ELI-NP Young Scientist and Young Engineer Days

Non-canonical method of producing graphene using Graphite Intercalated Compound exfoliated by Microwave Radiation

Doctoral School of Engineering and Applications of Lasers and Accelerators (SDIALA)

Laser System Department (LSD)

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Acknowledgements:

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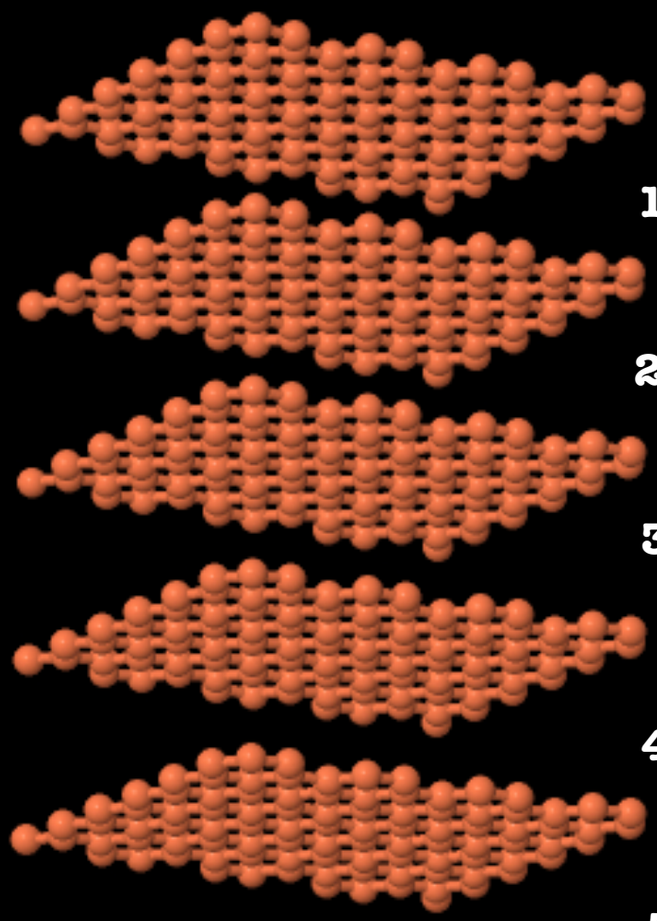
Dr. Cristina GHEORGHIU
Dr. Nikolay DJOURELOV
PhD Student Laurențiu DINCĂ

Bucharest, 2024

Outline



1. Experiments with Graphite Intercalated Compound (GIC)

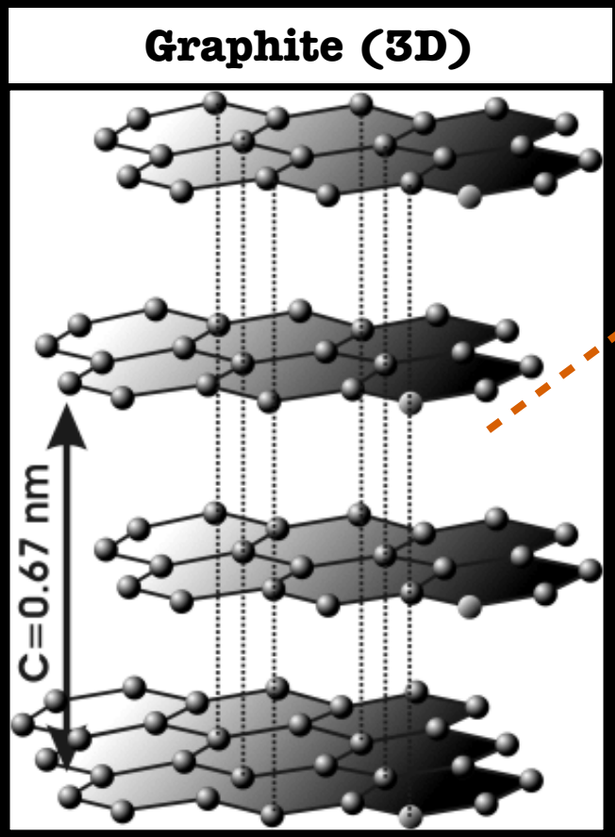
2. Identifying the best precursor (graphite powder) for efficient intercalation

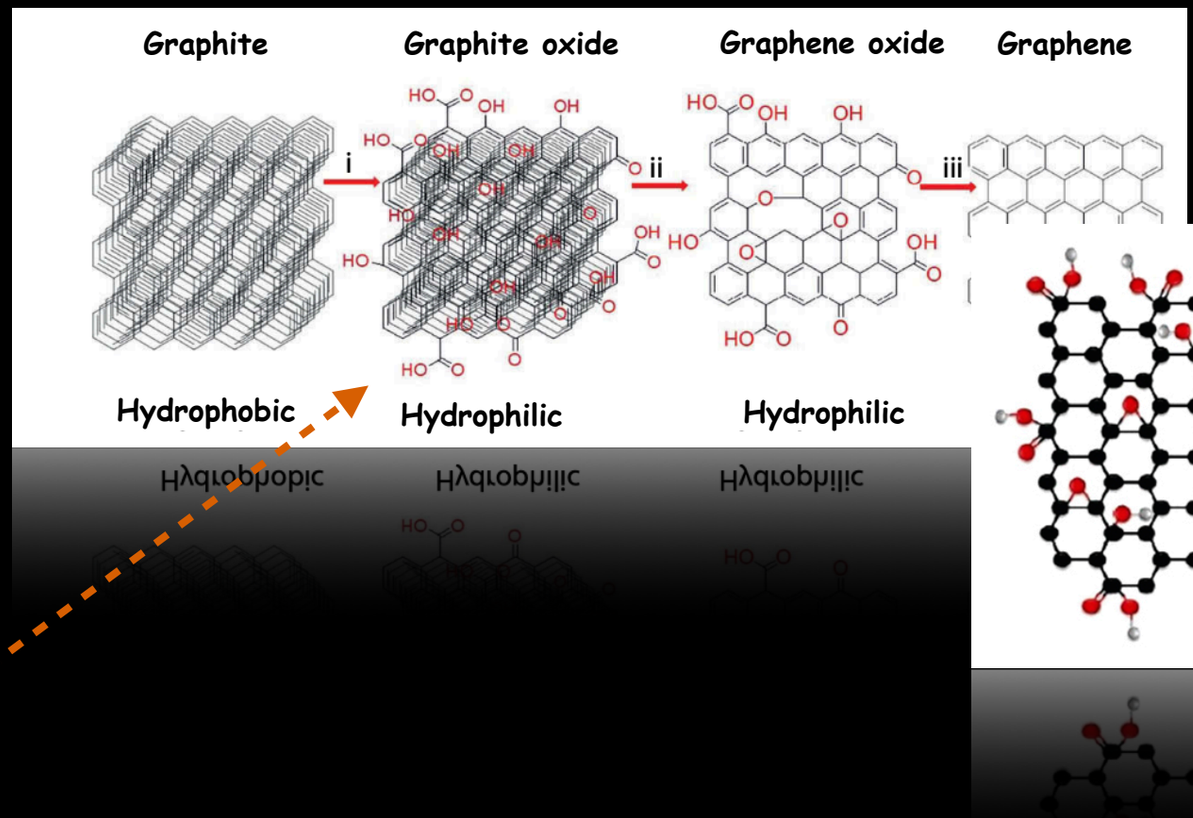
3. Chemical Synthesis of H_2SO_4-GIC

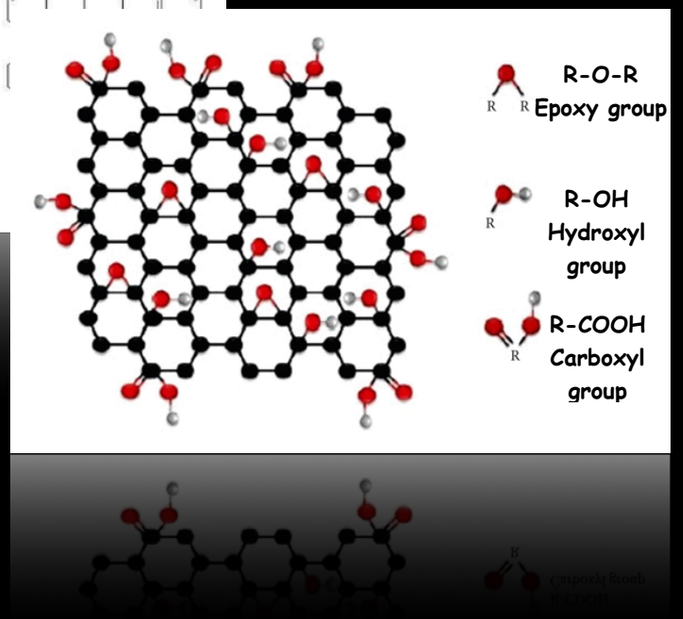
4. Characterization of H_2SO_4-GIC
   - XPS Analysis
   - SEM/EDS
   - AFM

5. Conclusions

1. Experiments with Graphite Intercalated Compound (GIC)







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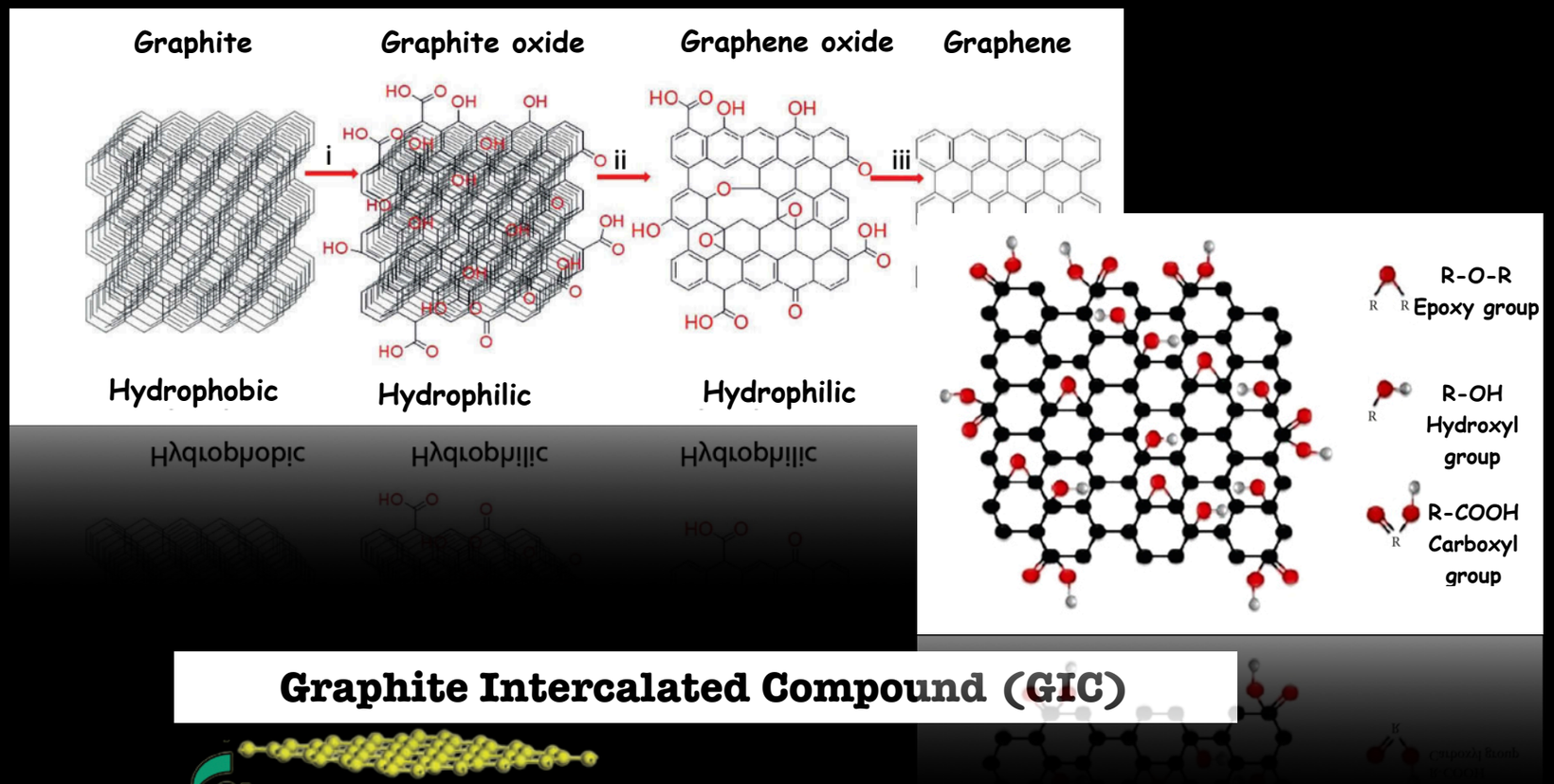
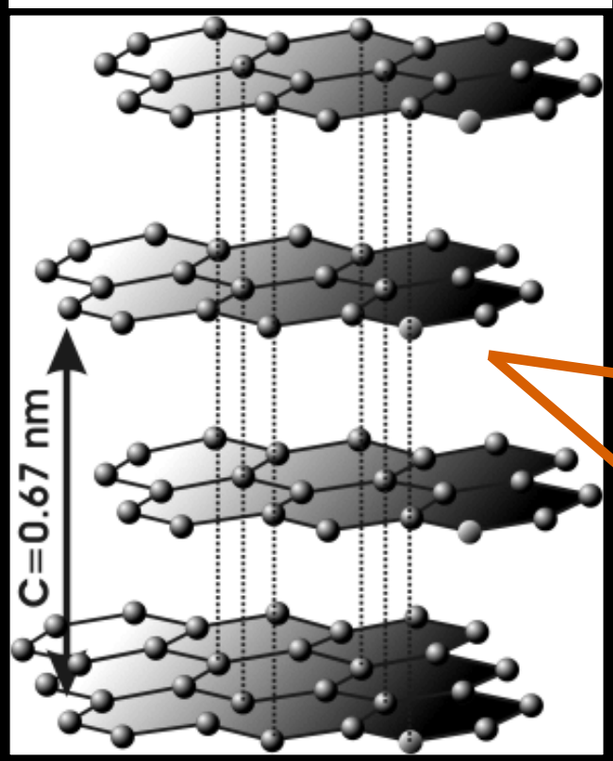
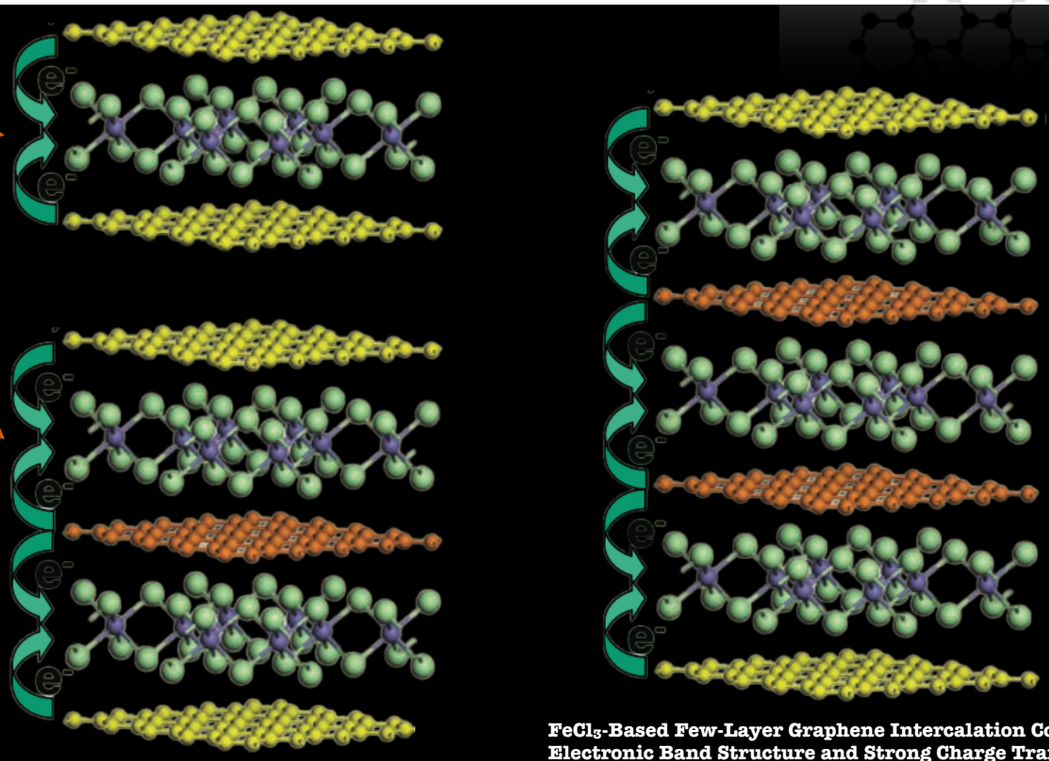


Graphite (3D)



Graphite Intercalated Compound (GIC)

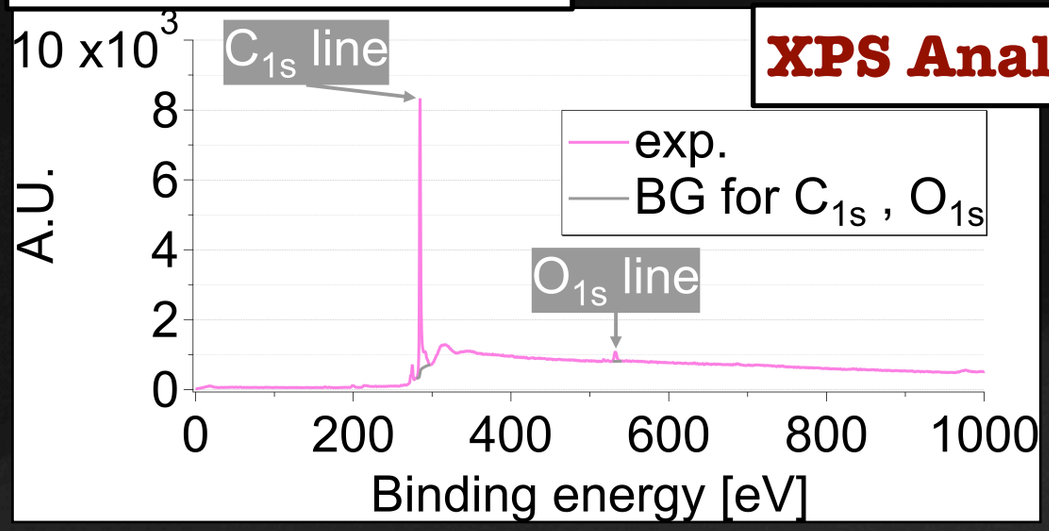


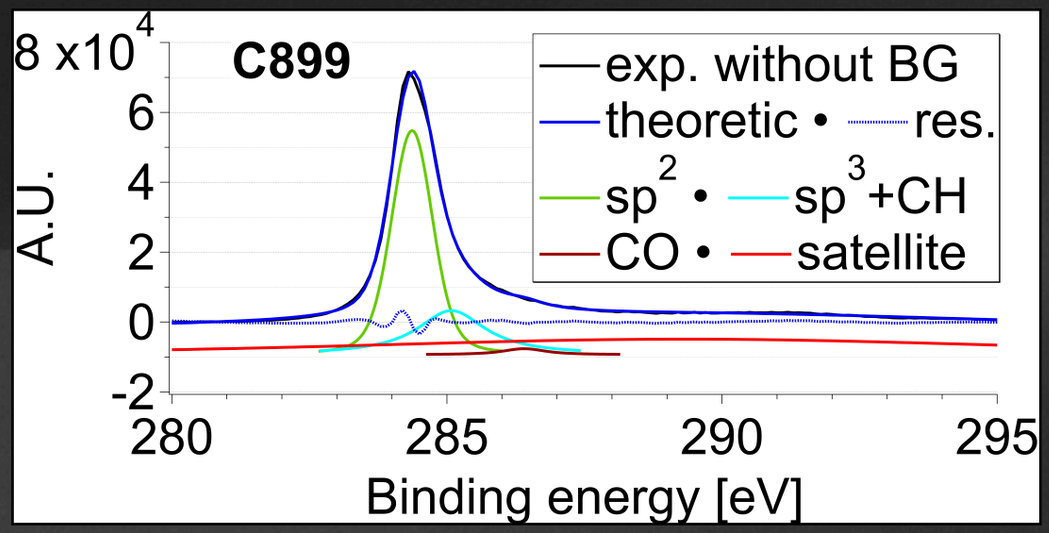
$FeCl_3$-Based Few-Layer Graphene Intercalation Compounds: Single Linear Dispersion Electronic Band Structure and Strong Charge Transfer Doping -Da Zhan, Li Sun, Zhen Hua Ni et al

2. Identifying the best precursor for efficient intercalation

Graphite (C_{899} powder)

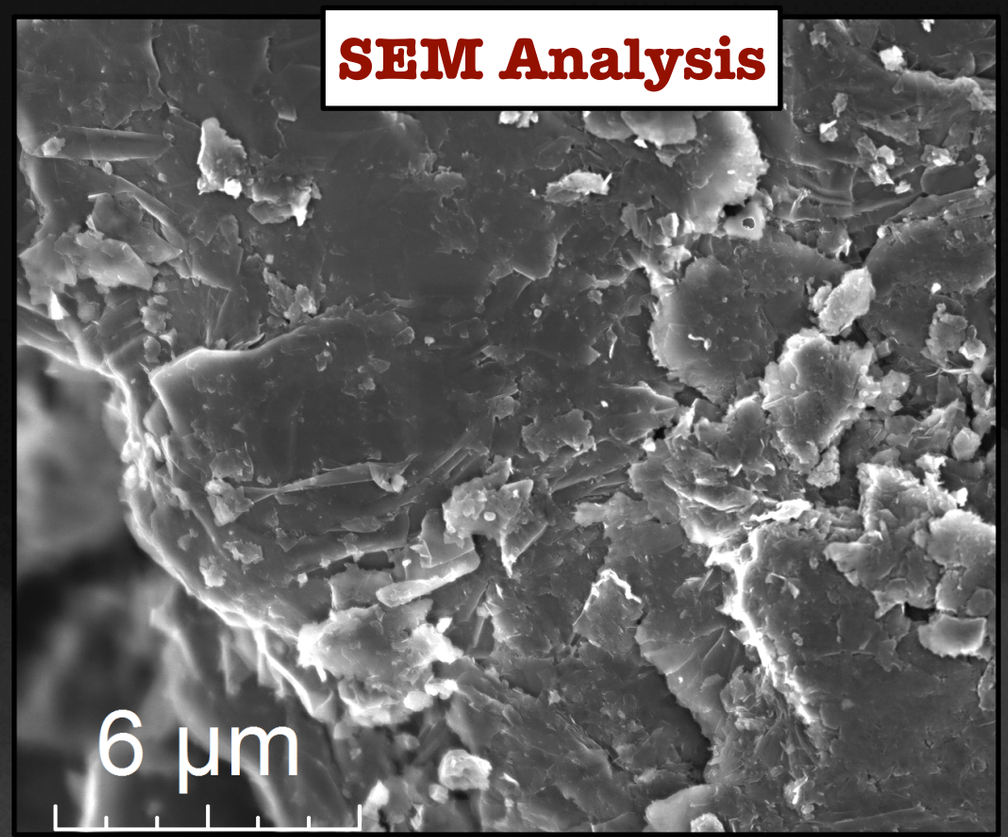
XPS Analysis



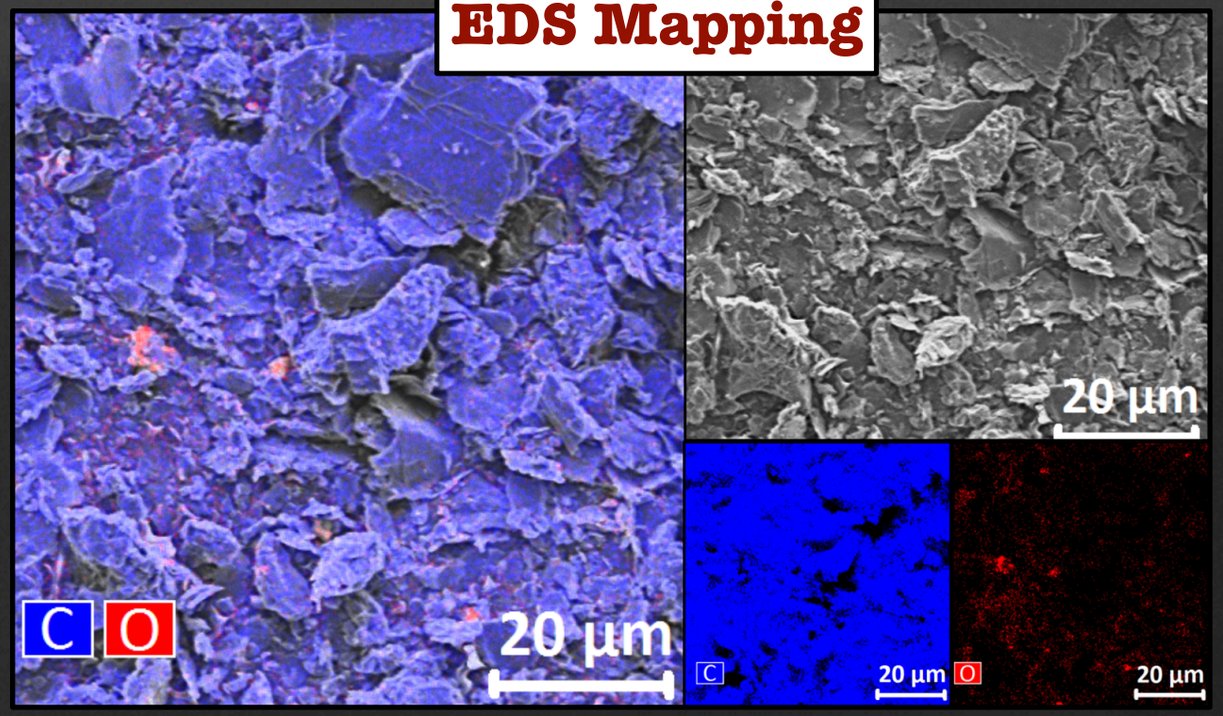


XPS analyzed sample	C_{899} powder
Survey O/(C+O)	2.1 %
C - O	1.52 %
C-C (sp^2 hybridization)	54.2%
sp^2 / (sp^3+CH)	3.26
sp^2 / (sp^2+(sp^3+CH))	76.5 %

SEM Analysis



EDS Mapping



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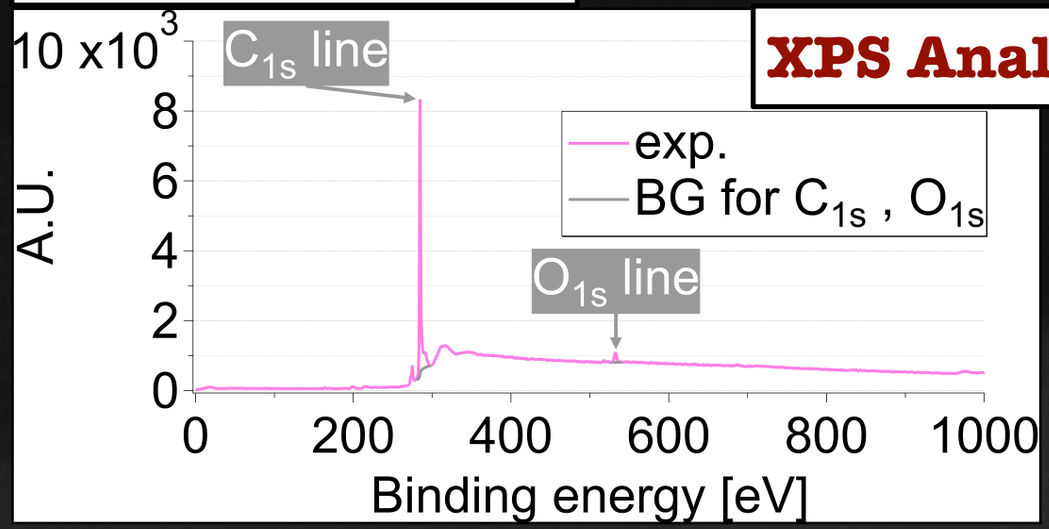

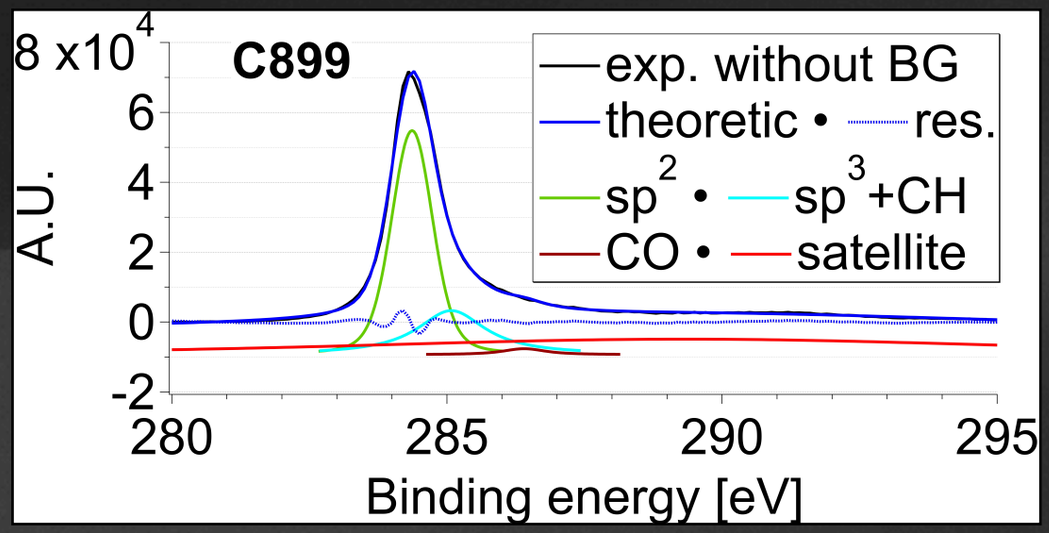

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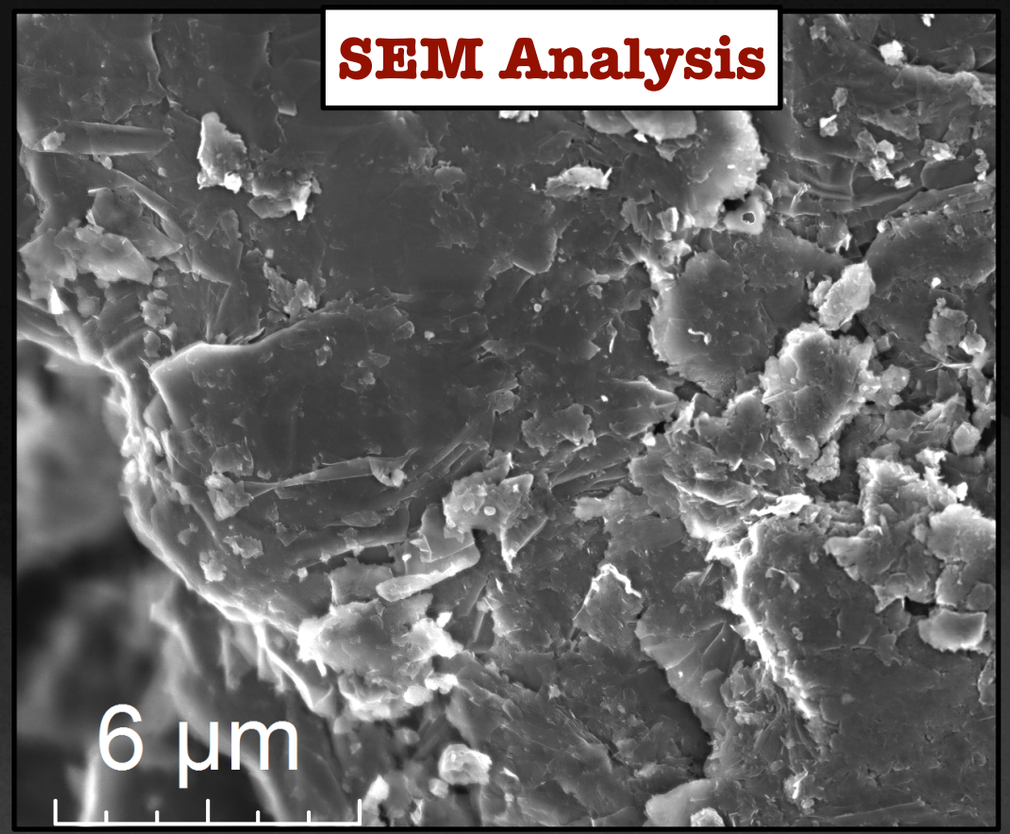

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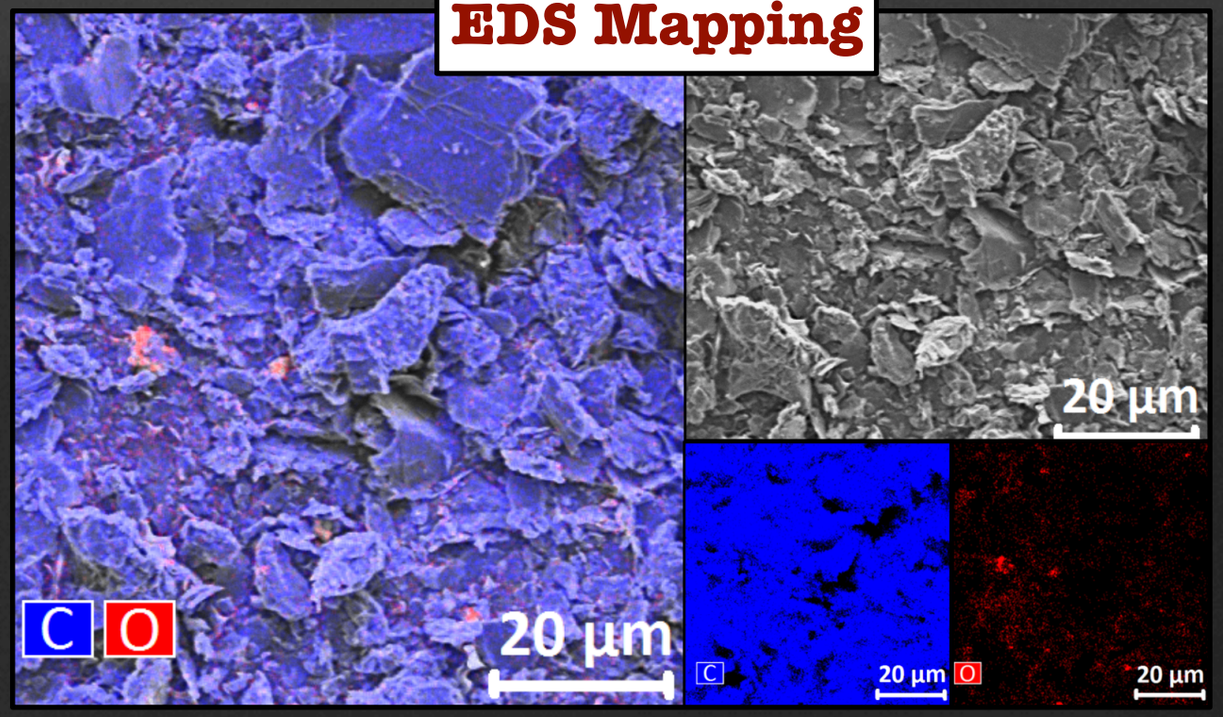

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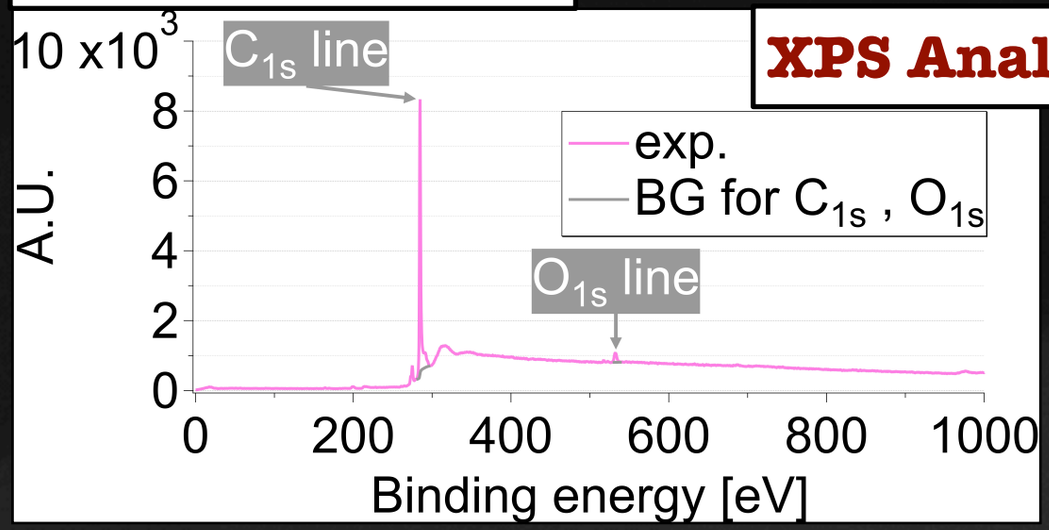

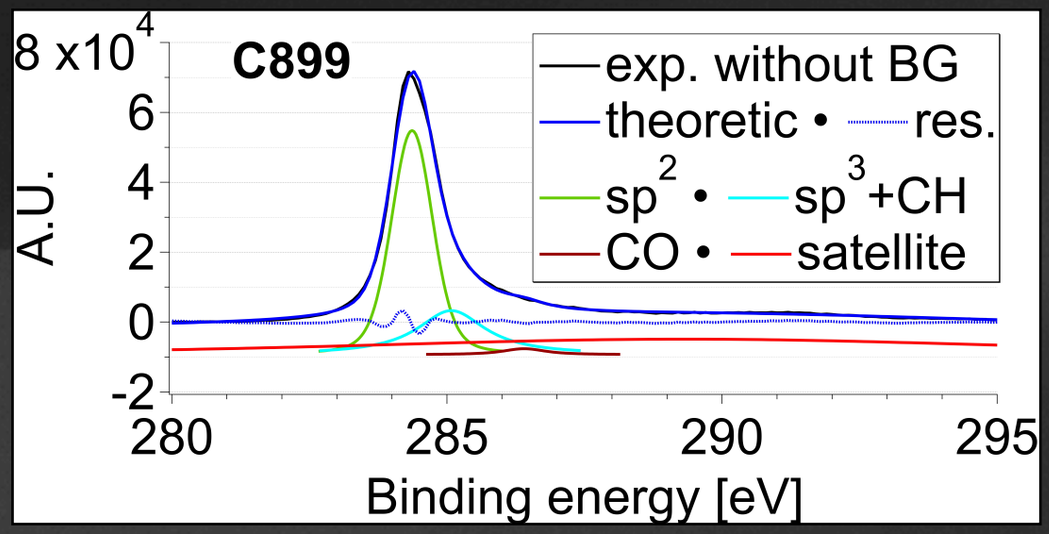

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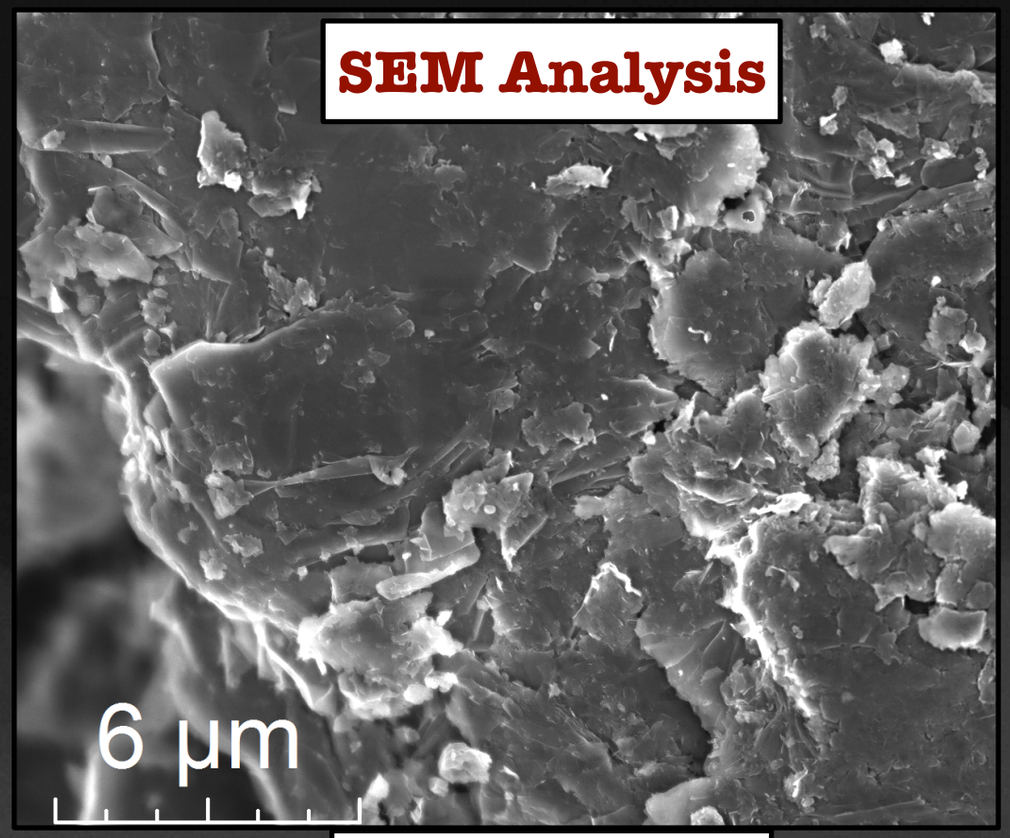
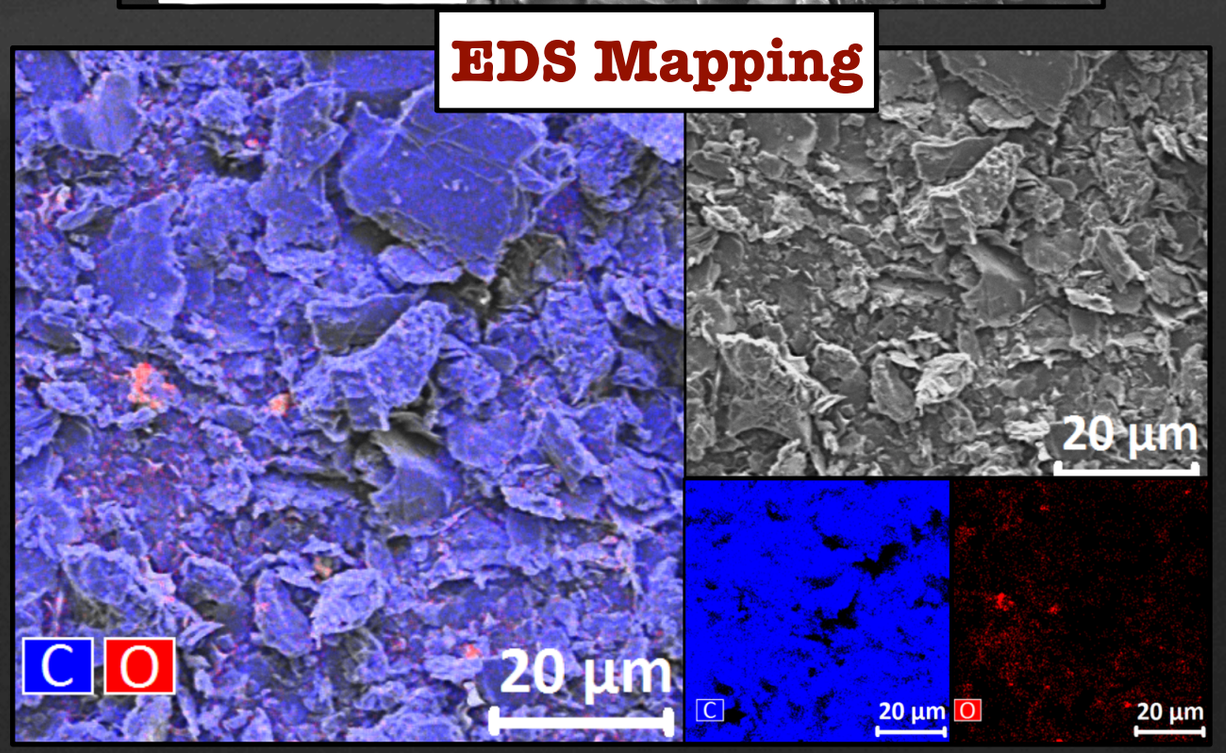
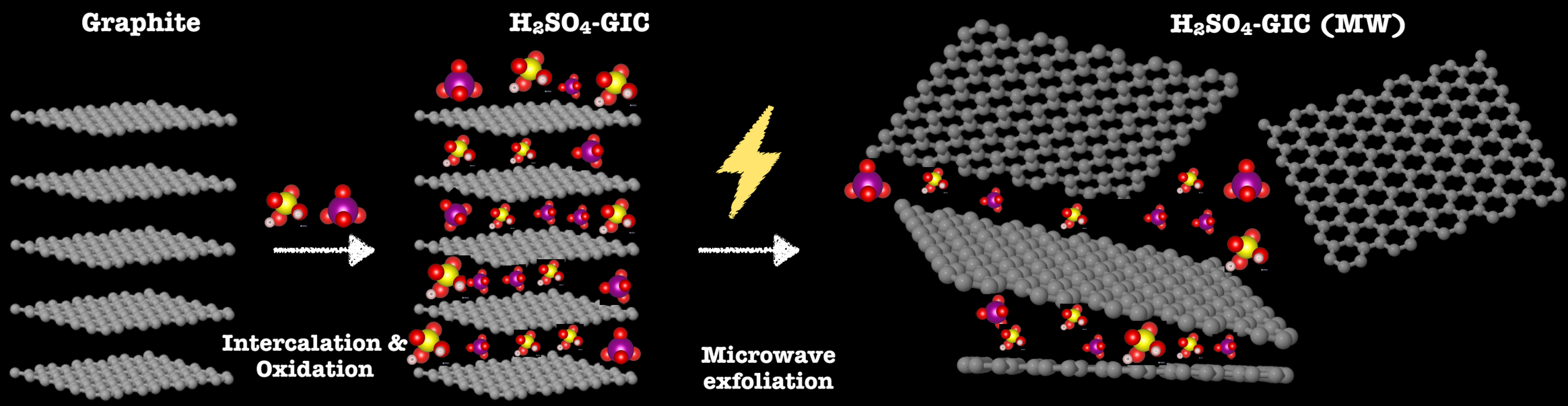




3.Chemical Synthesis of H_2SO_4-GIC



: Intercalant agent (H_2SO_4)

: Oxidizing agent (MnO_4^-)

Intercalation & Oxidation Processes

- 5 g Graphite powder (300 µm large flakes)
- 12.5 ml H_2O (DI) + 50 ml H_2SO_4 (75-98%)
- 2.5 g $KMnO_4$

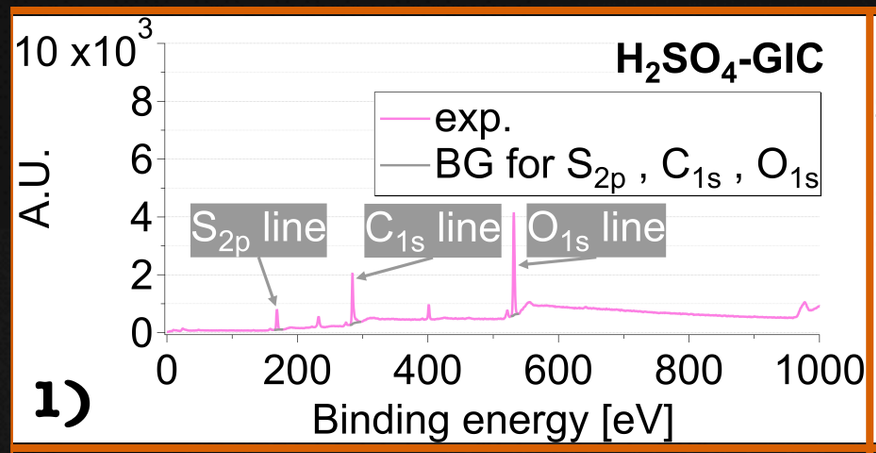
Microwave exfoliation

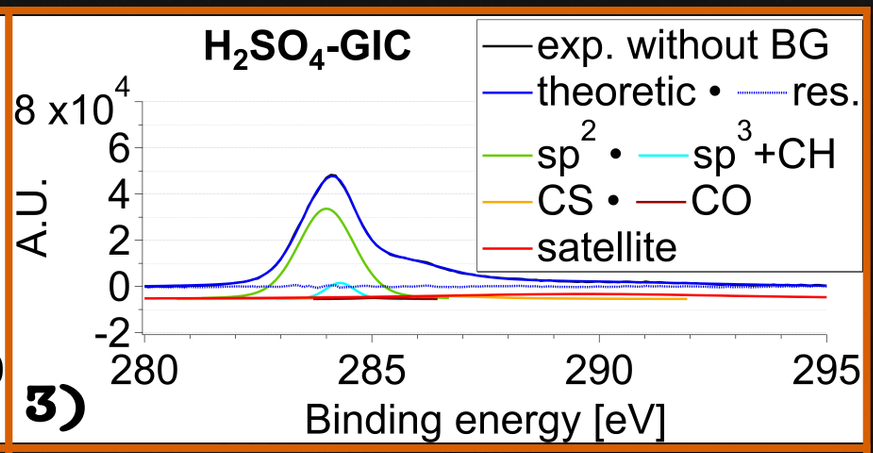
- Power: 800 W
- Frequency: 2.45 GHz
- Time: <10 s

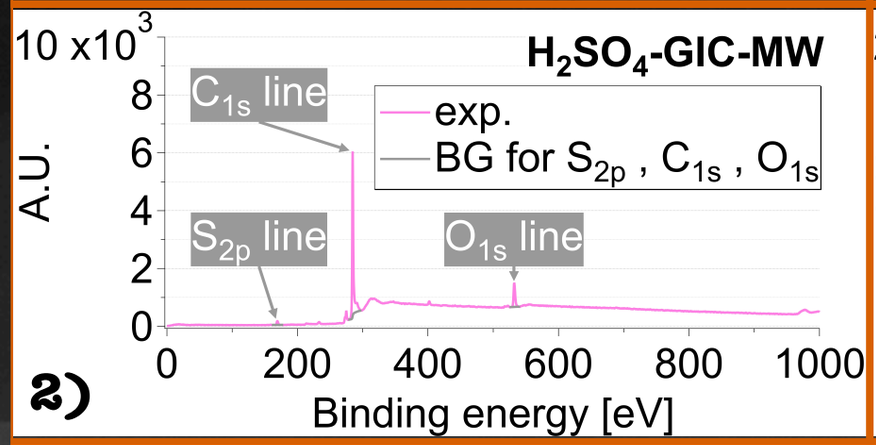
Tests and measurements

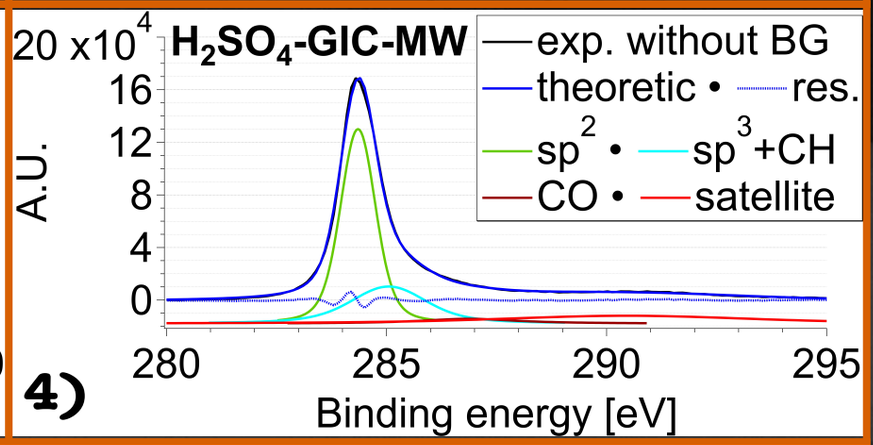
- XPS
- SEM/EDS
- AFM

4. Characterization of H_2SO_4-GIC









H_2SO_4-GIC / H_2SO_4-GIC (MW)

XPS Analysis

XPS analyzed sample	H_2SO_4 – GIC	H_2SO_4 – GIC (MW)
Survey O/(C+O)	39.39 %	6.43 %
C - O	19.7%	6.3%
C-C (sp^2 hybridization)	55.2%	54.9%
(sp^3+CH)	5.3%	18.9%
C-S	4.2%	< 1%
π-π*	15.6%	19.8%

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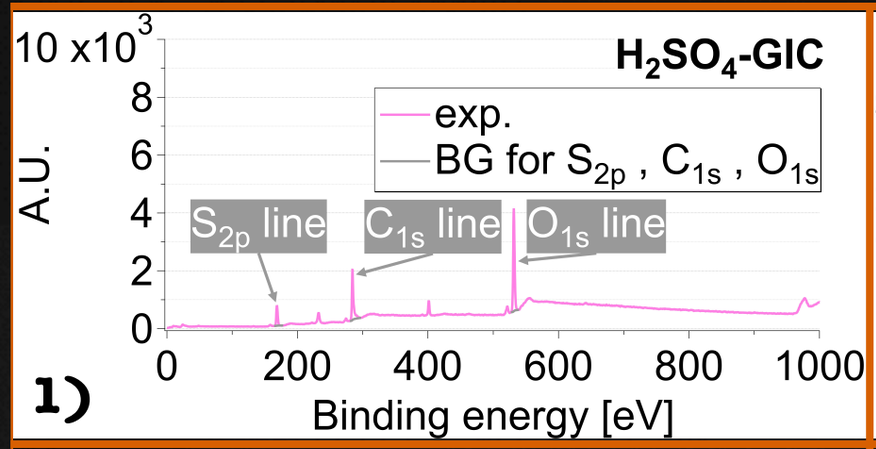

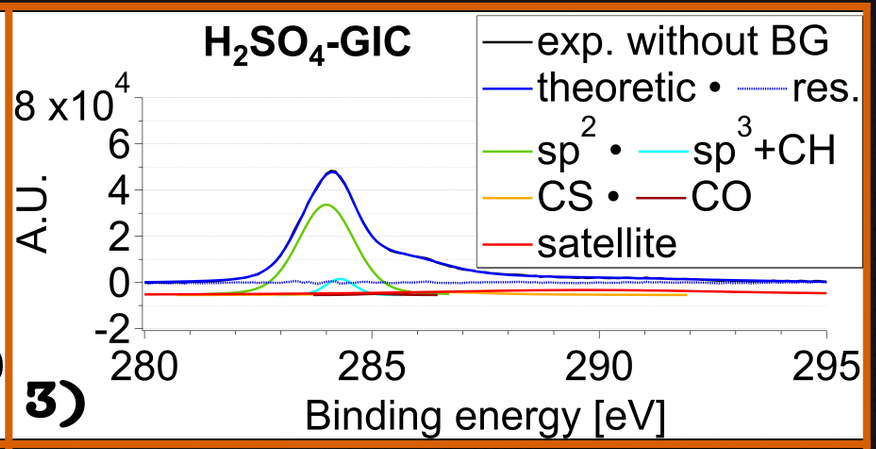

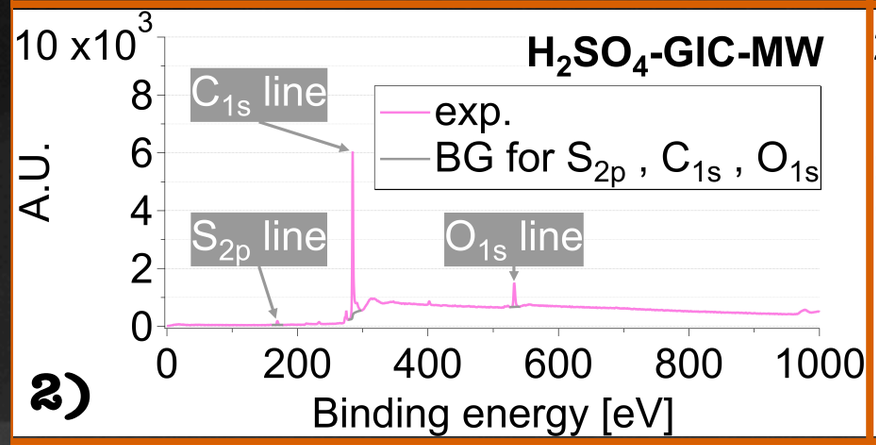

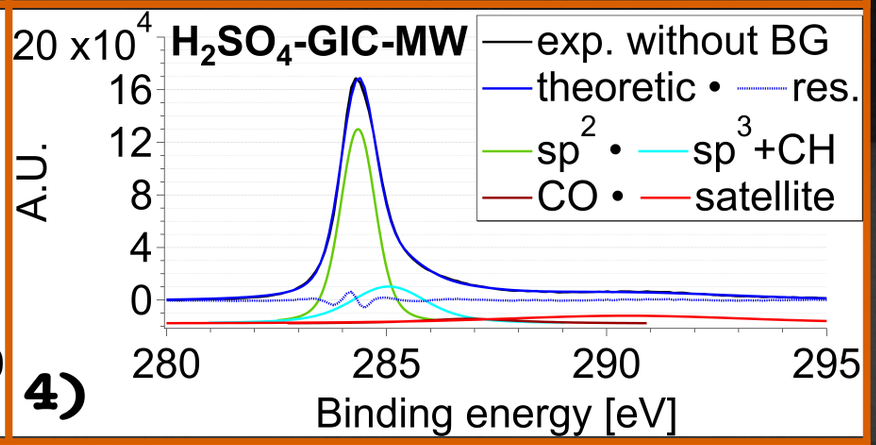

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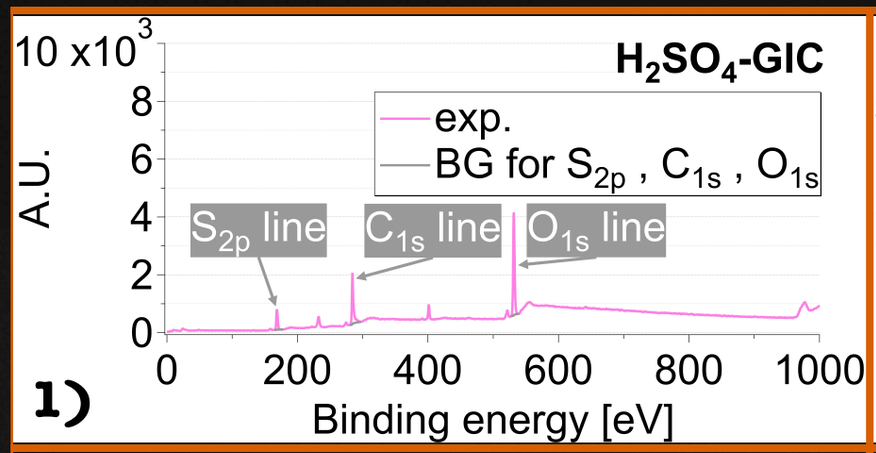

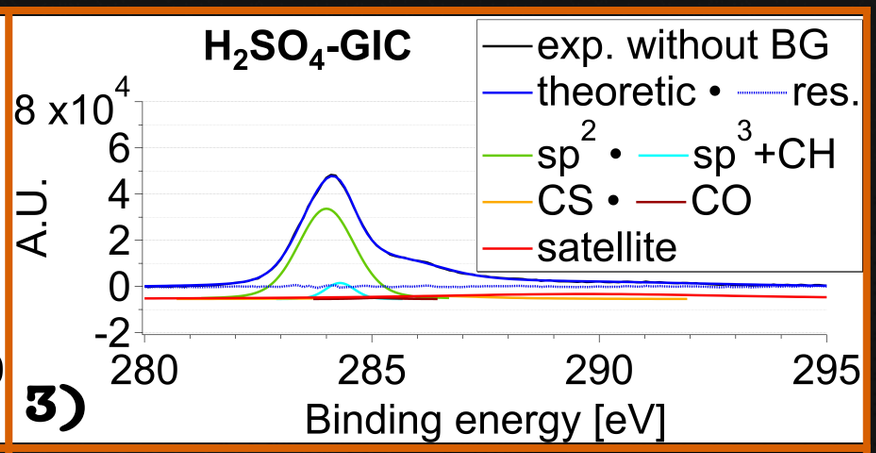

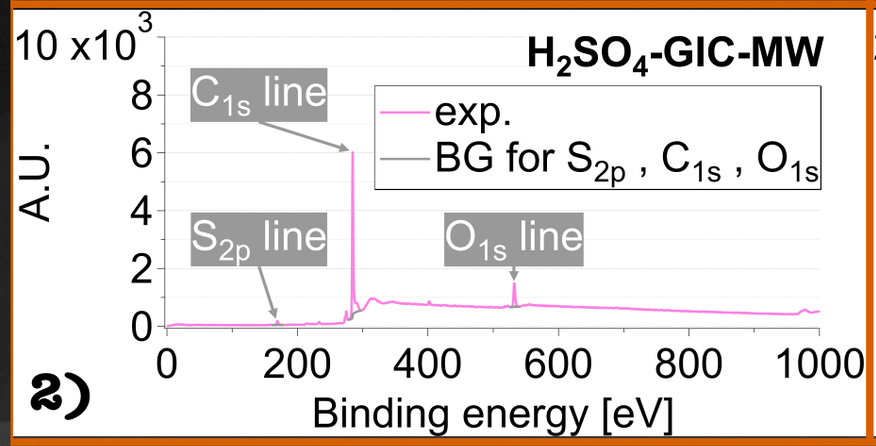

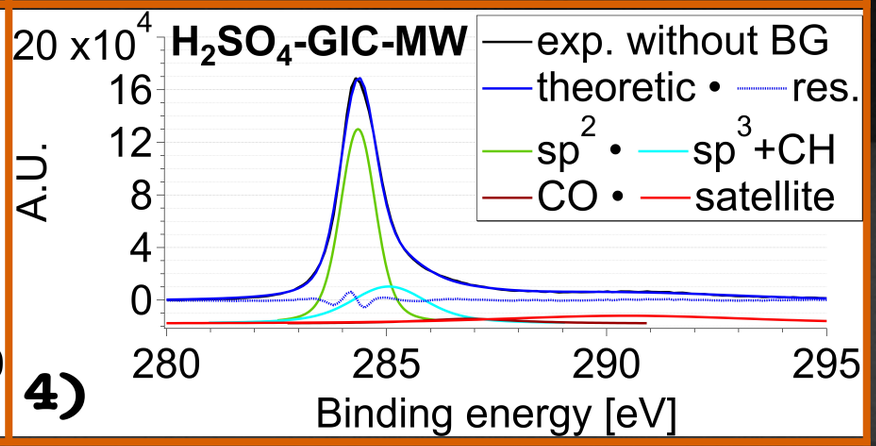

H_2SO_4-GIC /H_2SO_4-GIC (MW)

XPS Analysis

H_2SO_4-GIC (MW)

sp^2 / (sp^2+(sp^3+CH)) = 75.3%

~

Graphite (C_{899} powder)

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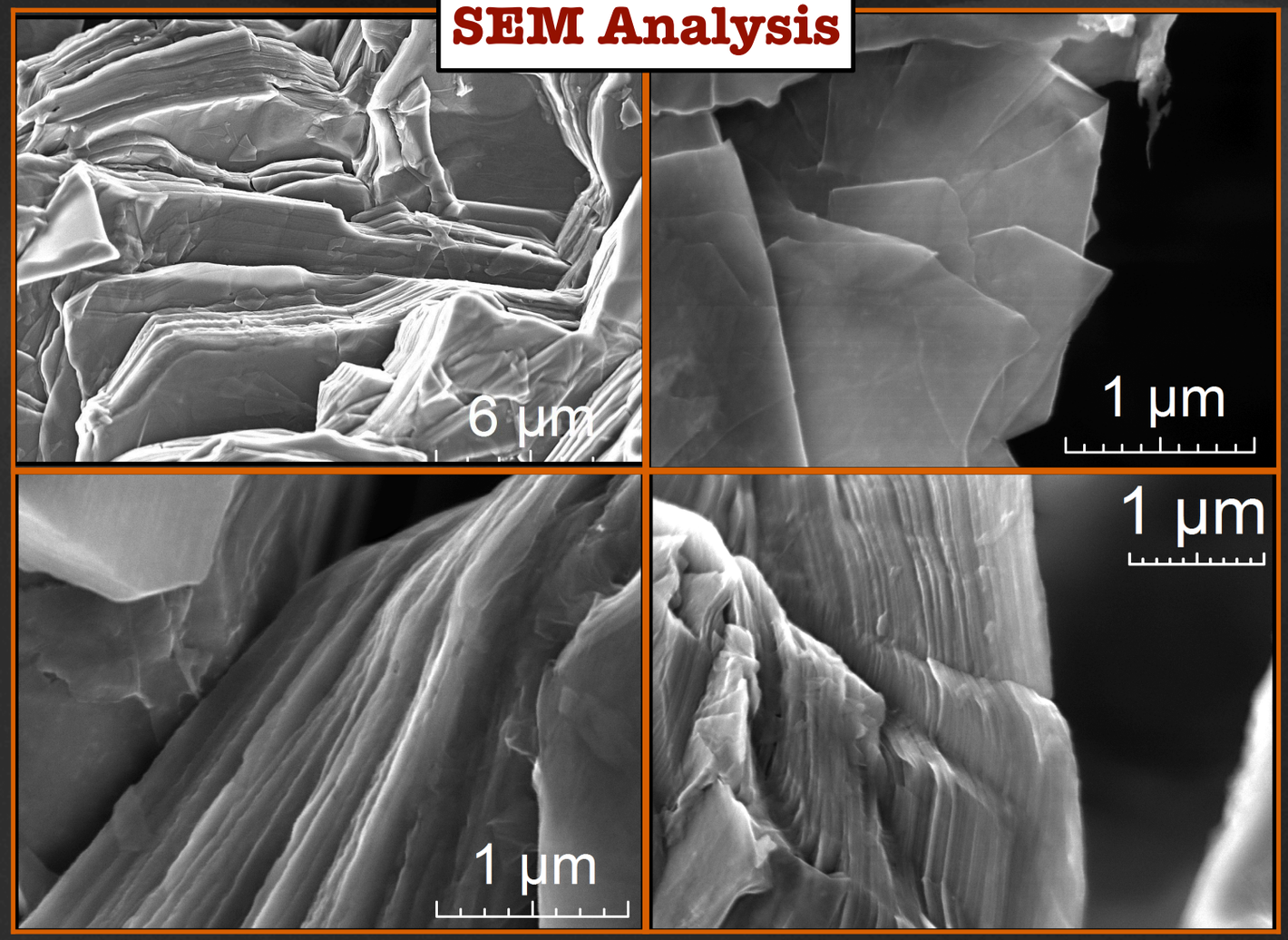
4. Characterization of H_2SO_4-GIC

H_2SO_4-GIC
- after chemical synthesis -

- Much rougher basal plane
- Sharp and delaminated edges
- Very good ordering of the H_2SO_4-GIC layering
- Many areas with clear boundaries between them (graphite intercalation stages)

SEM Analysis



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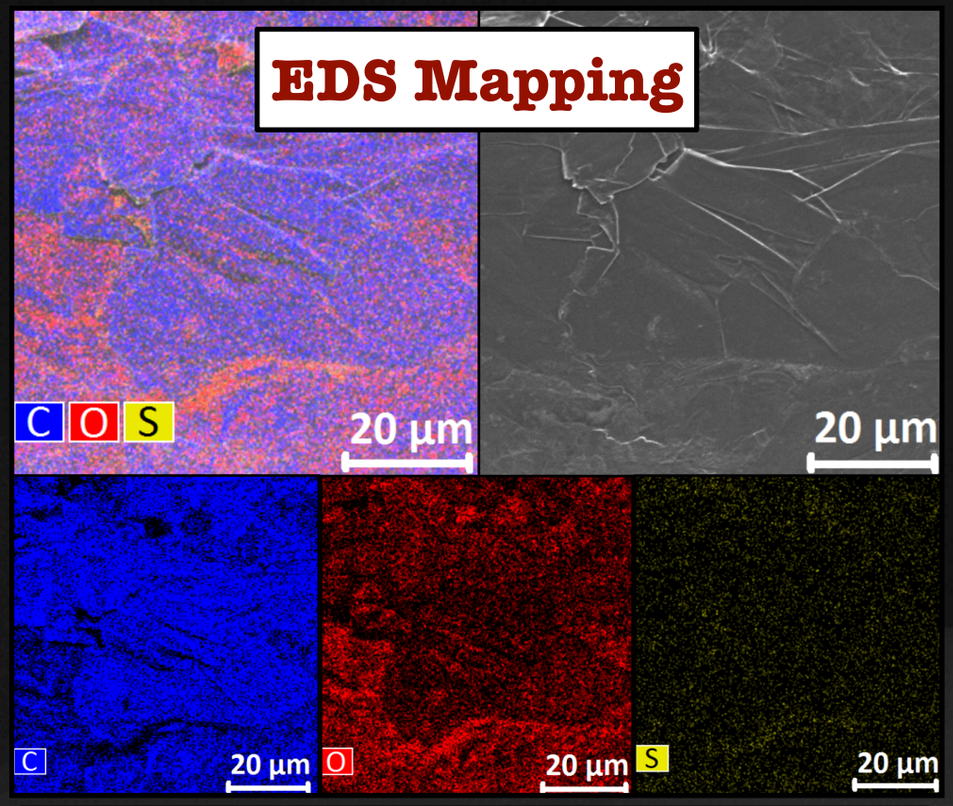
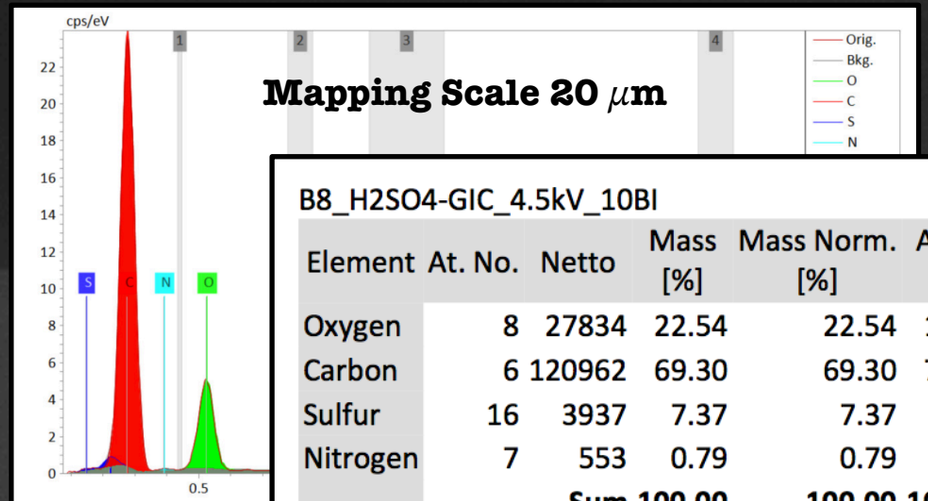


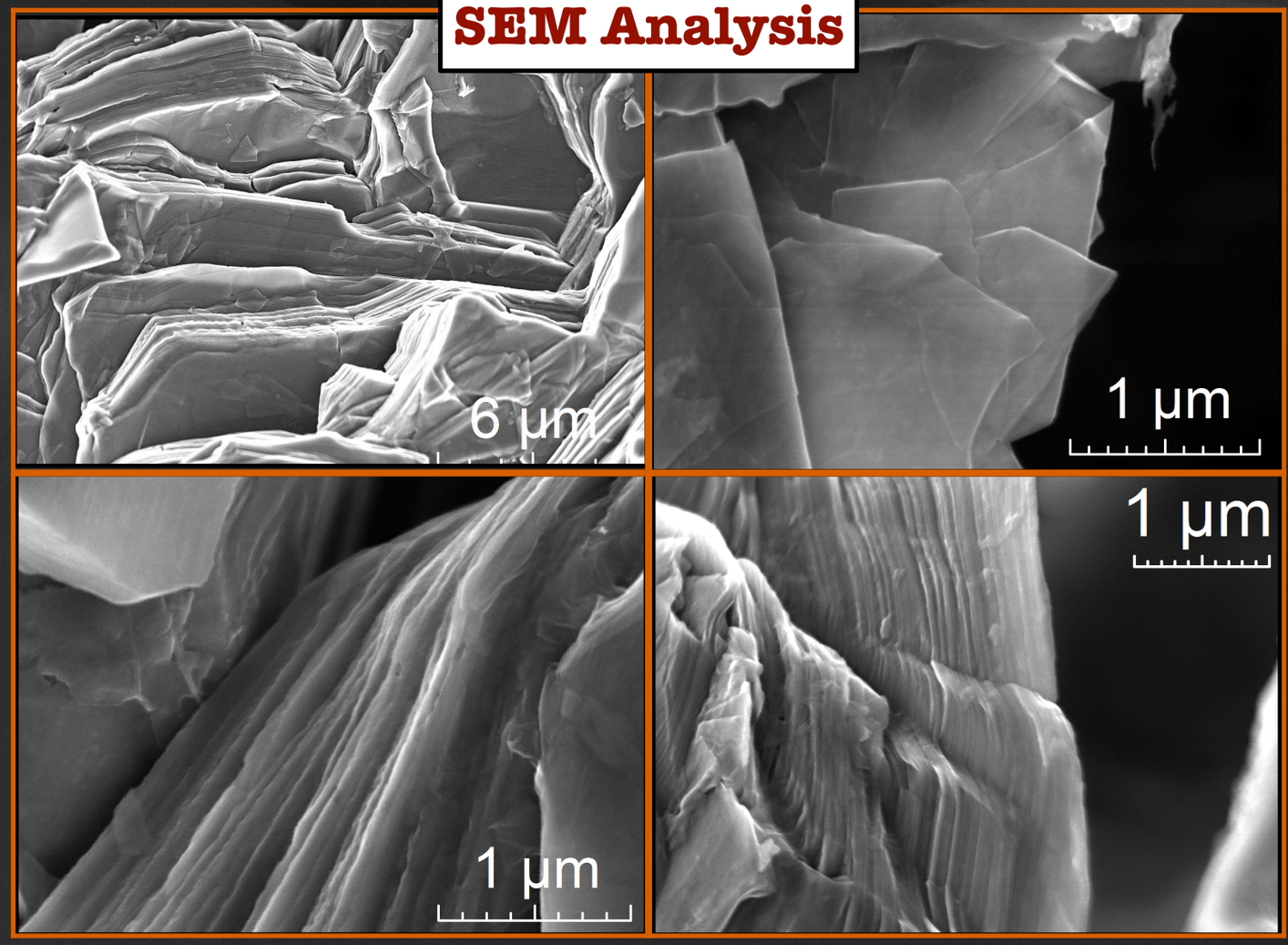


B8_H2SO4-GIC_4.5kV_10BI

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]
Oxygen	8	27834	22.54	22.54	18.87
Carbon	6	120962	69.30	69.30	77.30
Sulfur	16	3937	7.37	7.37	3.08
Nitrogen	7	553	0.79	0.79	0.75
		Sum	**100.00**	**100.00**	**100.00**

- High amount of oxygen (~19%) - formation of functional groups after use of oxidising agent
- Amount of sulphur (~ 3.08 at. %) - a sign of the use of intercalants in chemical synthesis
- No contaminants were observed
- Homogeneous elemental distribution - uniform distributed intercalation within the sample

SEM Analysis

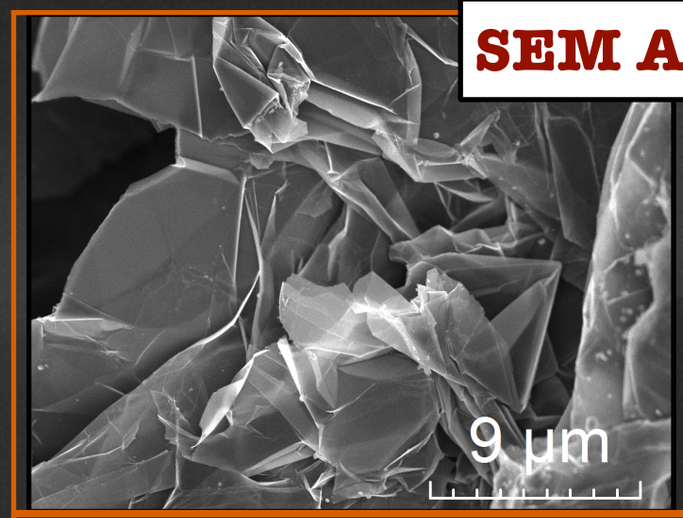


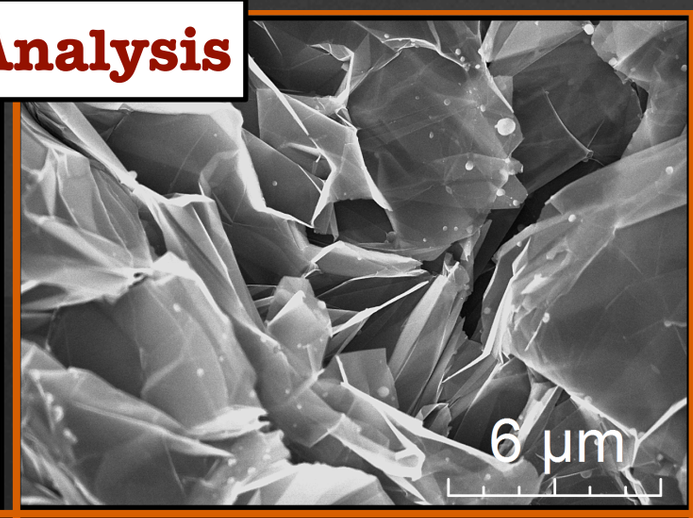
4. Characterization of H_2SO_4-GIC

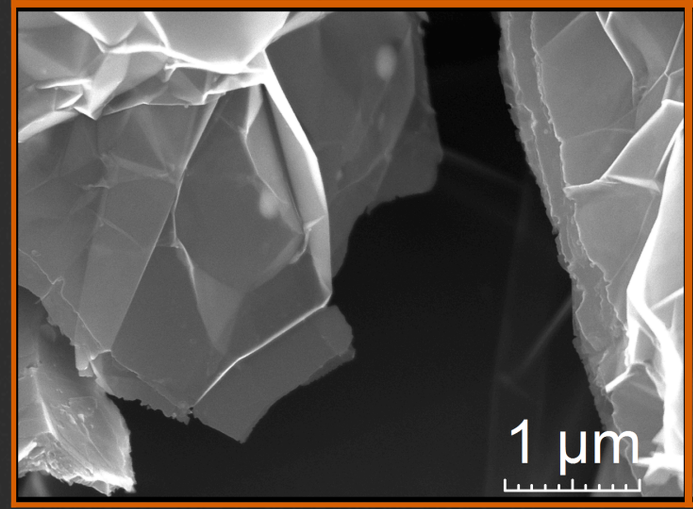
H_2SO_4-GIC (MW)
- after exfoliation process -

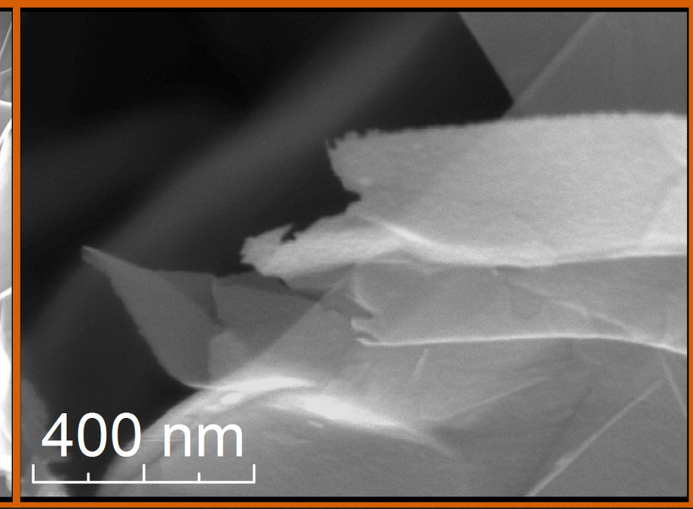
- Some large lateral dimensions (several microns)
- Many areas of easily exfoliated graphene
- The disappearance of the layered and well-organized structure of the H_2SO_4-GIC
- A larger specific surface area
- A larger expansion volume - a very slightly low density appearance
- Almost transparent - good exfoliation behaviour of H_2SO_4 - GIC - presence of few layers of graphene

SEM Analysis









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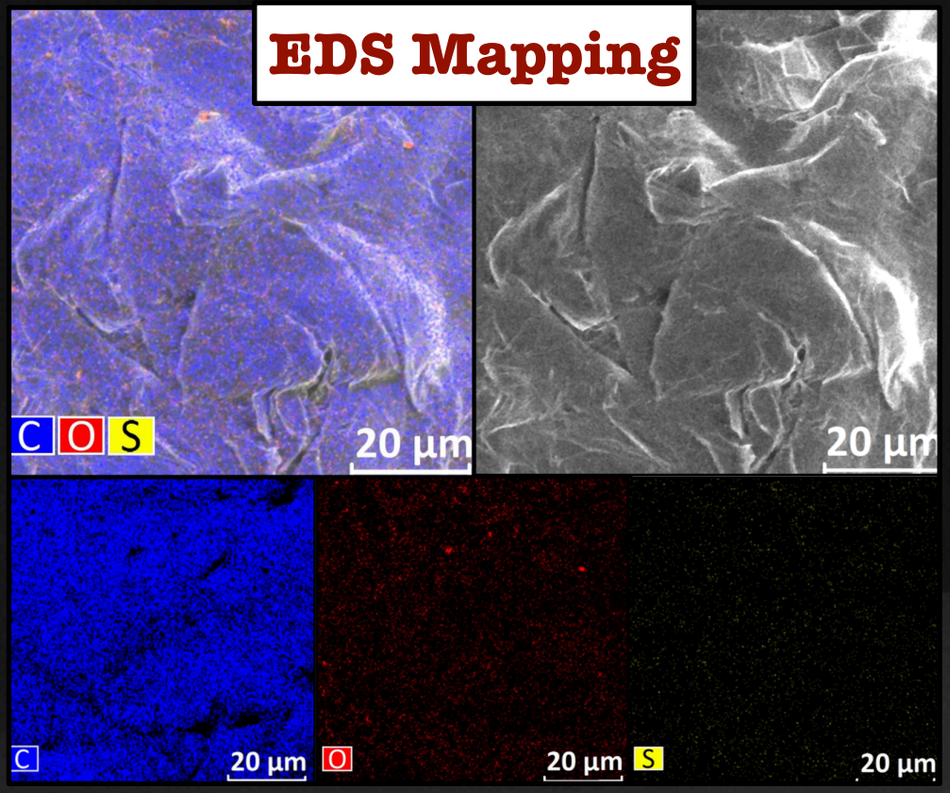

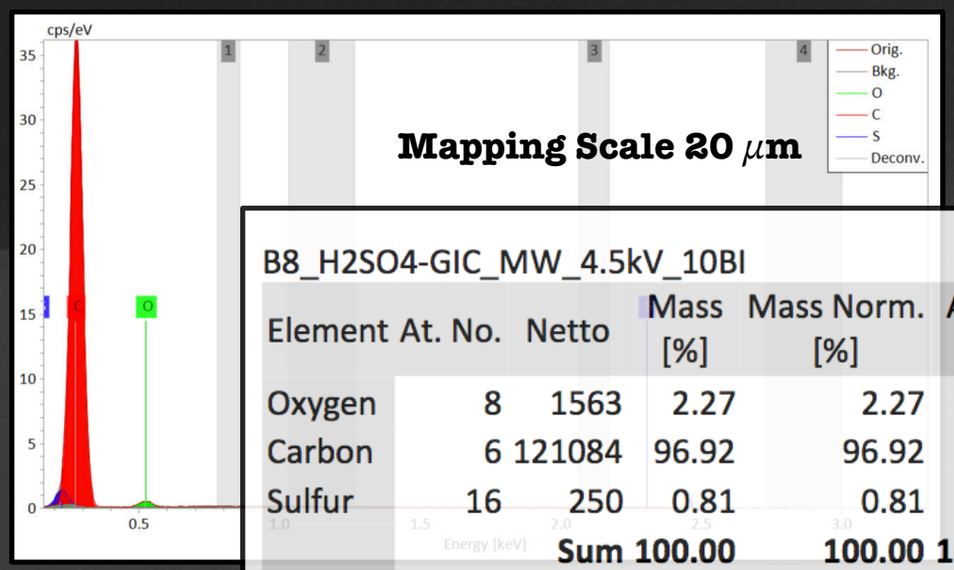

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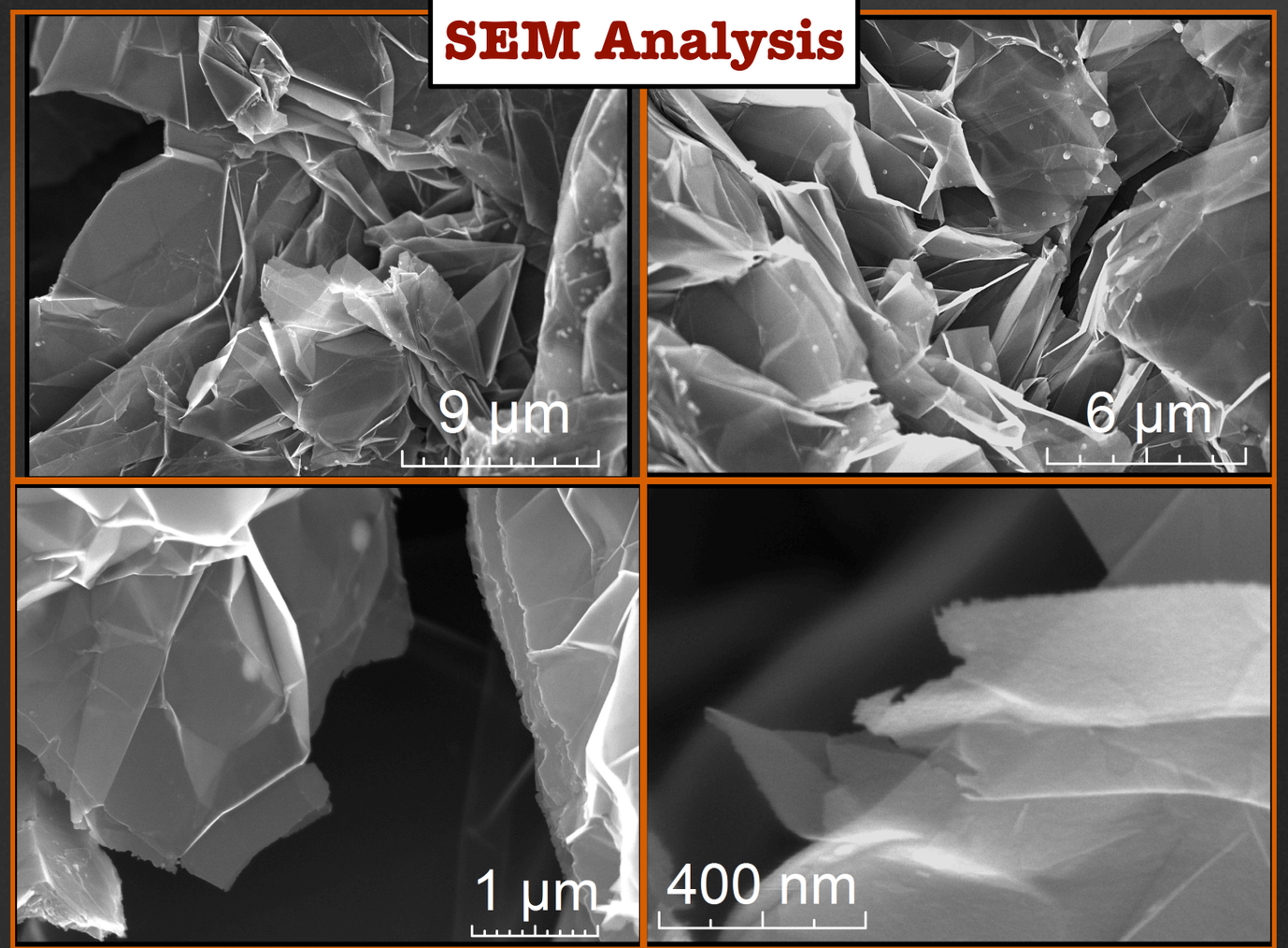
Oxygen	8	1563	2.27	2.27	1.72
Carbon	6	121084	96.92	96.92	97.97
Sulfur	16	250	0.81	0.81	0.31
		Sum	100.00	100.00	100.00

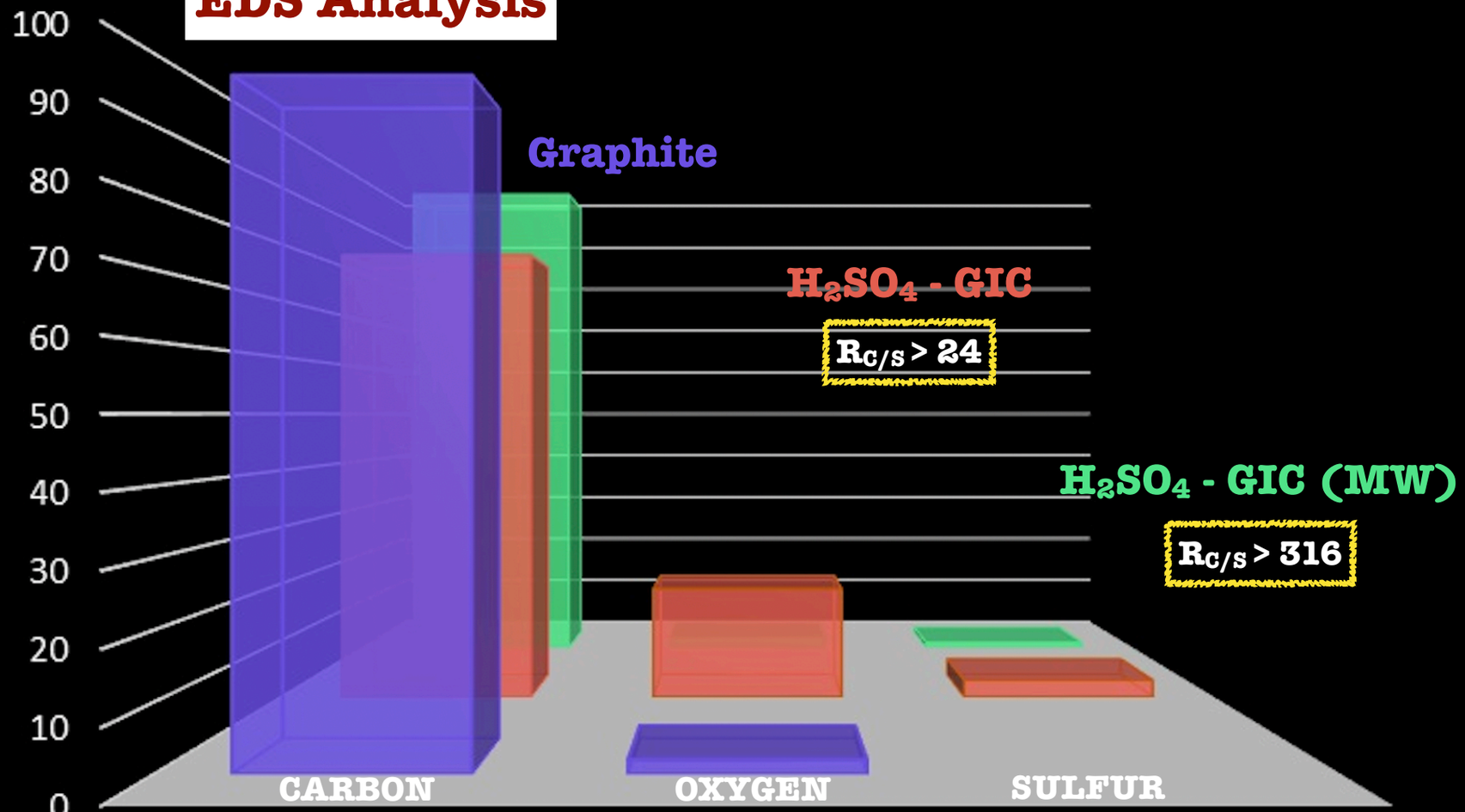
- Very clean sample (terms of purity) exhibiting carbon (~ 97 at.%), drastically decrease in oxygen concentration (~ 2 at.%), the lack of sulfur (~ ±1 at.%)

SEM Analysis

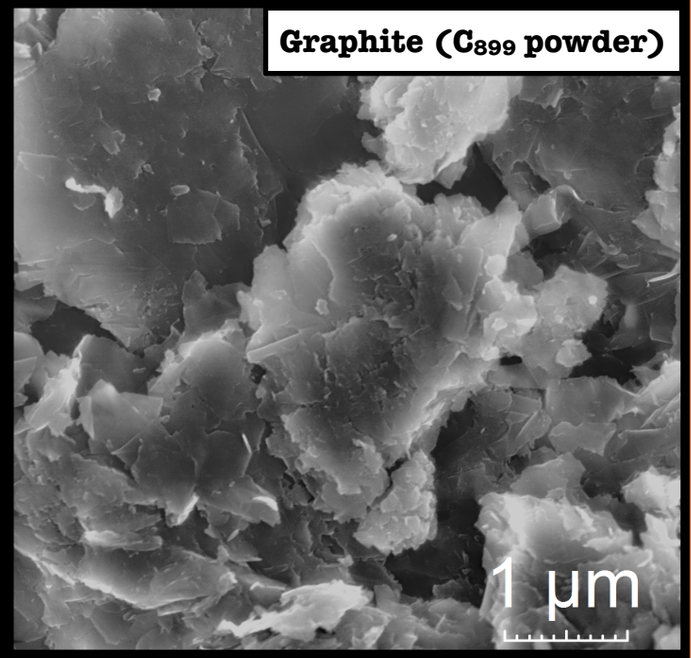


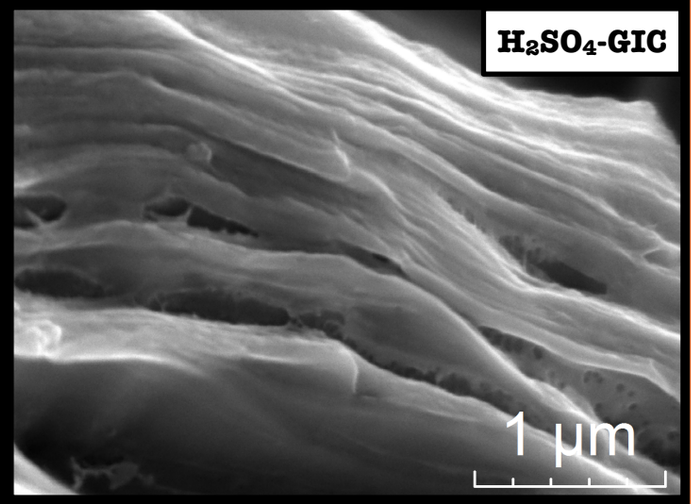
4. Characterization of H_2SO_4-GIC

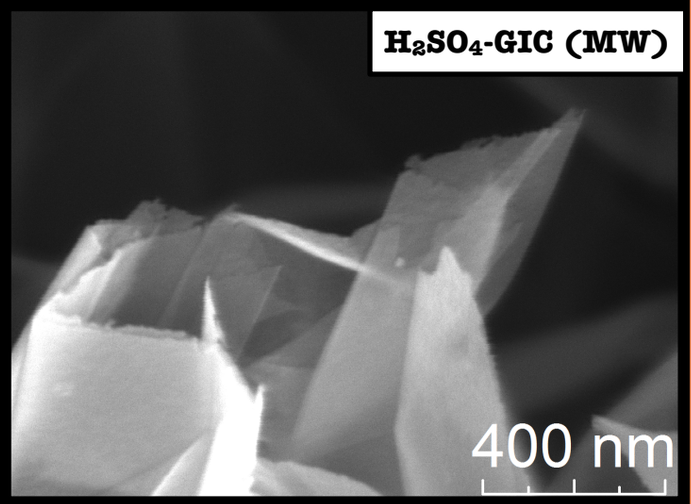
EDS Analysis



SEM Analysis

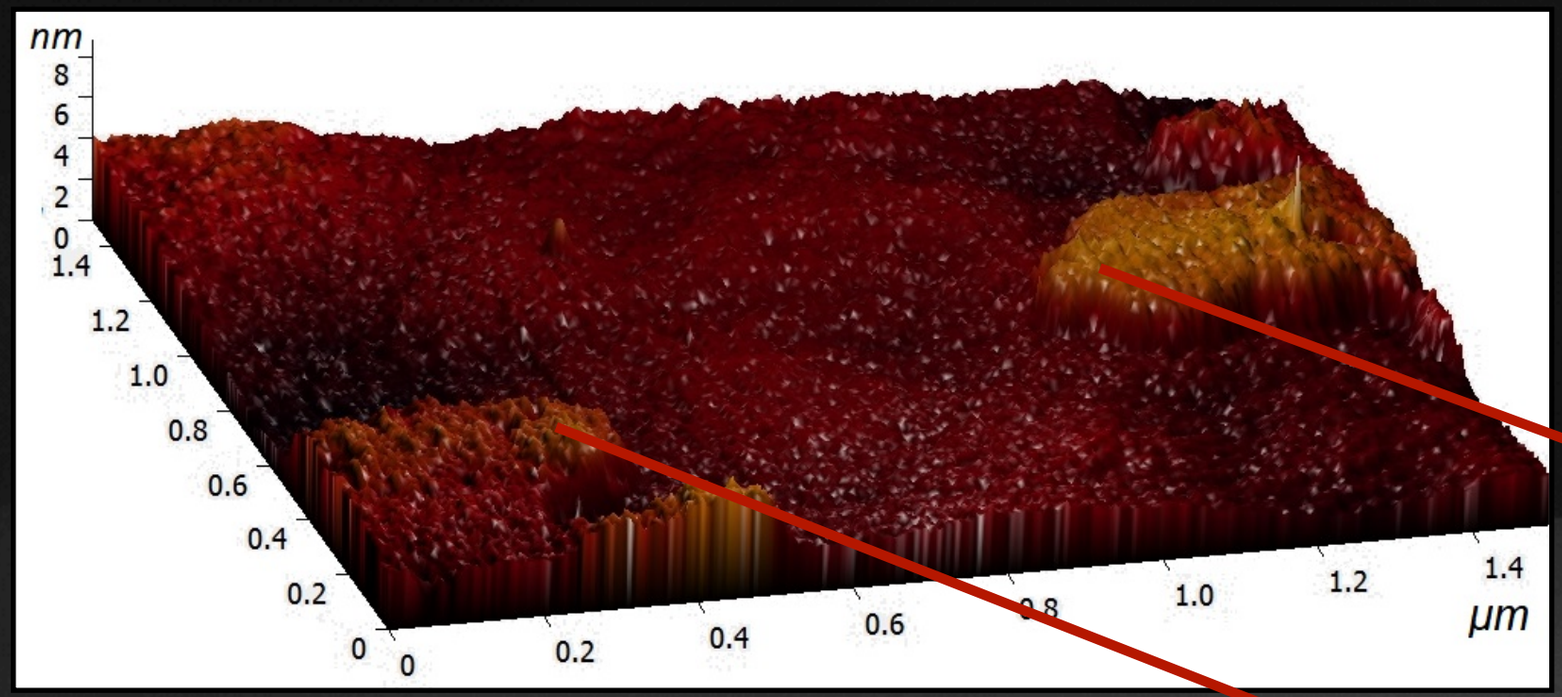


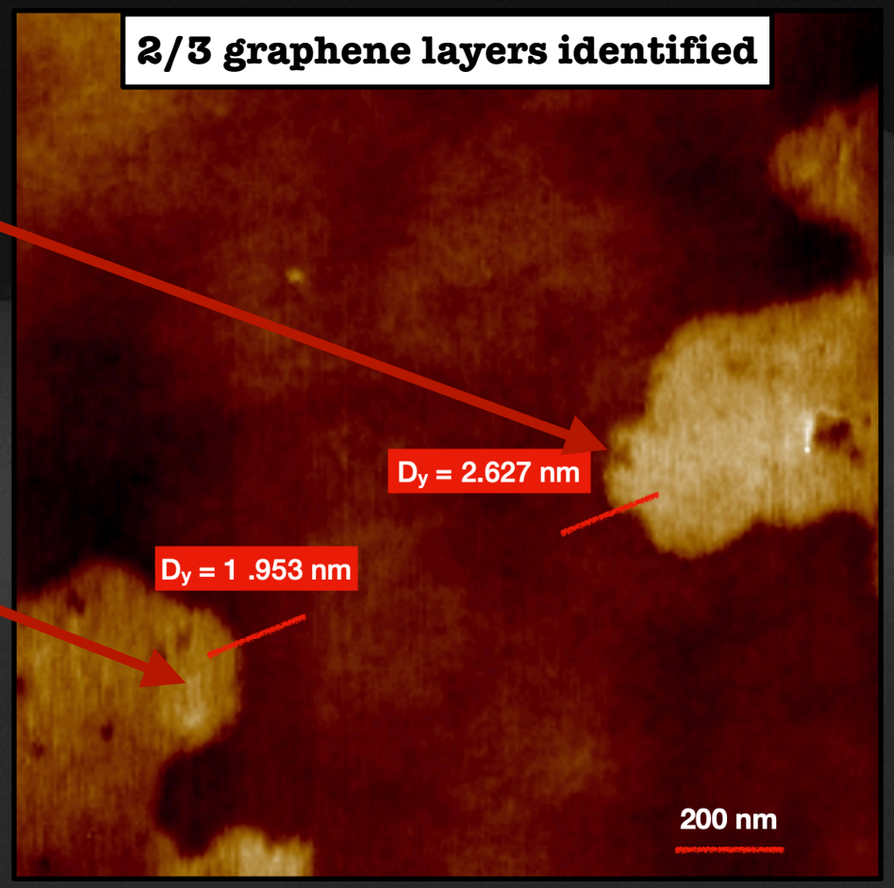




4. Characterization of H_2SO_4-GIC

AFM Analysis





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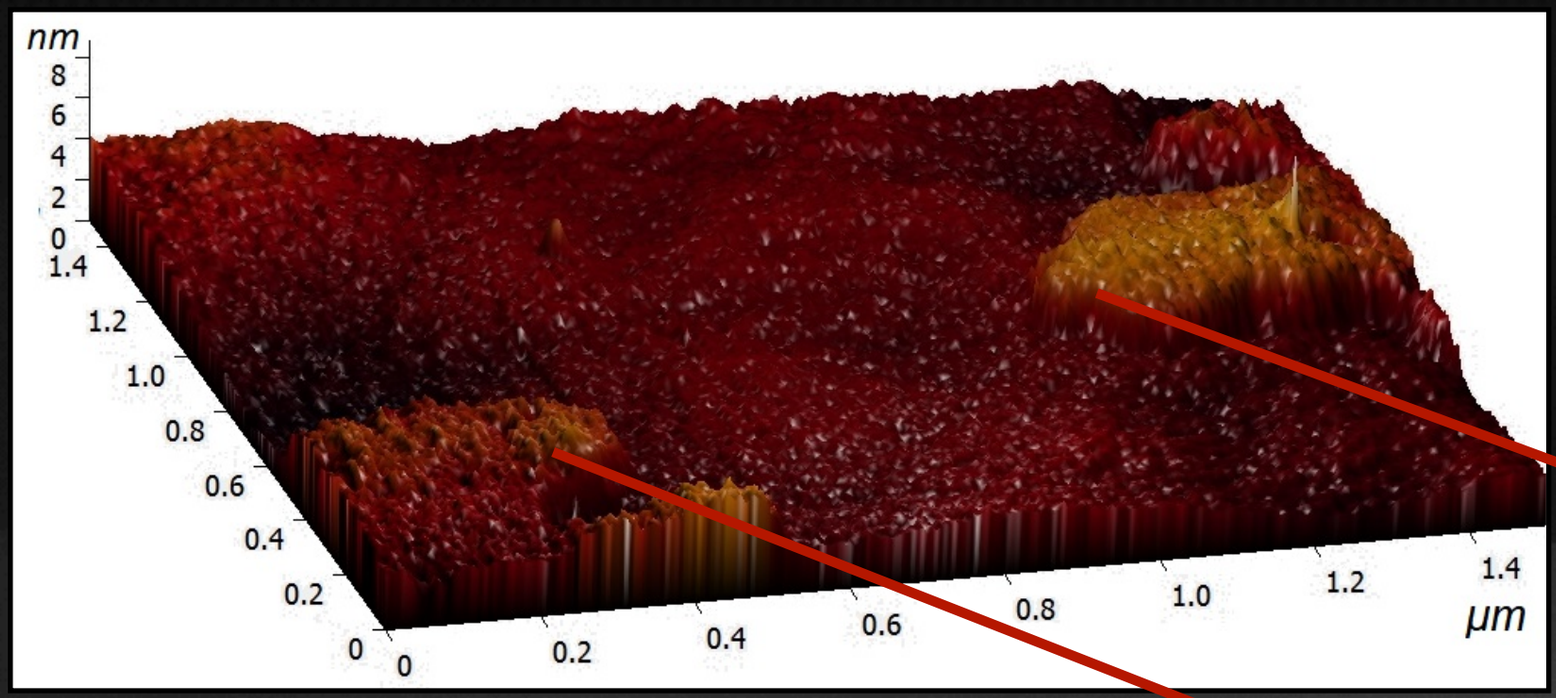
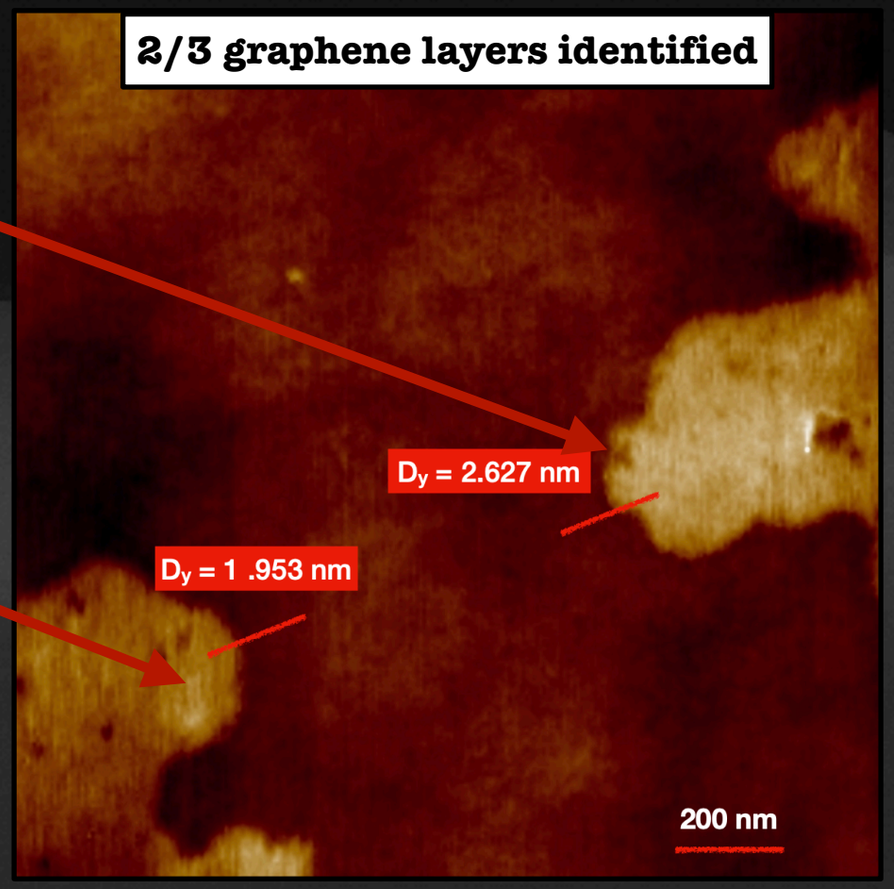
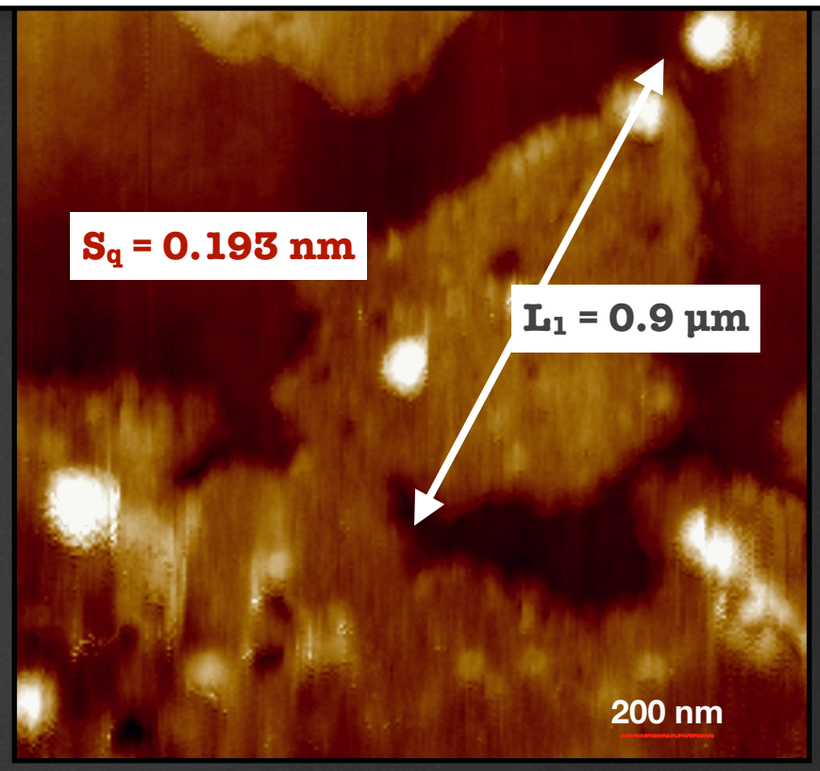
AFM Analysis

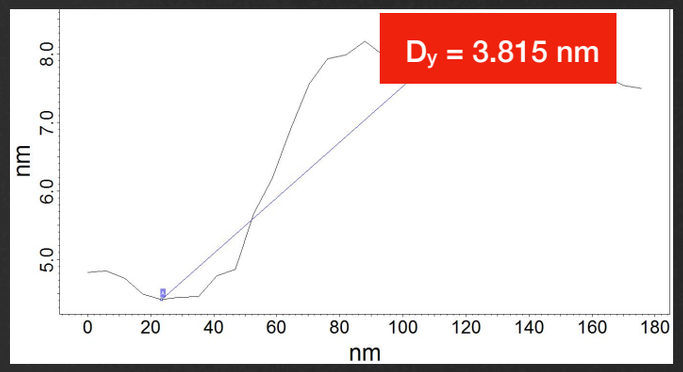


2/3 graphene layers identified



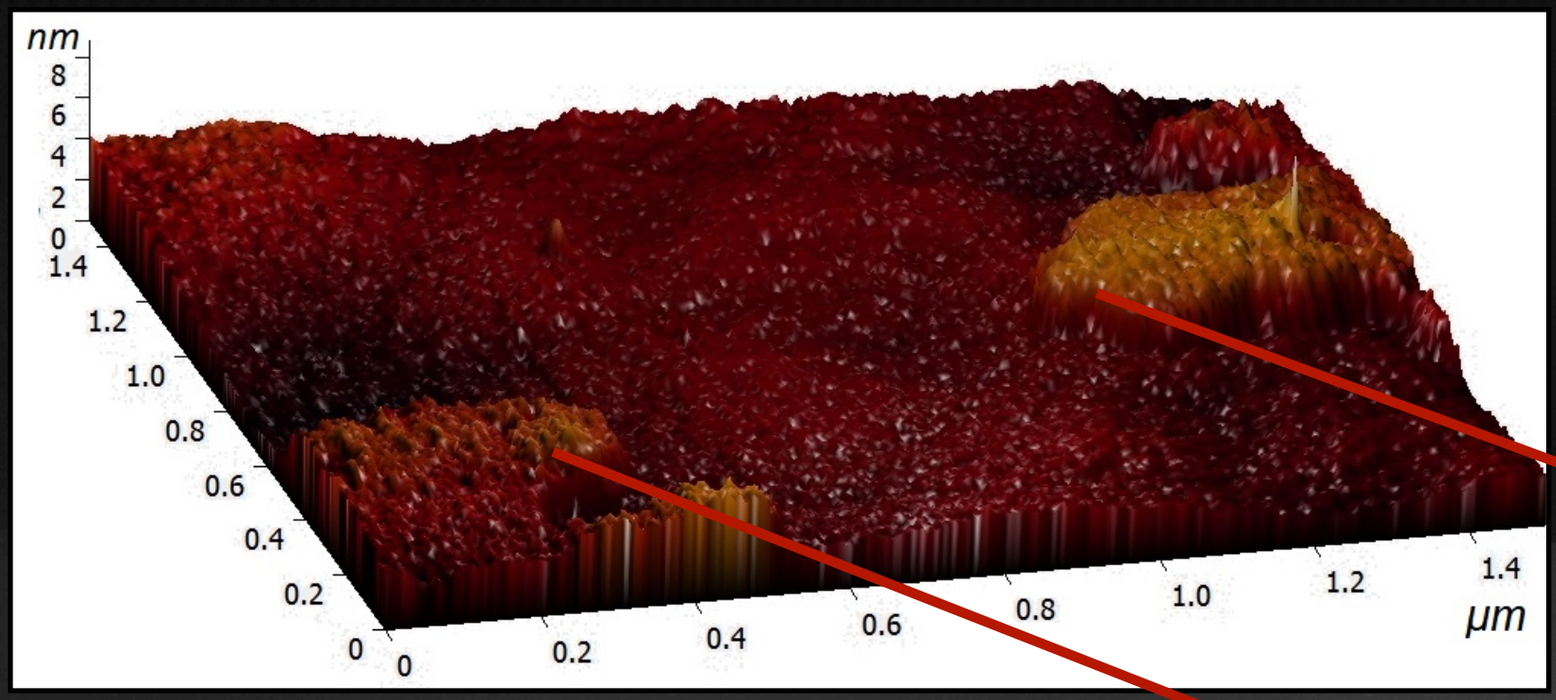
Lateral stretch and roughness of few (4) graphene layers



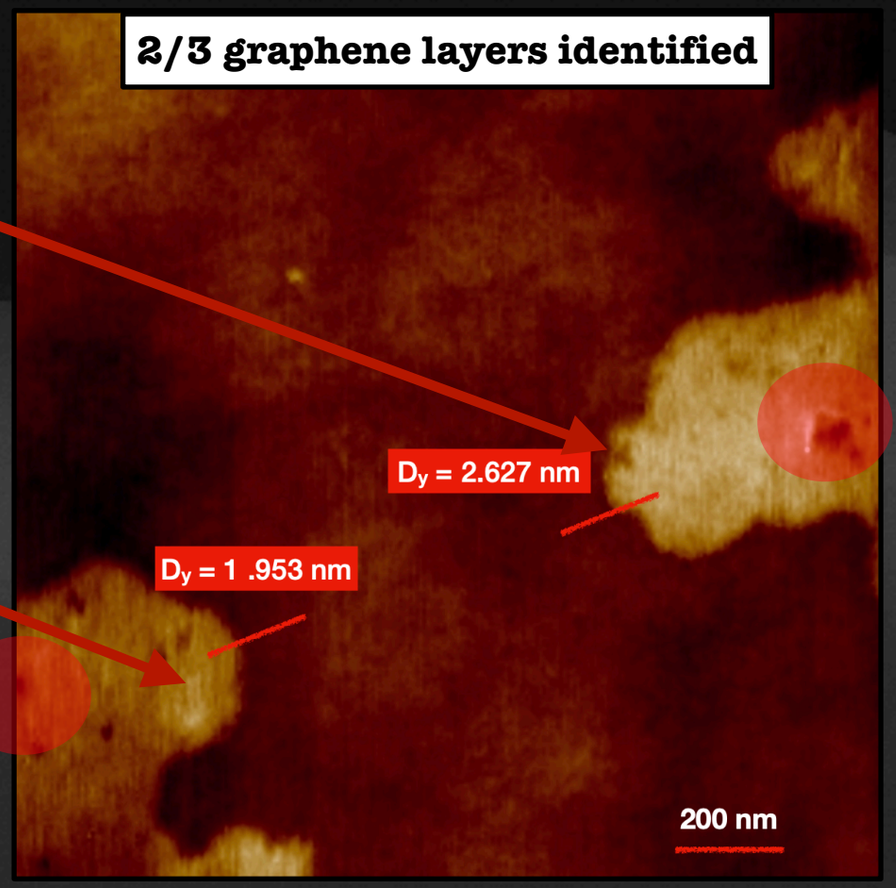


4. Characterization of H_2SO_4-GIC

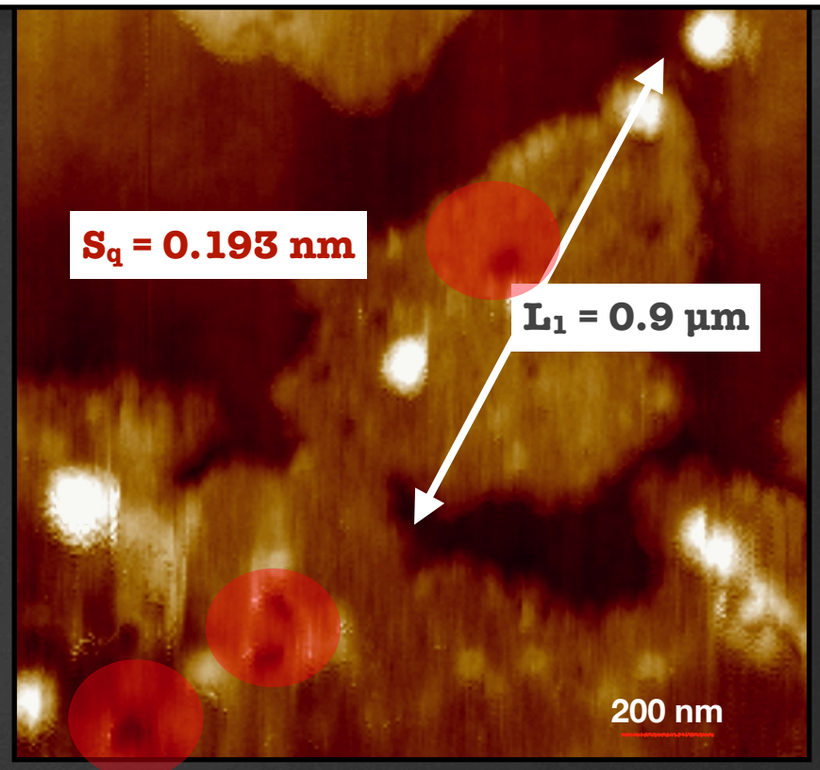
AFM Analysis



2/3 graphene layers identified

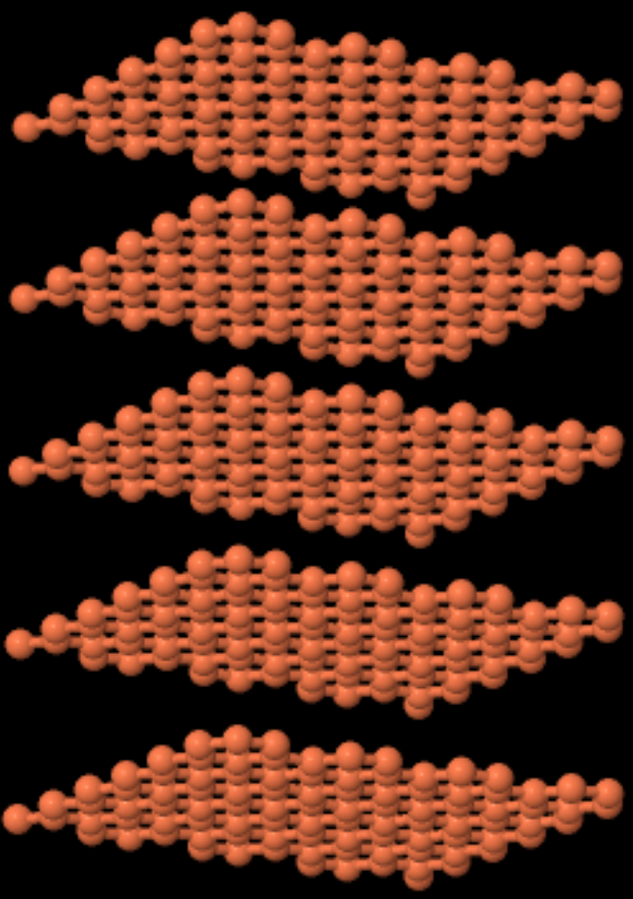


Lateral stretch and roughness of few(4) graphene layers



XPS analyzed sample	H_2SO_4 – GIC	H_2SO_4 – GIC (MW)
(sp^3+CH)	5.3%	18.9%

5. Conclusions



- A less common method of graphite intercalation, exfoliation, was implemented in a one-step synthesis of graphite intercalated compounds using H_2SO_4 as an intercalant, $KMnO_4$ as an oxidizing agent and finally a microwave irradiation treatment

- In this process, all operations were carried out under ambient conditions, which offers the main advantage of safer and simpler working conditions suitable for large-scale production

- SEM images confirming the morphology of the graphene sheets obtained after microwave treatment as almost transparent

- AFM images come and further highlight that individual graphene sheets with thicknesses between 1.9 and 2.6 nm have been identified, claimed to be double/triple layers

- Through careful planning, these graphene layers could be used in the future as Carbon Targets for High-Power Laser Acceleration Experiments

ELI-NP Young Scientist and Young Engineer Days

Thank You for Your Attention!