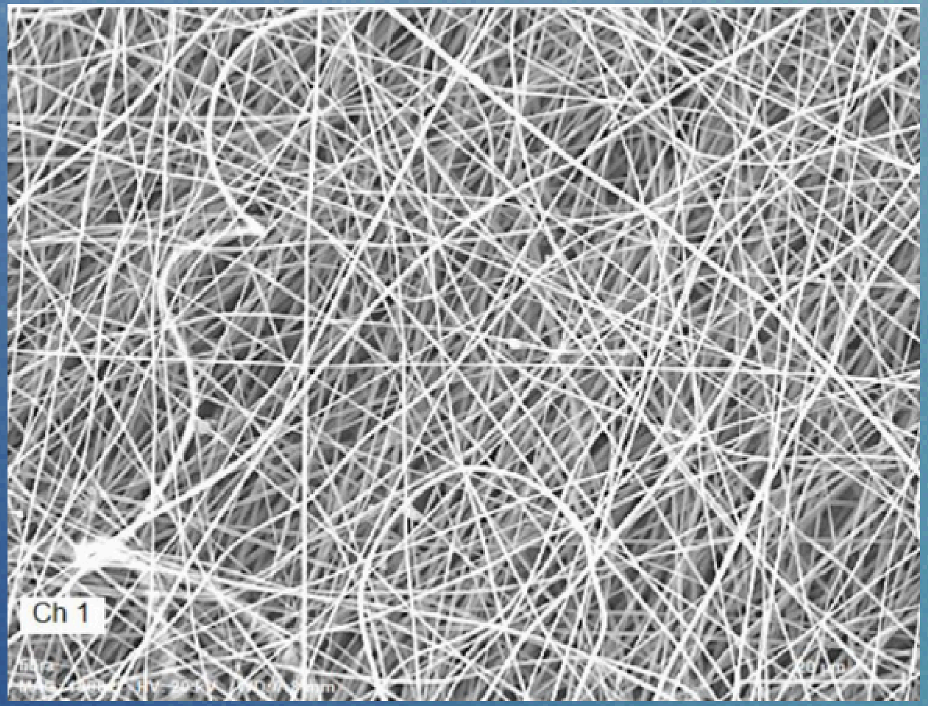
Cluster density in the sample. Mag = 1000×

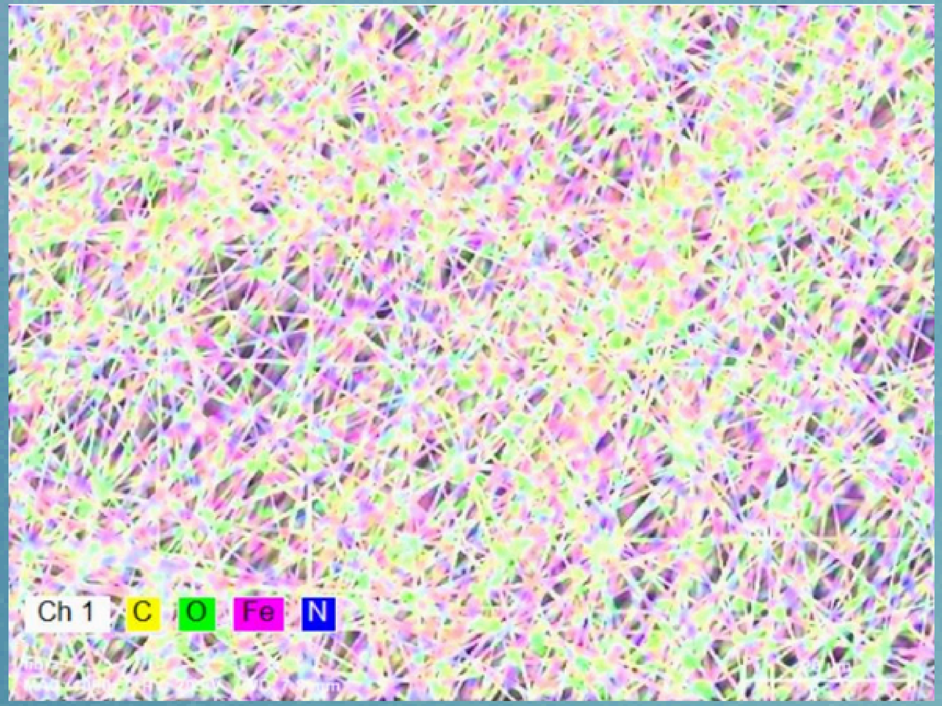


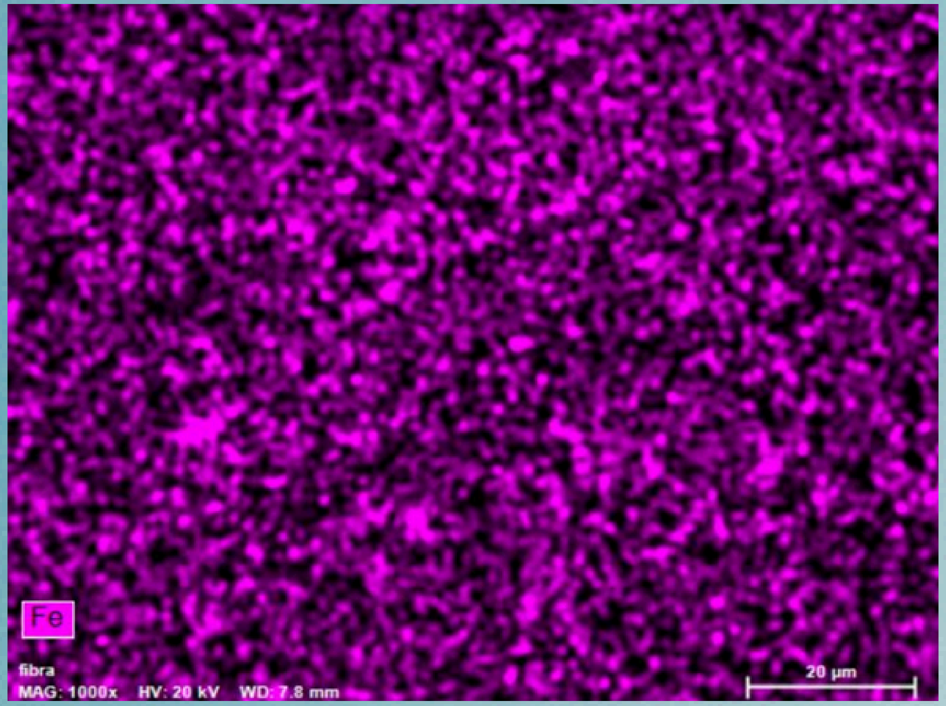
Cluster density in the control sample. Mag = 500×

Chemical Mapping by EDS

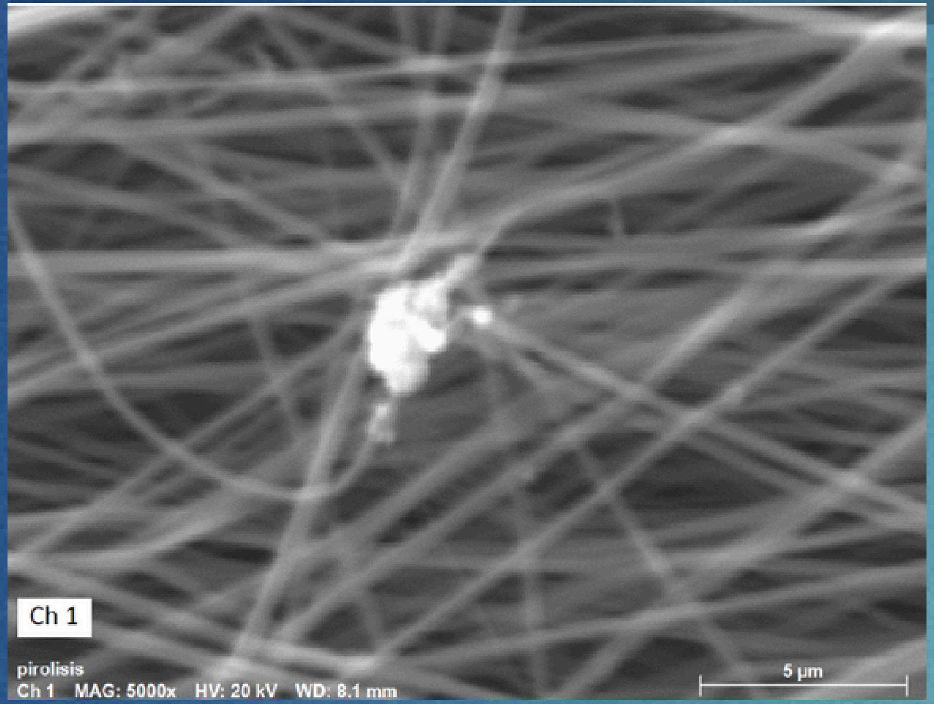
PAN fibers with FeNPs

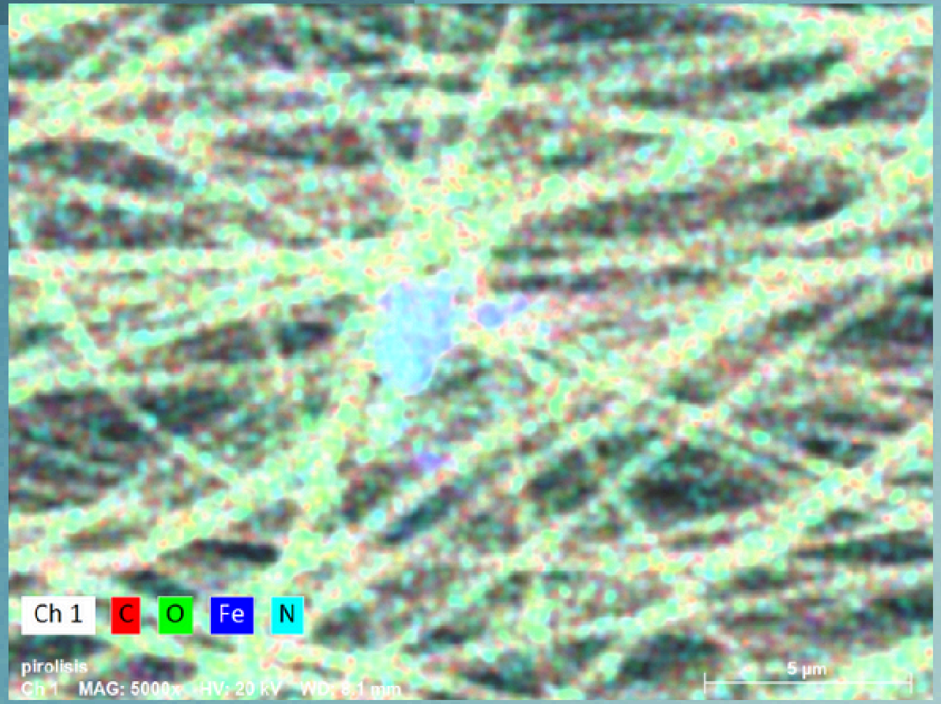


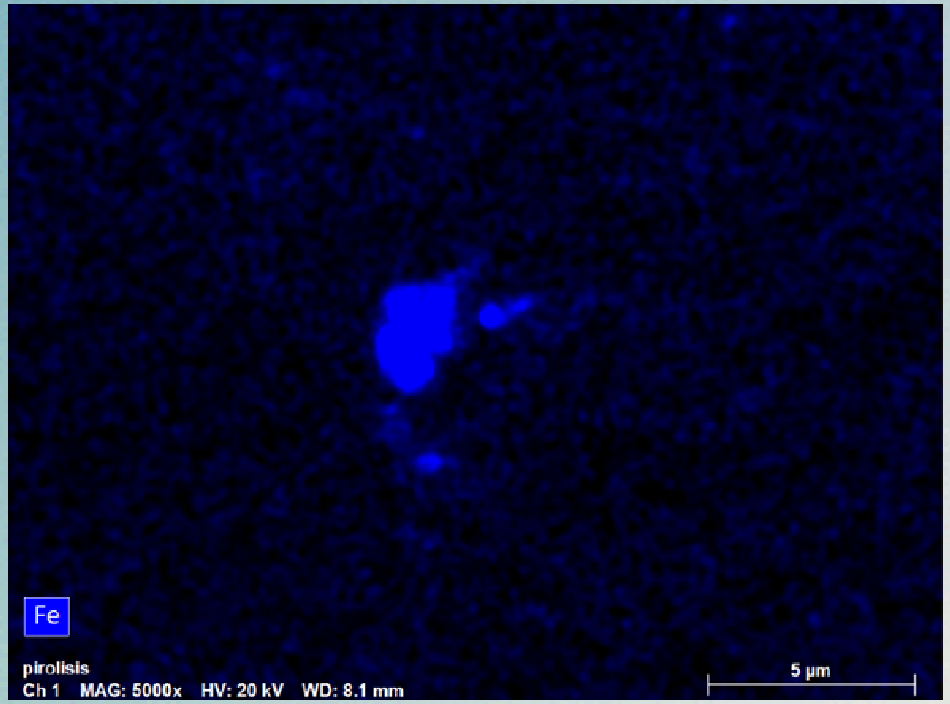




PAN fibers with FeNPs after pyrolysis



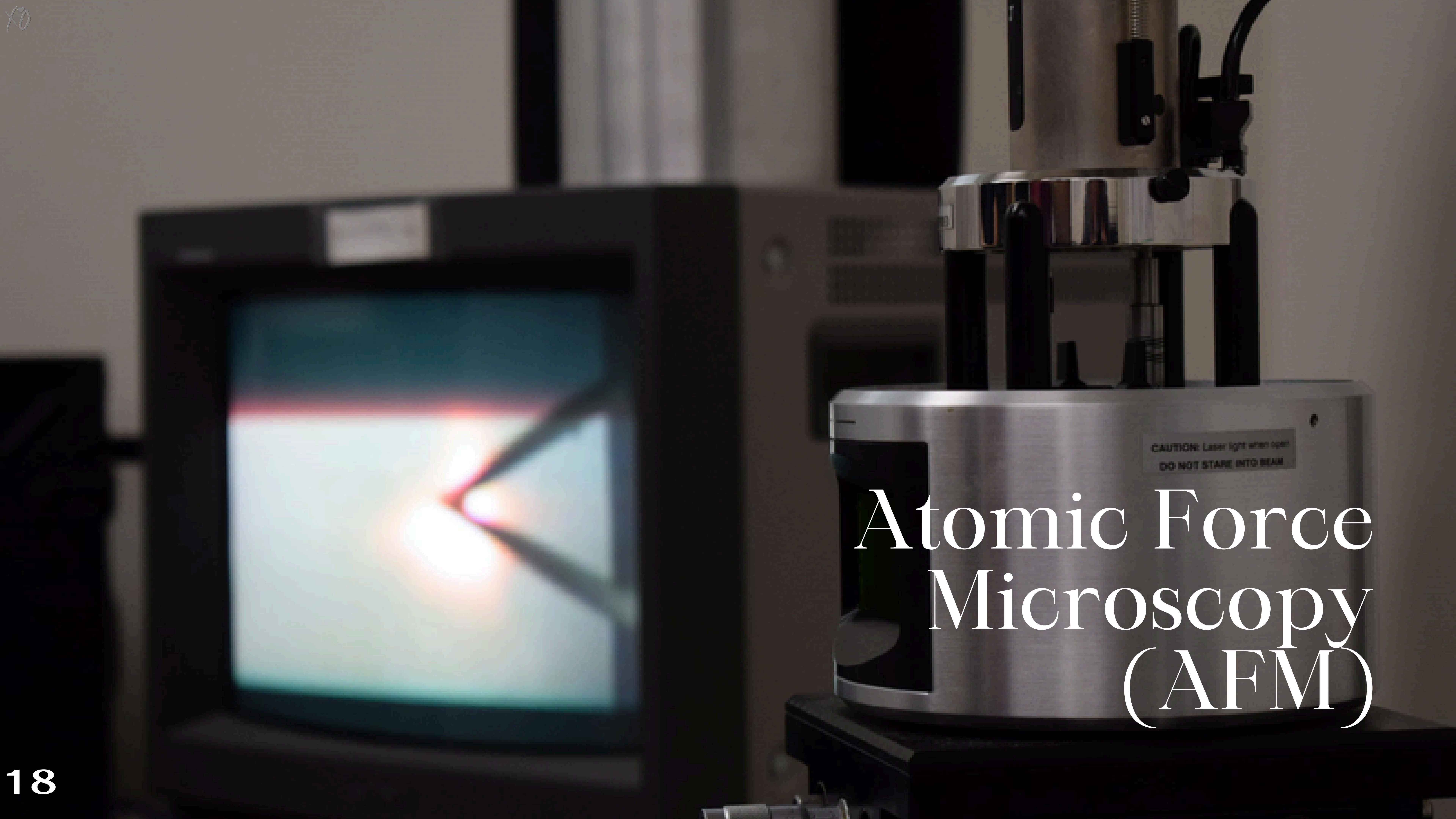




Elemento	Fibras PAN (%)	Pirolisis (%)
Carbono	67.3	74.46
Oxígeno	24.76	12.32
Nitrógeno	6.13	10.44
Hierro	1.8	2.77

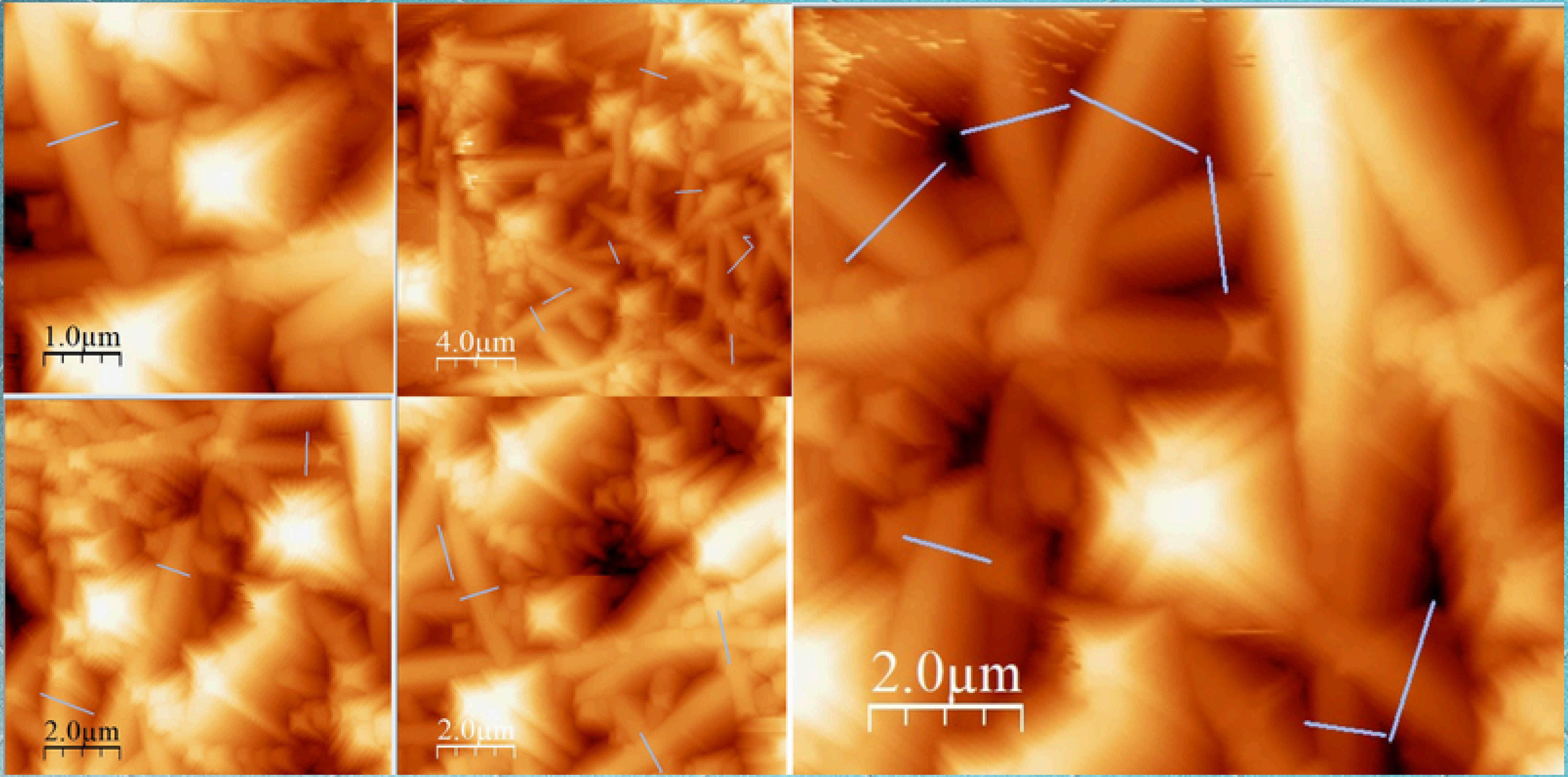
Elements found in the samples by EDS

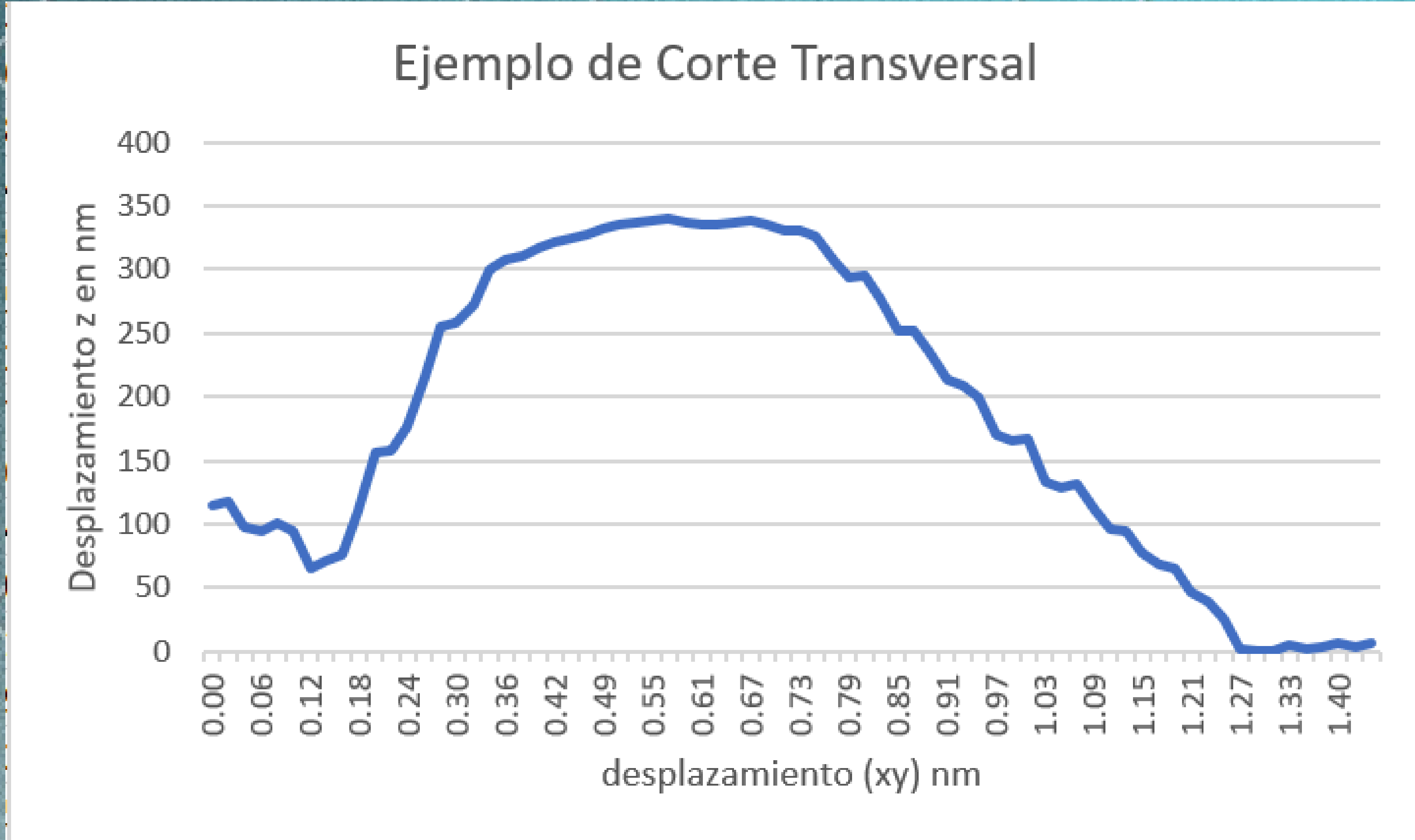
The results show that the iron concentration is significantly lower than expected, which could explain the inhibition of nanotube growth in the pyrolyzed samples. The insufficient concentration suggests the presence of contaminants or issues in the pyrolysis process.

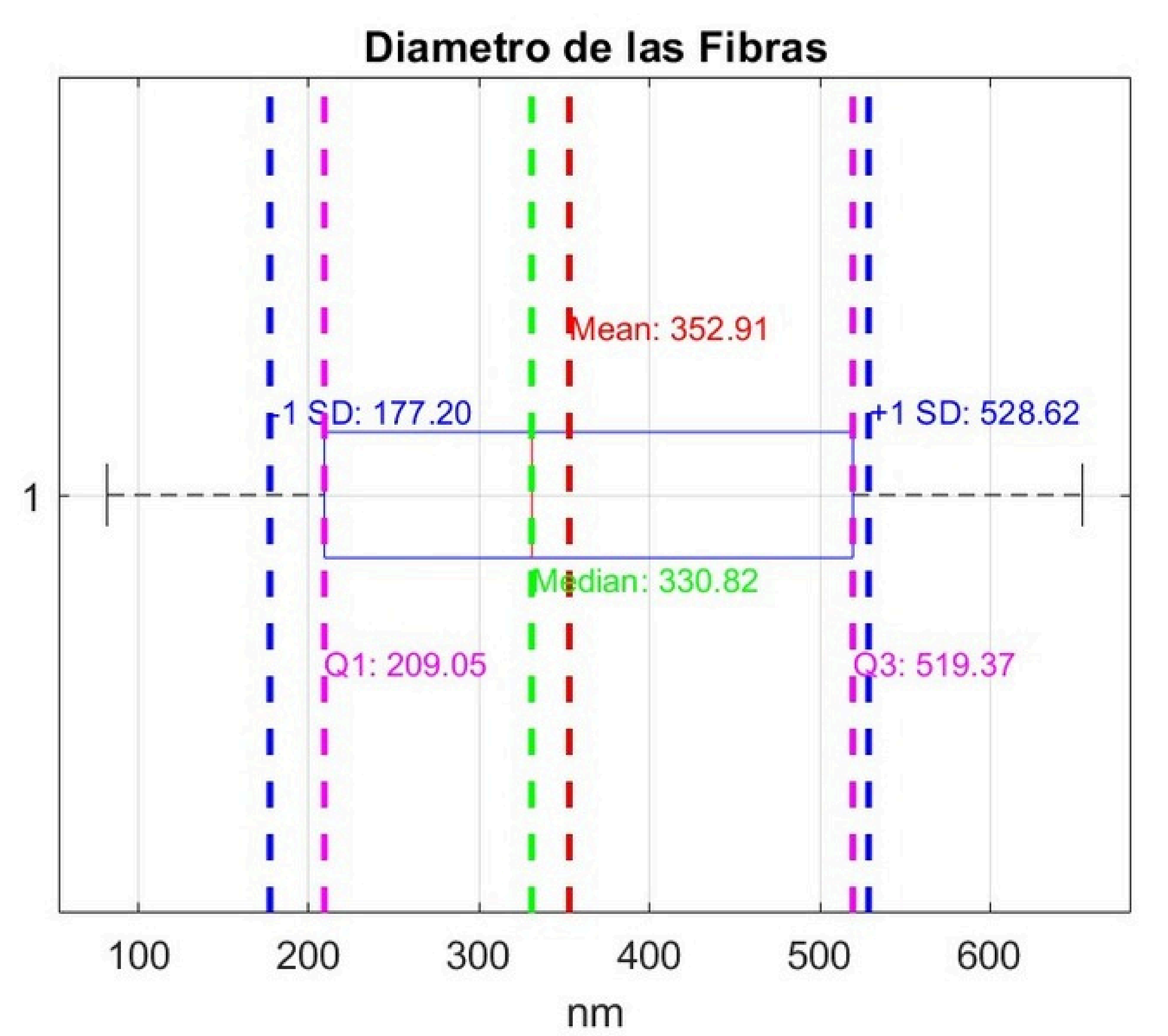


Atomic Force Microscopy (AFM)

Cross-Sections




Ejemplo de Corte Transversal
Desplazamiento z en nm
400
350
300
250
200
150
100
50
0
0.00
0.06
0.12
0.18
0.24
0.30
0.36
0.42
0.49
0.55
0.61
0.67
0.73
0.79
0.85
0.91
0.97
1.03
1.09
1.15
1.21
1.27
1.33
1.40
desplazamiento (xy) nm

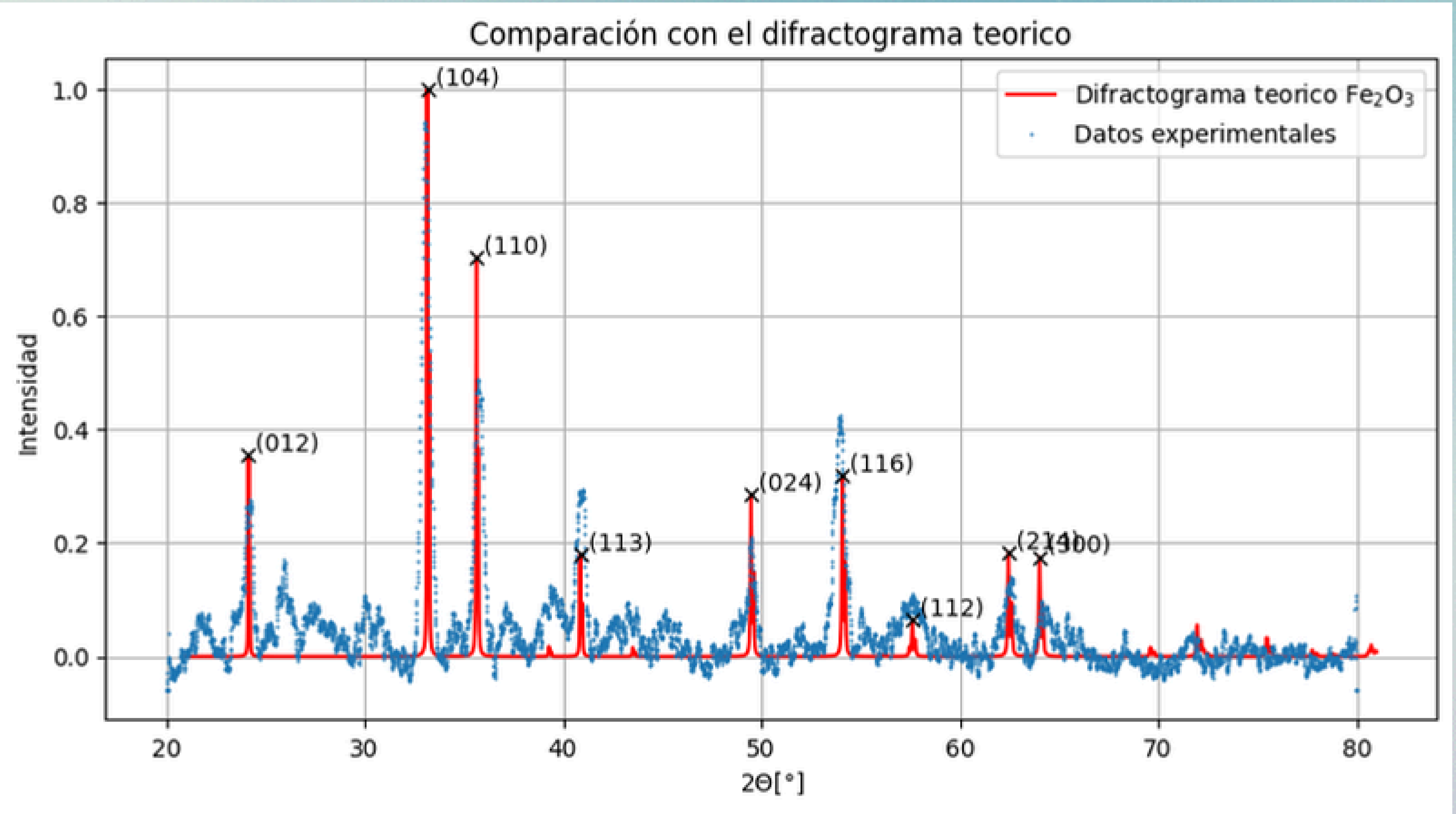

Diametro de las Fibras
Mean: 352.91
-1 SD: 177.20
+1 SD: 528.62
1
Median: 330.82
Q1: 209.05
Q3: 519.37
100
200
300
400
500
600
nm

X-ray Diffraction (XRD)

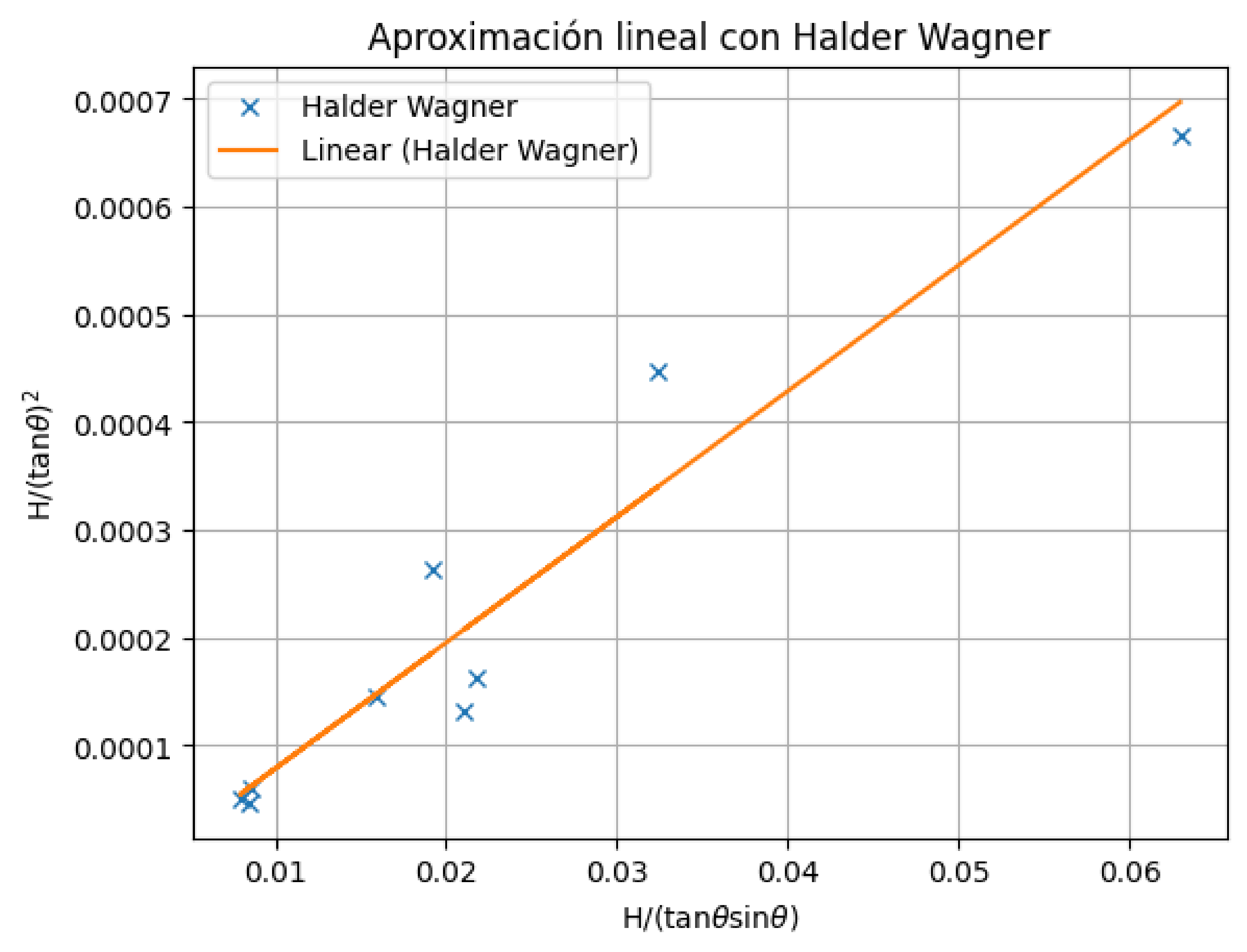
Parámetro	Calculado con PreDict	Parámetro de la red cristalina en COD
a (Å)	5.01649	5.0347
b (Å)	5.01649	5.0347
c (Å)	13.78803	13.7473
α	90°	90.004°
β	90°	90.004°
γ	90°	120.001°
V(Å^3)	300.49	301.76

- **λCu = 1.540 Å**
- **Scan rate = 0.33 °/minute**
- **Rhombohedral structure**

Comparative table between the experimentally calculated values and those from COD (entry 9015964) for the unit cell parameters of the Fe_2O_3 crystalline lattice.



Comparison of the experimental data with the theoretical diffractogram



Halder-Wagner Approximation

SCHRRER:

- The crystallite size was determined to be 12.918 nm.

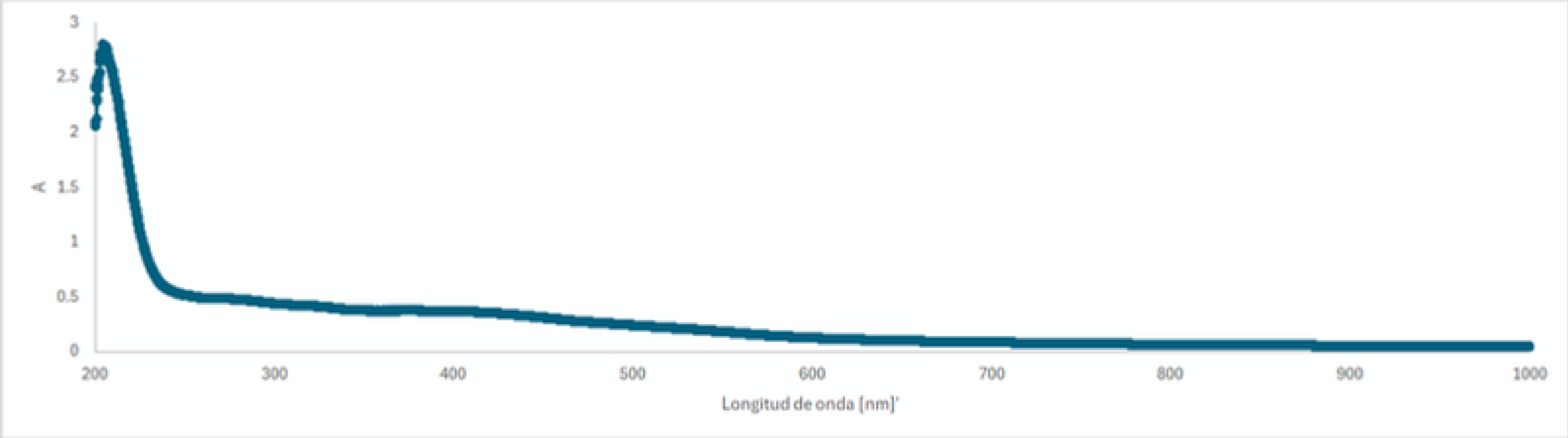
HALDER-WAGNER:

- The crystallite size was determined to be 13.197 nm.
- The microstrain was calculated as 0.155%.

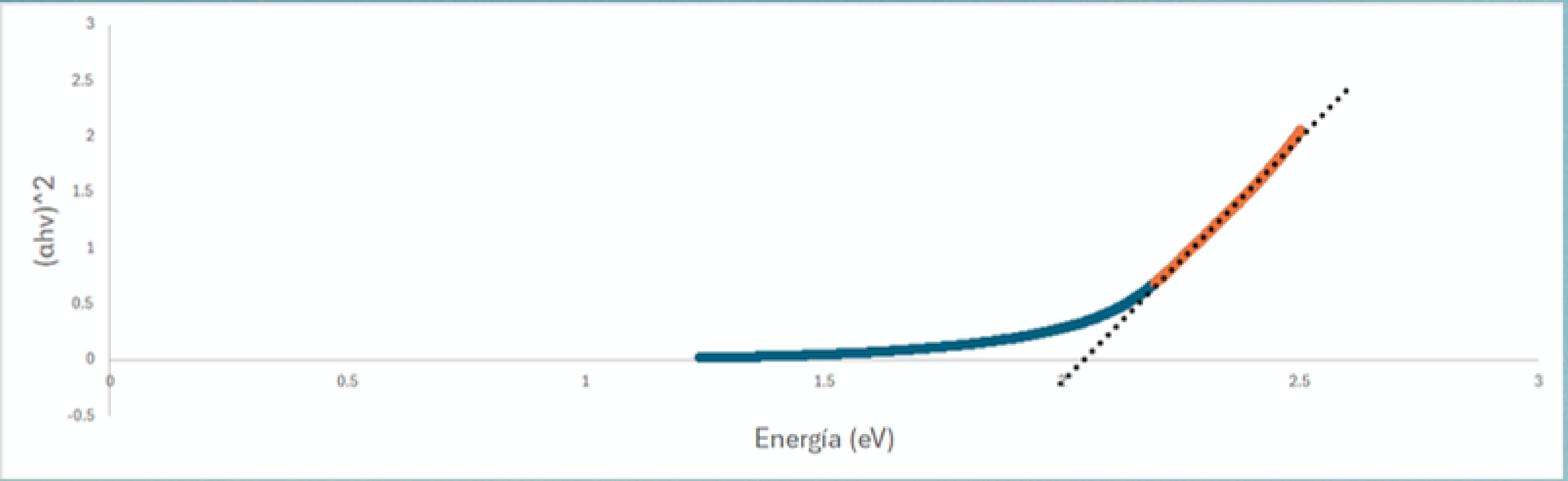
These values fall within the range reported in the literature for Fe_2O_3 crystallites (5–70 nm)

UV-Vis

Absorbance vs. wavelength (nm) graph



Tauc plot: the orange section is the first absorption band, and the black dotted line is its linear regression.



Band gap: 2.04 eV

UV-Vis

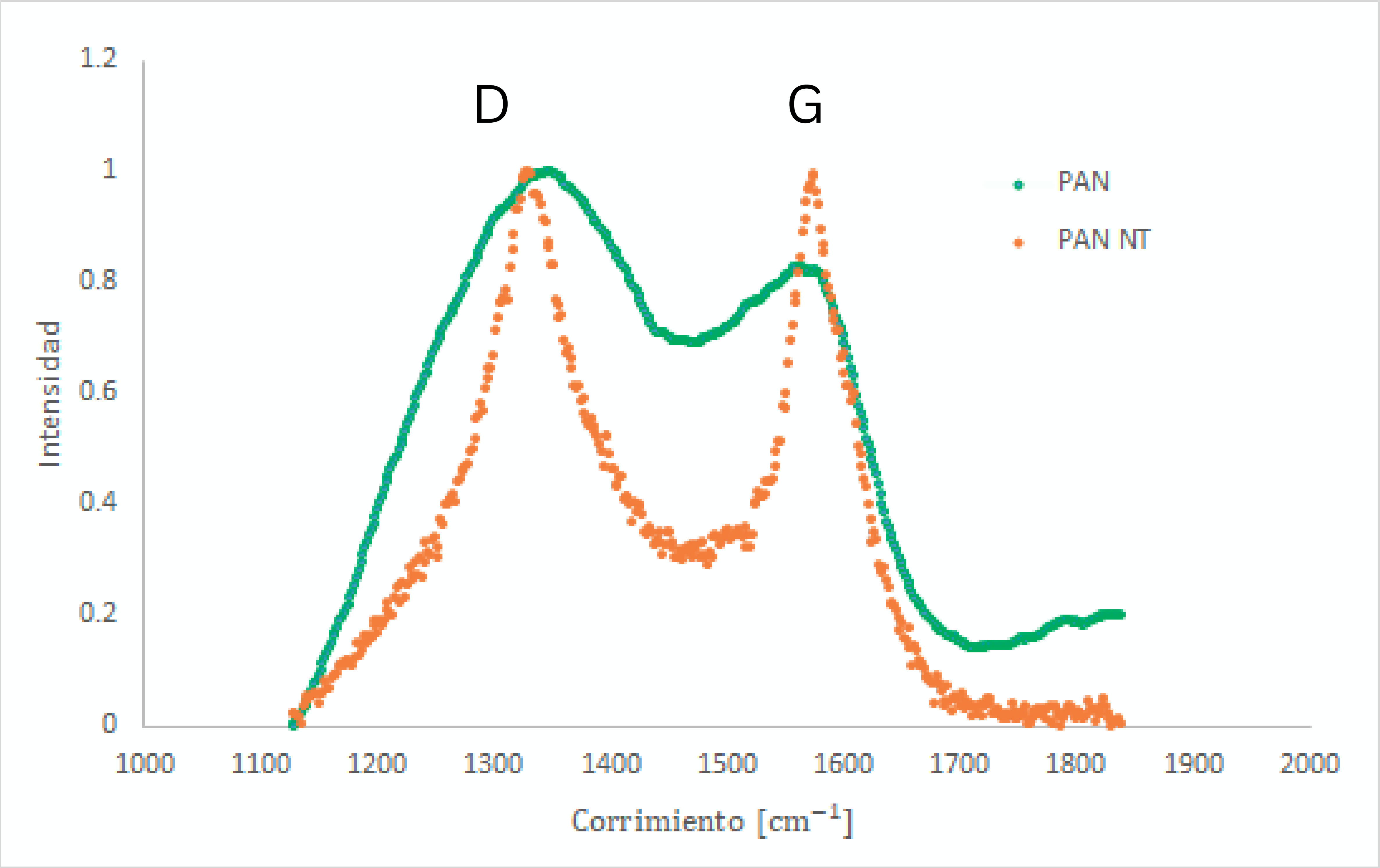
Since the goal is to minimize the band gap, it is recommended to dope with silver nitrate ($AgNO_3$) at a concentration of 0.15 M to reduce it.

Tavera, A. C., Santiago, A. H., Méndez, H. F., Sánchez, M. M., Serrano, R. A., & Hernández, R. G. (2015). Síntesis y caracterización de nanopartículas de Fe203 dopadas con plata con propiedades f otoactivas. Quimica Hoy, 5(3), 1-5.

Raman

he D/G ratio is a good indicator of defects in CNTs

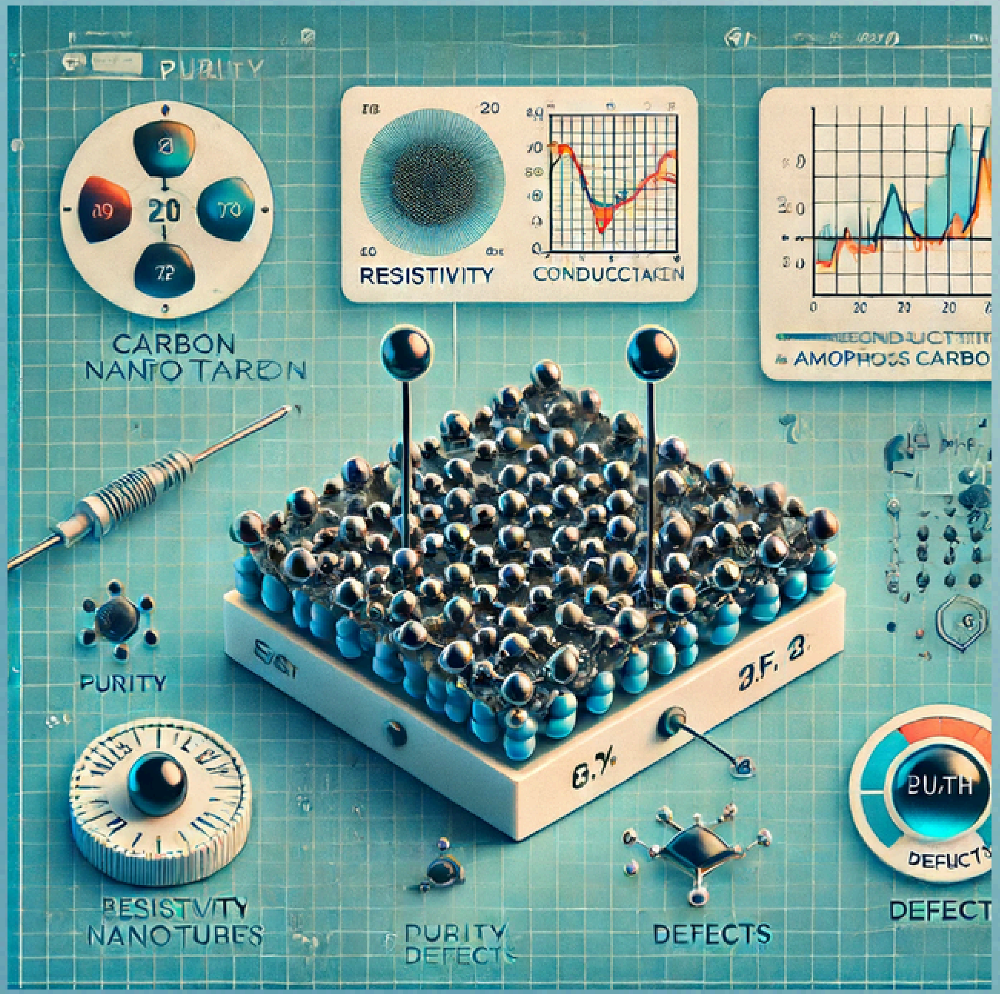
D (defects and disorder)
G (graphite)



$$R_{\mathrm{PAN}\,NT} \approx 1$$

$$R_{\mathrm{PAN}} \approx 1.21$$

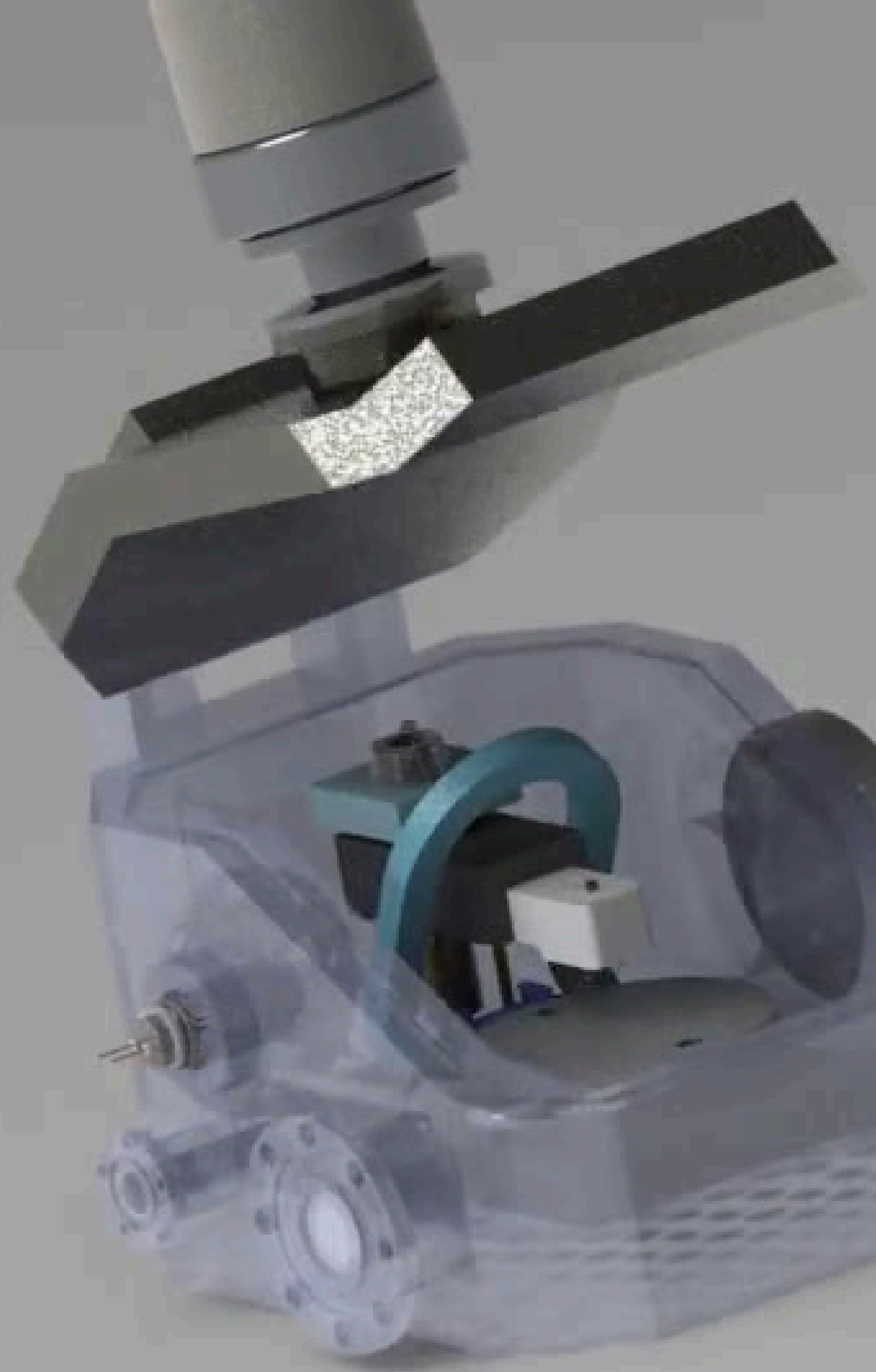
Electronic characterization

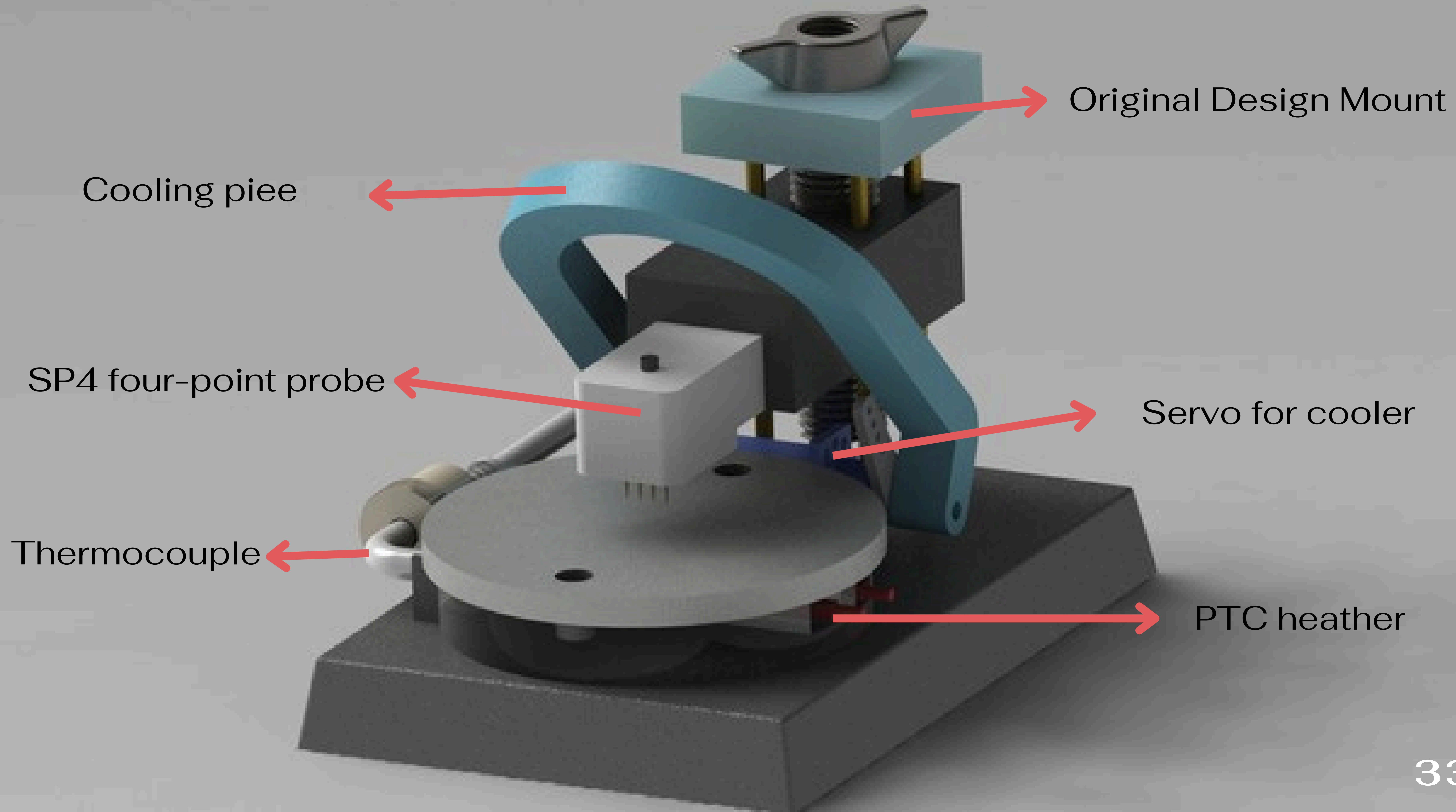


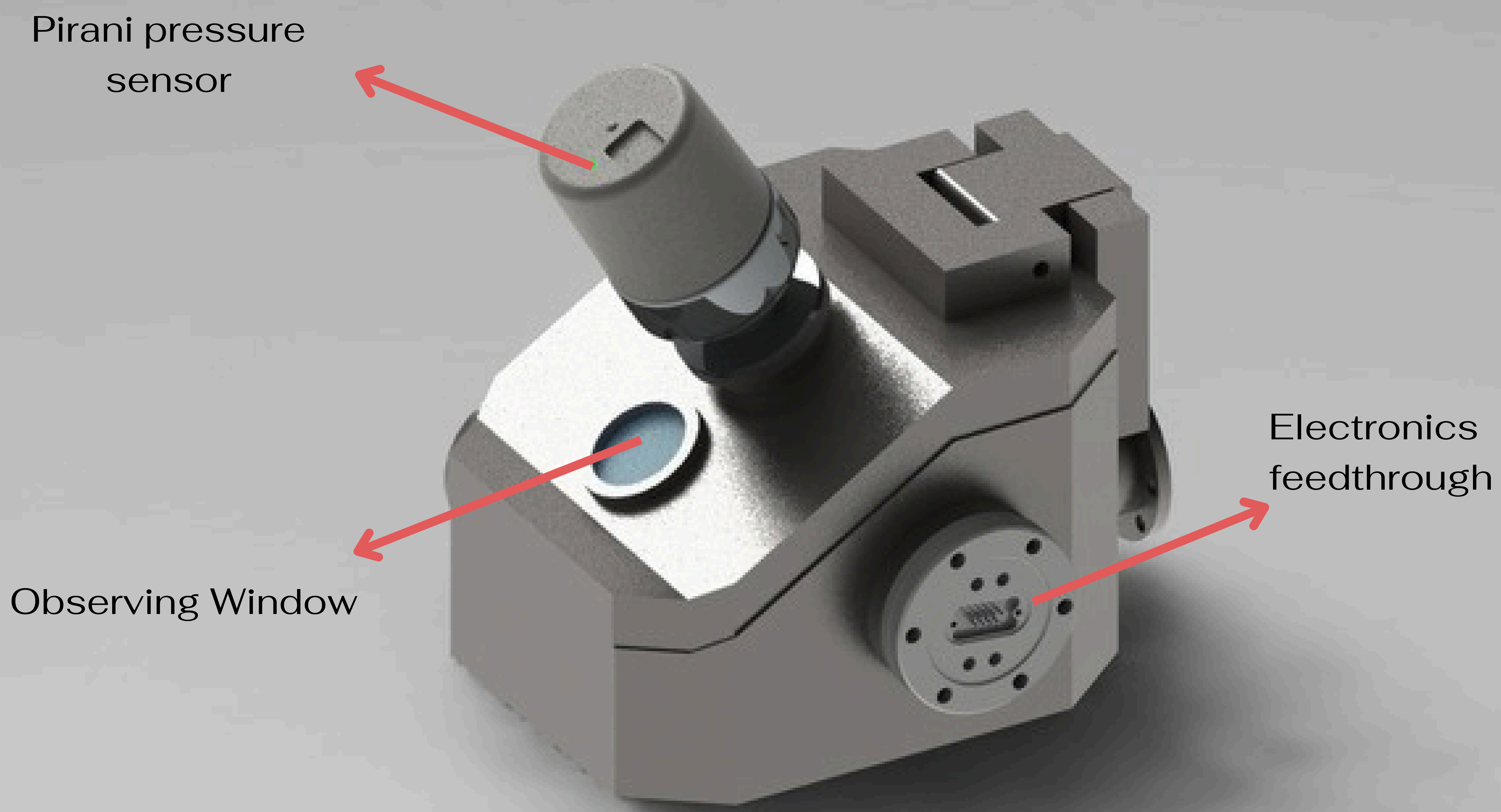
Resistivity

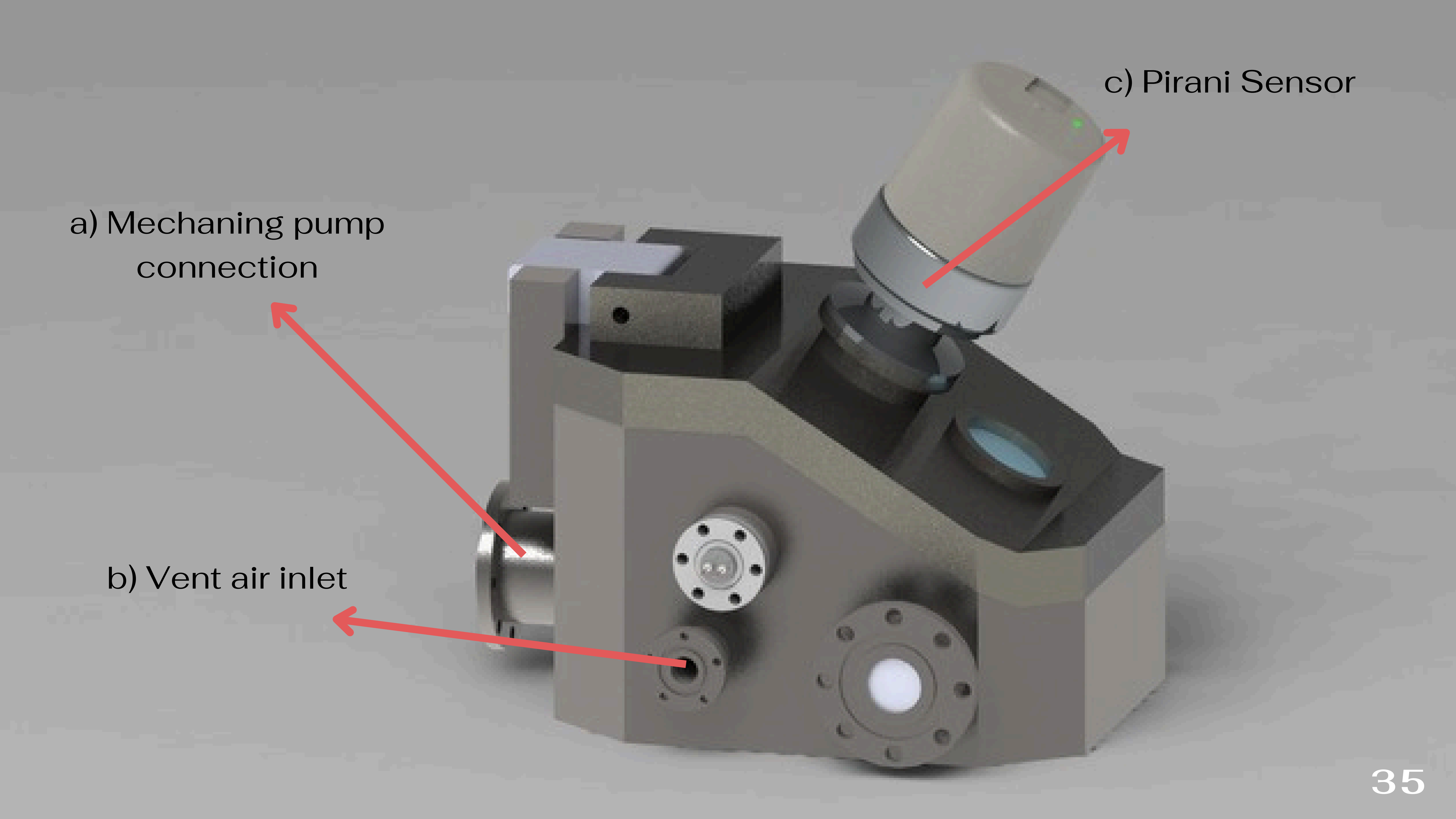
- Electrical conductivity.
- Material identification.
- Detection of impurities and defects.
- Optimizes electronic components.
- Manages heat dissipation.
- Reveals properties in nanomaterials.

Characterization chamber design



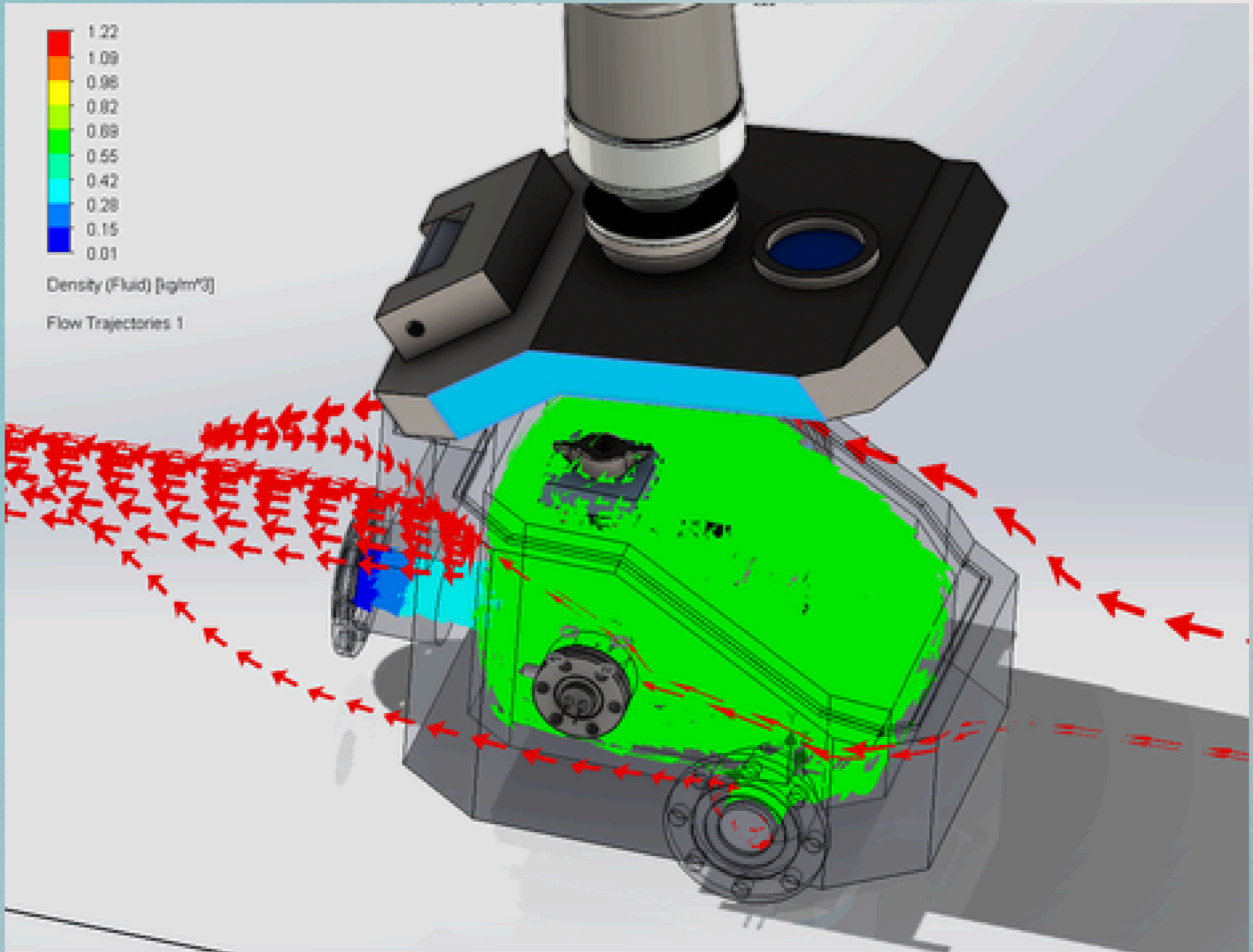

Original Design Mount
Cooling piee
SP4 four-point probe
Servo for cooler
Thermocouple
PTC heather

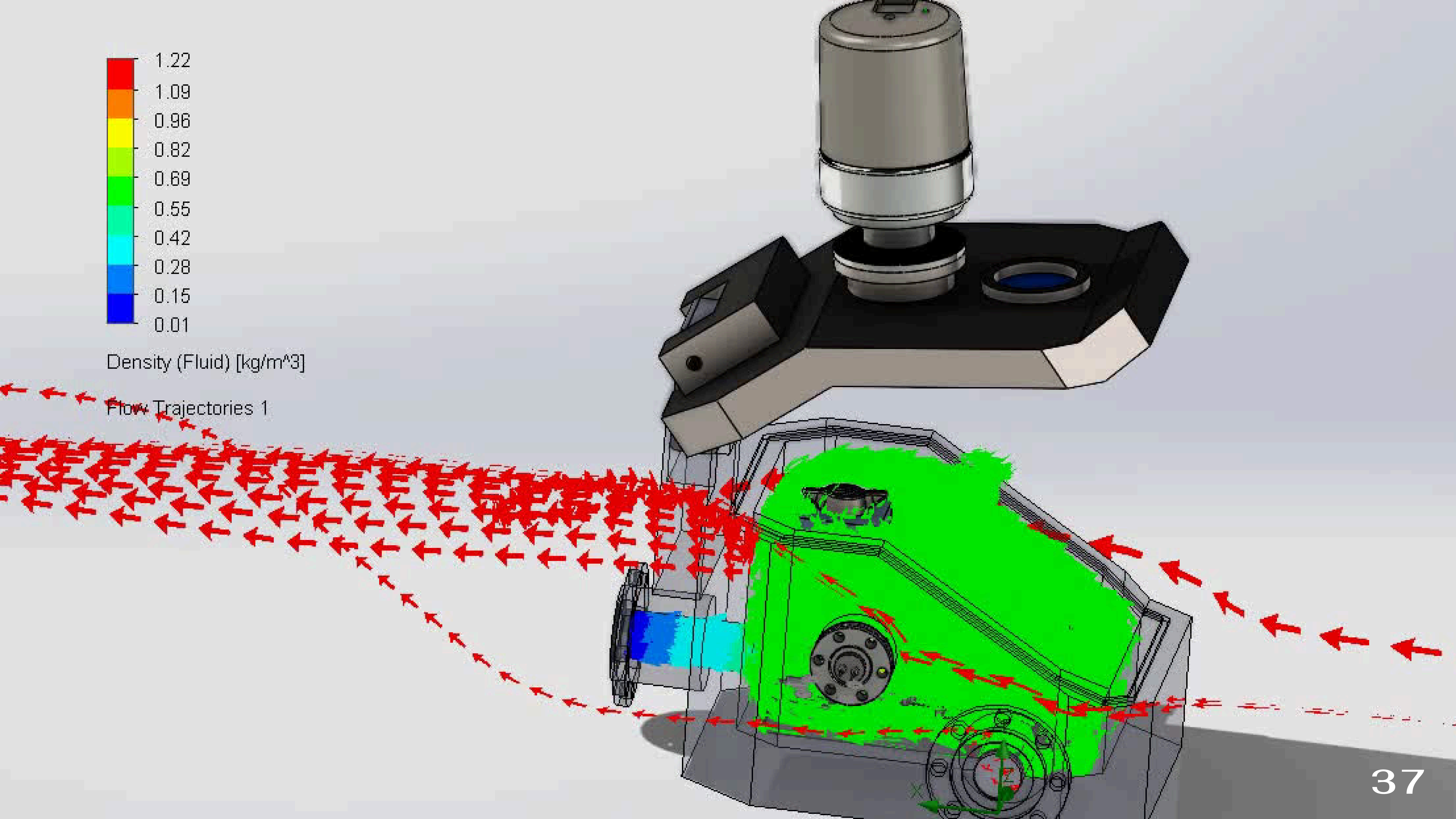

Pirani pressure
sensor
Observing Window
Electronics
feedthrough


c) Pirani Sensor
a) Mechaning pump
connection
b) Vent air inlet

Simulation

1.44 L




1.22
1.09
0.96
0.82
0.69
0.55
0.42
0.28
0.15
0.01
Density (Fluid) [kg/m^3]
Flow Trajectories 1

Vacuum pump

$2,300 USD.

Agilent Varian DS 40M

Technical Specifications

Free air displacement	l/min (cfm) [m^3/h]	50 Hz:	37 (1.3) [2.2]
		60 Hz:	43 (1.5) [2.6]
Pumping speed*	l/min (cfm) [m^3/h]	50 Hz:	30 (1.1) [1.8]
		60 Hz:	37 (1.3) [2.2]
Ultimate total pressure*	Pa (torr) [mbar]		6.7×10^{-1} (5.0×10^{-3}) [6.7×10^{-3}]
Noise level	dB(A)	50 Hz:	45
		60 Hz:	46
Oil capacity max	liters		0.37
Motor rating 1ph	kW	50 Hz:	0.1
		60 Hz:	0.1
Nominal rotation speed	rpm	50 Hz:	2280
		60 Hz:	3480
Weight	Kg (lb)		9.3 (20.5)
Dimensions	mm		120 (W) x 289 (L) x 203 (H)
Power cord included	Yes	50 Hz:	EU plug / UK plug
		60 Hz:	US plug
Inlet flange			16KF DN
Exhaust flange			16KF DN
Certifications			CE, cTUVus

* According to PNEUROP 6602

Costs

Components

Manufacturable components

Elemento	Cantidad	Precio (USD)	Total
Pfieffer Pirani Gauge KF40	1	$2,400.00	$2,400.00
Servomotor MD70MH v1	1	$55.00	$55.00
Bomba de Vacio	1	$2,300.00	$2,300.00
CF16_ElectricalFeedthrough_RadialORing	1	$40.00	$40.00
WATLOW 32-S0.125_n0.188D_Bayonet90	1	$87.00	$87.00
PTC heating plate	1	$48.91	$48.91
Perno	1	$15.00	$15.00
Steel Stick	1	$10.00	$10.00
Gasket	1	$472.00	$472.00
SIGNATONE SP4	1	$368.00	$368.00
VC1	1	$60.29	$60.29
VC2	1	$20.92	$20.92
VC3	1	$29.79	$29.79
VC4	1	$16.53	$16.53
VC5	1	$1,283.22	$1,283.22
VC6	1	$160.28	$160.28
VC7	1	$7.26	$7.26

Costs

Total	7374.2 USD	132,735.6 MXN

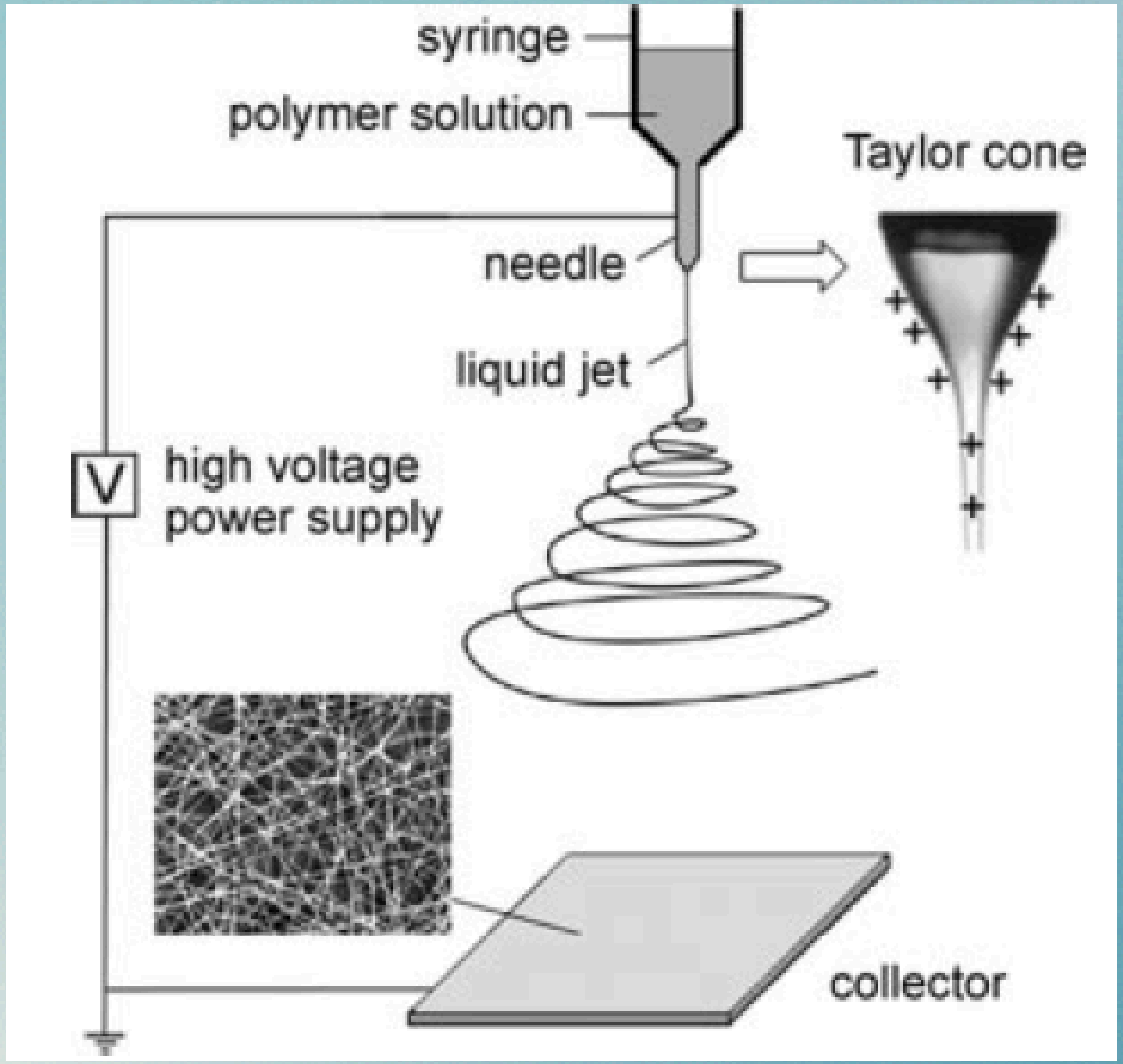
Conclusions

By means of advanced techniques, a material was characterized that could function as a supercapacitor. Possible improvements in the synthesis process were identified, and a vacuum chamber was also designed to carry out characterization processes in controlled environments.

¡Thanks!

Back up Slides

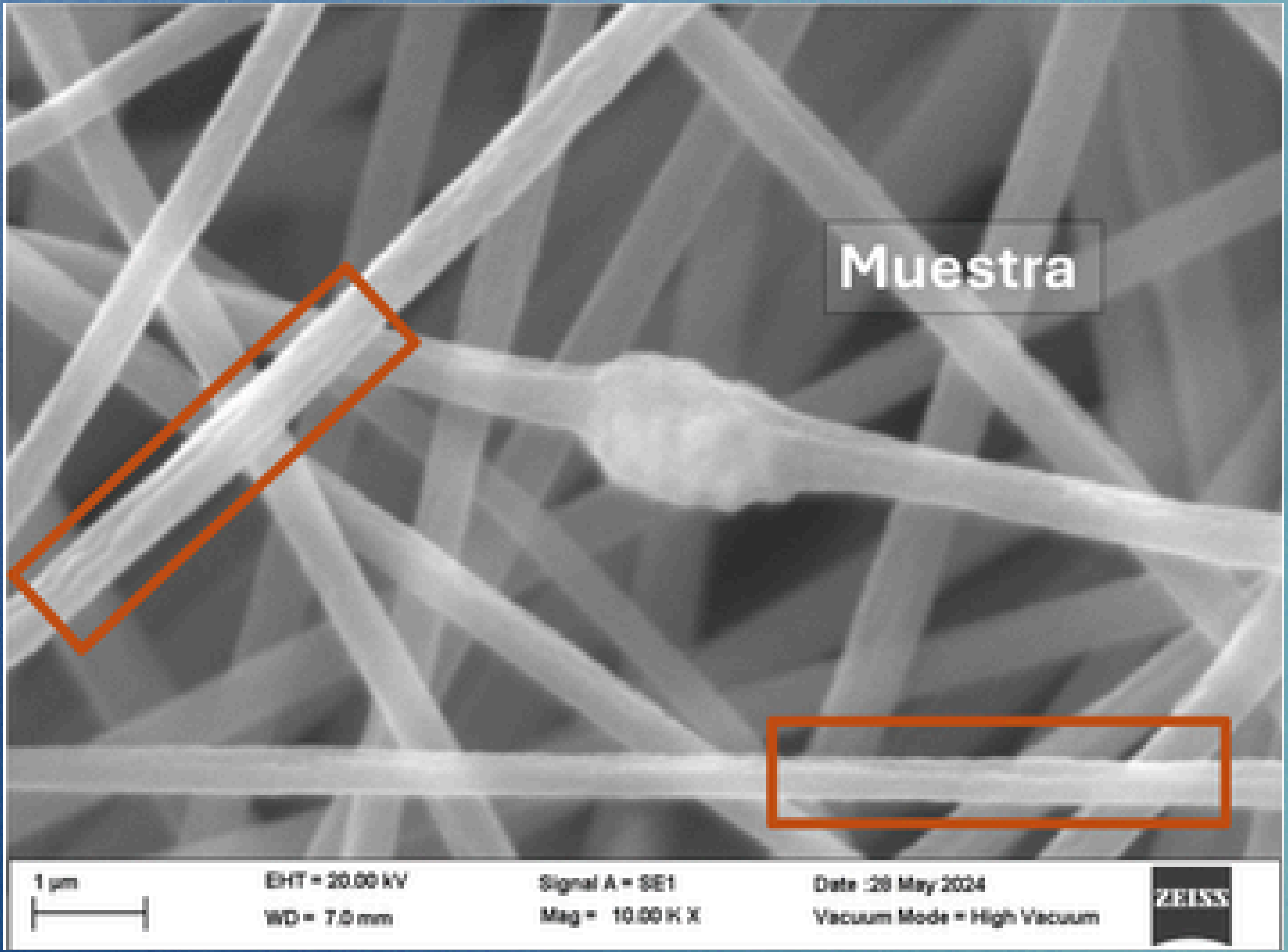
Electrospinning

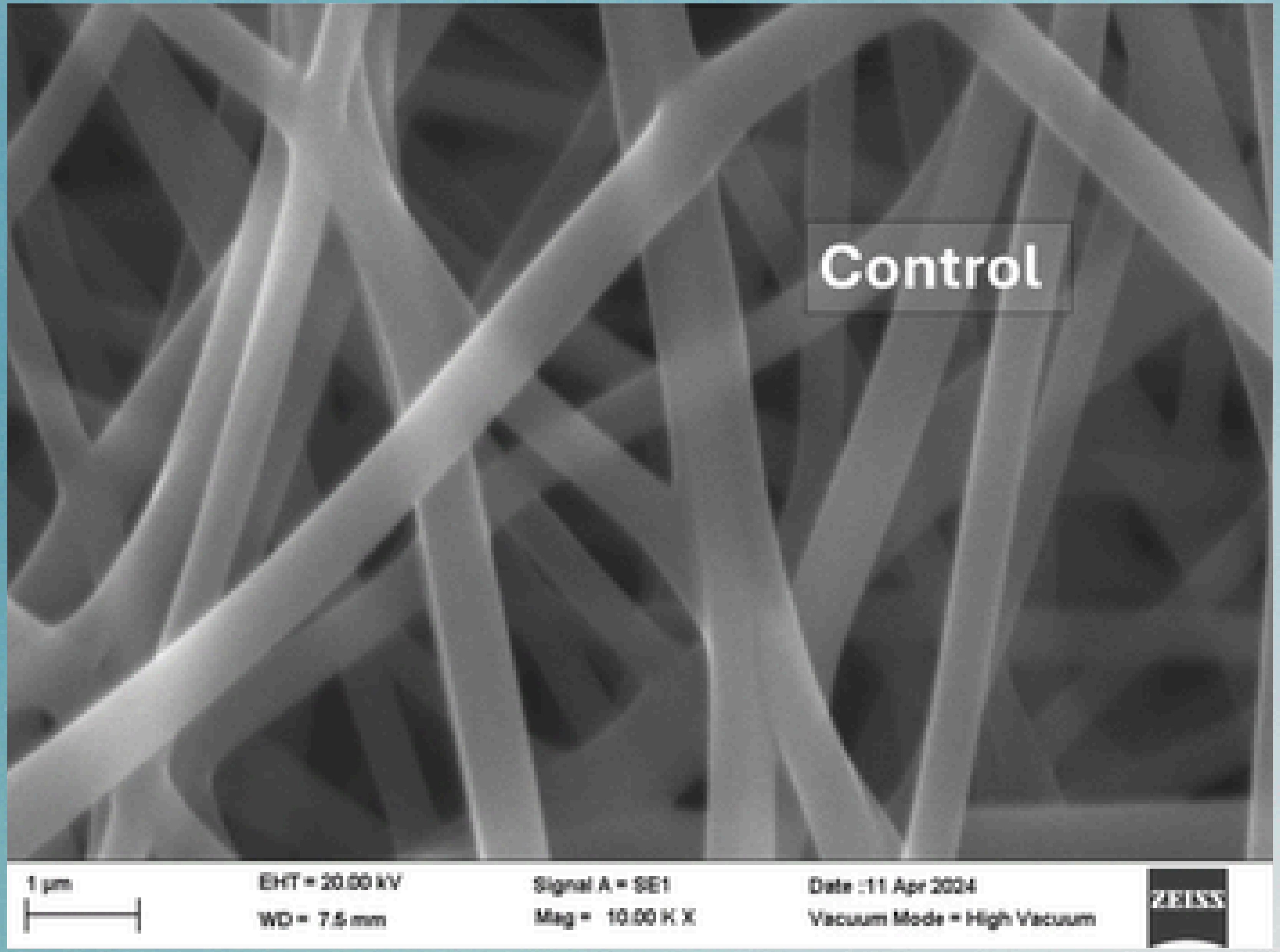


Due to the strong electric field, the drop coming out of the tip deforms and forms the characteristic 'Taylor cone,' from which an unstable jet of the solution emerges. The jet is attracted to the collector, and along the path from the tip to the collector, the solvent evaporates, solidifying the jet and forming very fine fibers that are deposited on the rotating collector, resulting in a nanofiber mat.

D.Li and Xia, Y. (2004). Electrospinning of nanofibers: Reinventing the wheel? Advanced Materials.

POLYMER MATRIX





Comparison of the polymer matrix distribution