Characterization of 3D graphene and graphene/metal oxide composite networks

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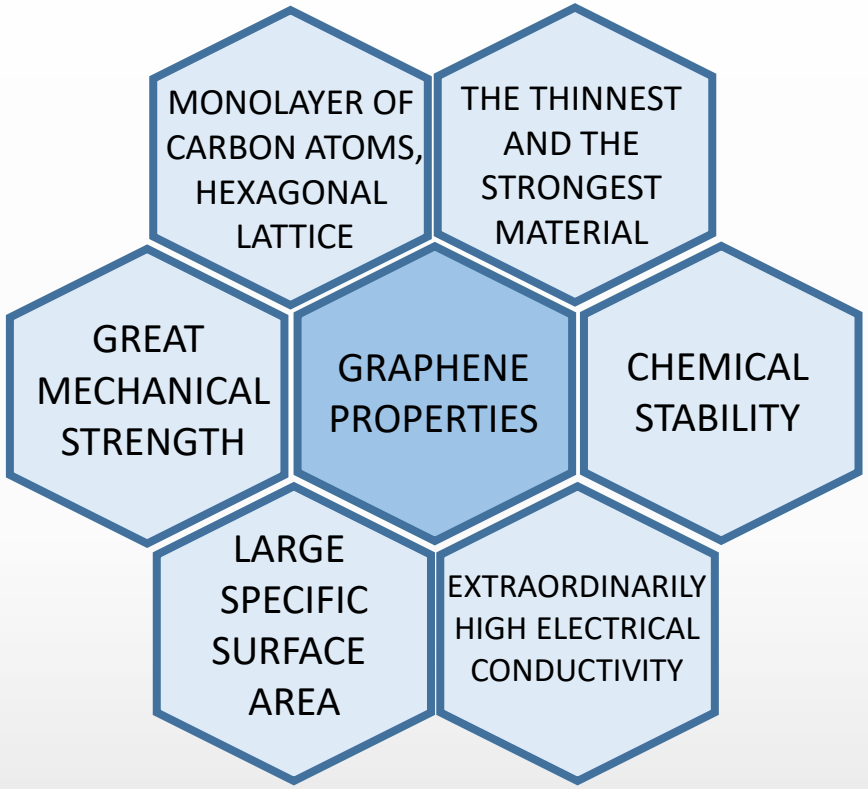
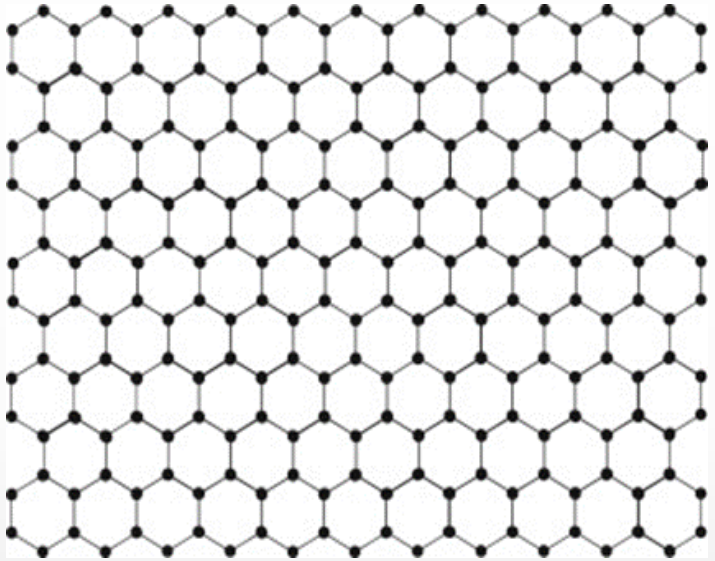
Frank Laboratory of Neutron Physics

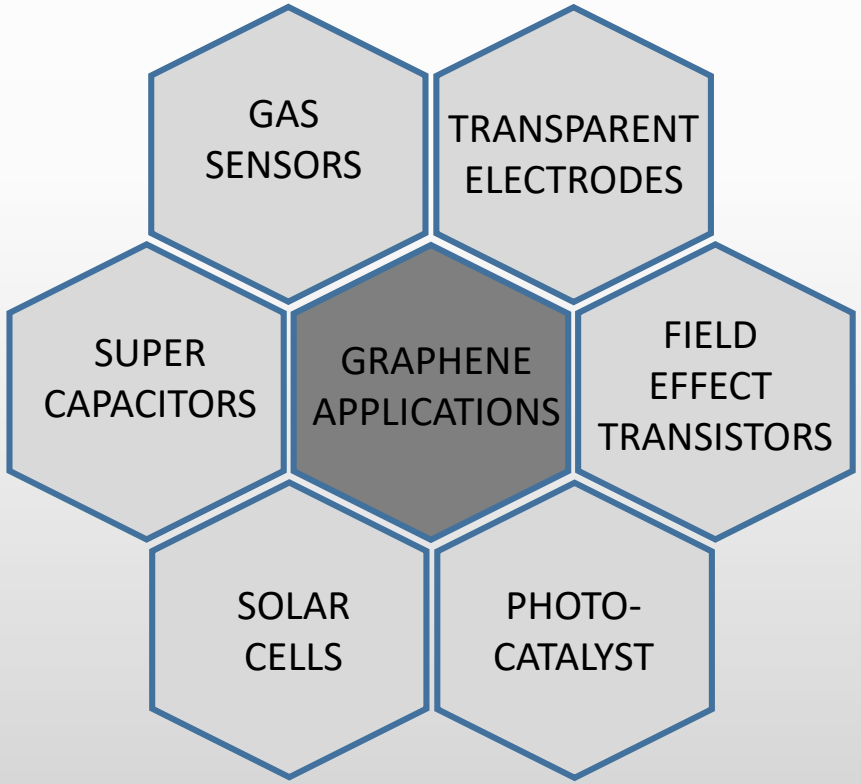
Graphene





Source: www.sciencedirect.com

The great interest of carbon materials especially graphene is connected with the practical applications of these materials in electronic, optoelectronic, and energy storage devices.



AIM OF THE PROJECT

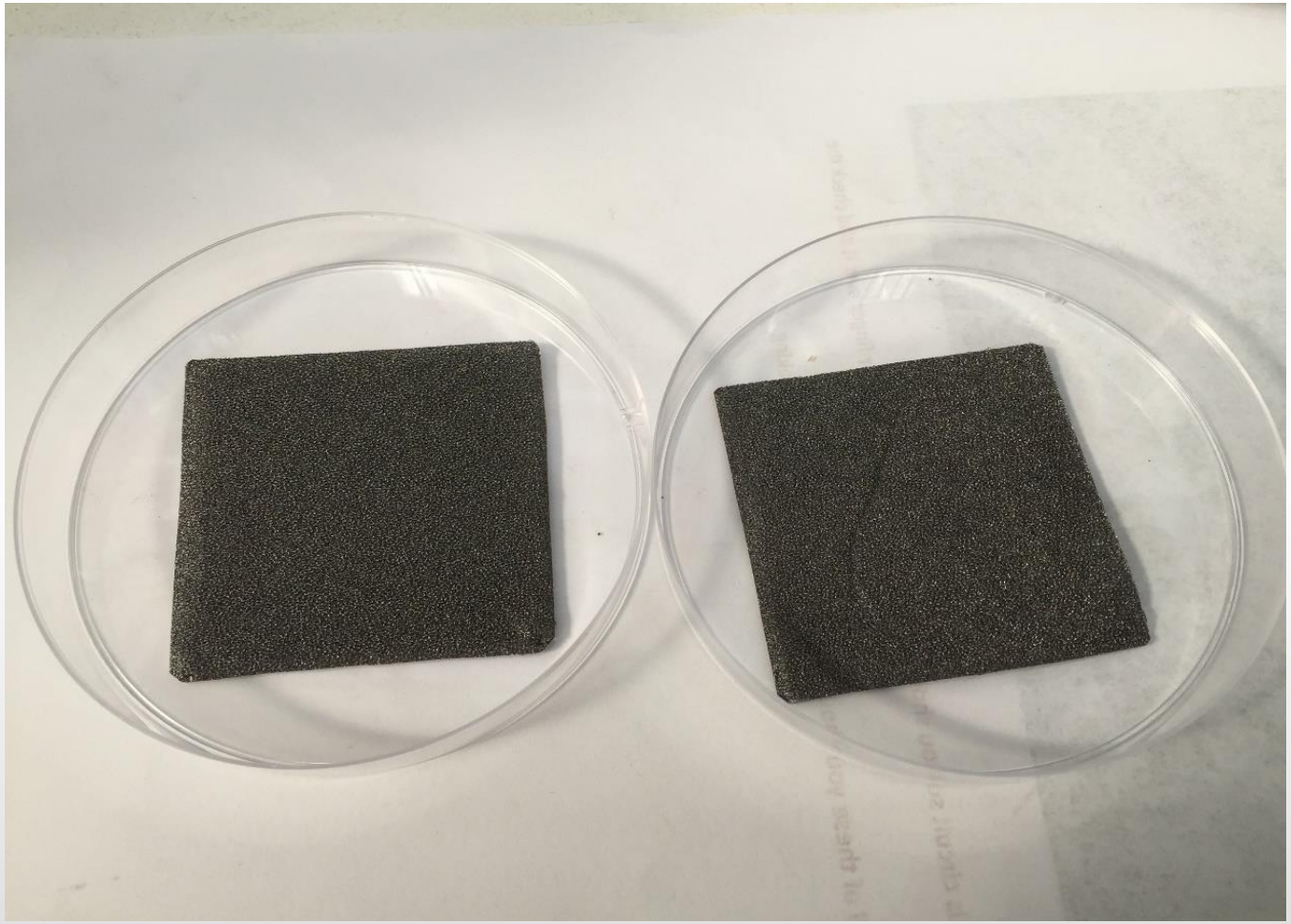
Structural investigation of 3D graphene and graphene/metal oxide composite networks:

Atomic Force Microscopy (AFM)

Raman Spectroscopy

X-ray diffraction (XRD)

Sample preparation – synthesis method



The samples were prepared by Cristina Banciu, National Institute for Research and Development in Electrical Engineering ICPE-CA, 030138, Bucharest, Romania

3D graphene (pure and with Ni foam as a template) and 3D graphene/ZnO composite networks were prepared by **chemical vapour deposition (CVD)** process with methane as the carbon source.

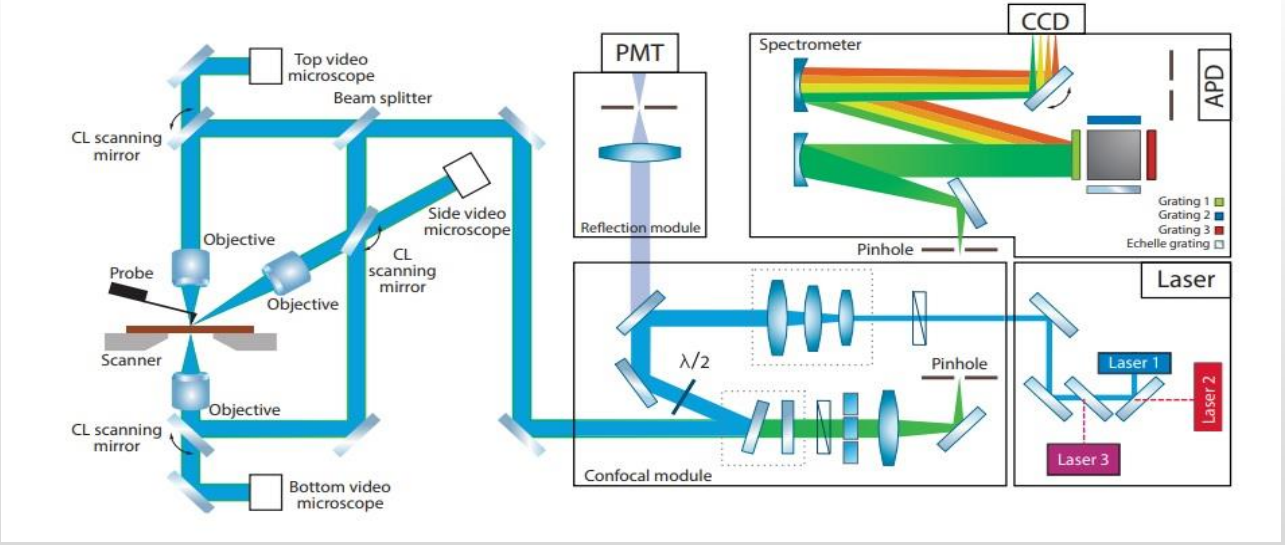
Atomic force microscopy (AFM) and Raman spectroscopy

Atomic force microscopy and Raman spectroscopy were performed using NTEGRA spectra.

Our samples were investigated by Raman spectroscopy in order to identify structural defects. The Raman spectroscopy was also very useful to determine the number of layers in the graphene networks.

AFM is useful for the characterization of graphene due to its high spatial resolution and the various modes that allow probing different physical properties. The combination with Raman spectroscopy, it allows the distinction between different numbers of graphene layers.

Source: https://www.ntmdt-si.com



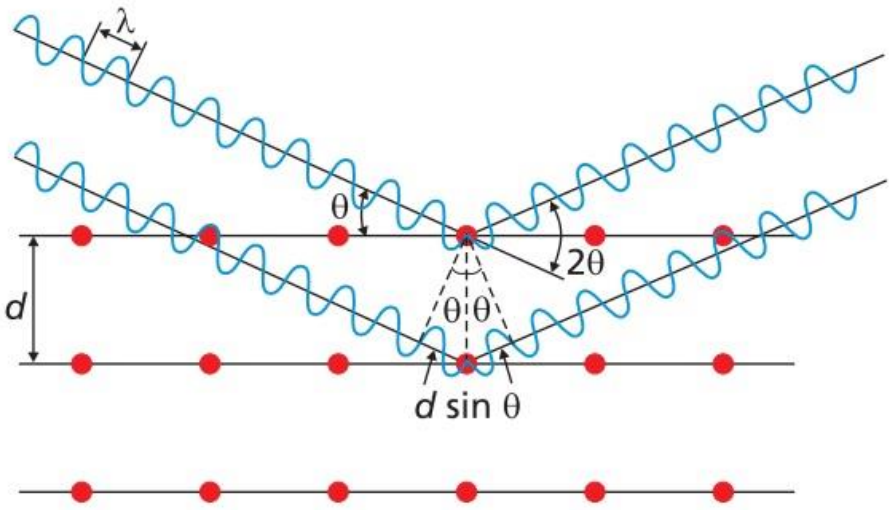
Source: https://www.ntmdt-si.com

X-ray diffraction (XRD)

Source: http://flnp.jinr.ru

Bragg's law:

$$n\lambda = 2d\sin\Theta$$



Source: XRD for the analyst: Getting acquainted with the principles, Martin Ermrich, Detlef Opper

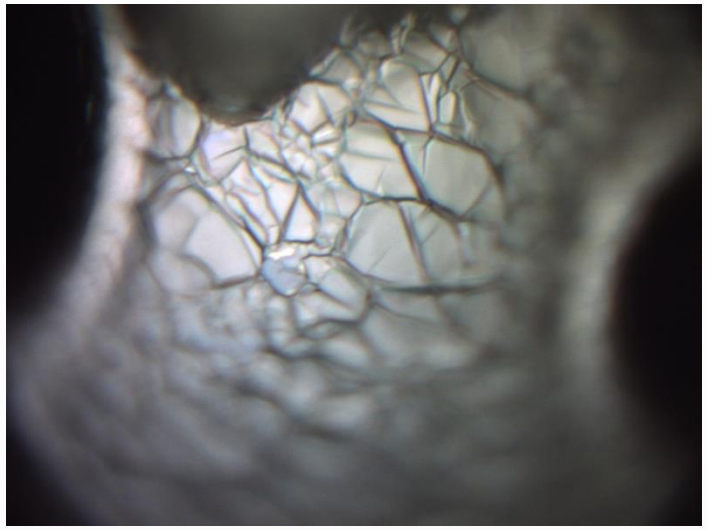
X-ray diffractometer EMPYREAN (PANalytical)

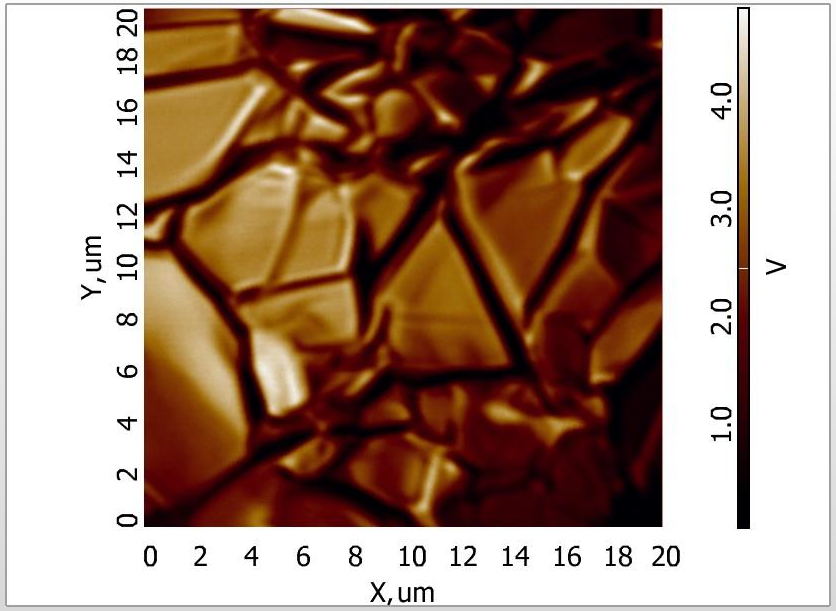
Basic parameters:

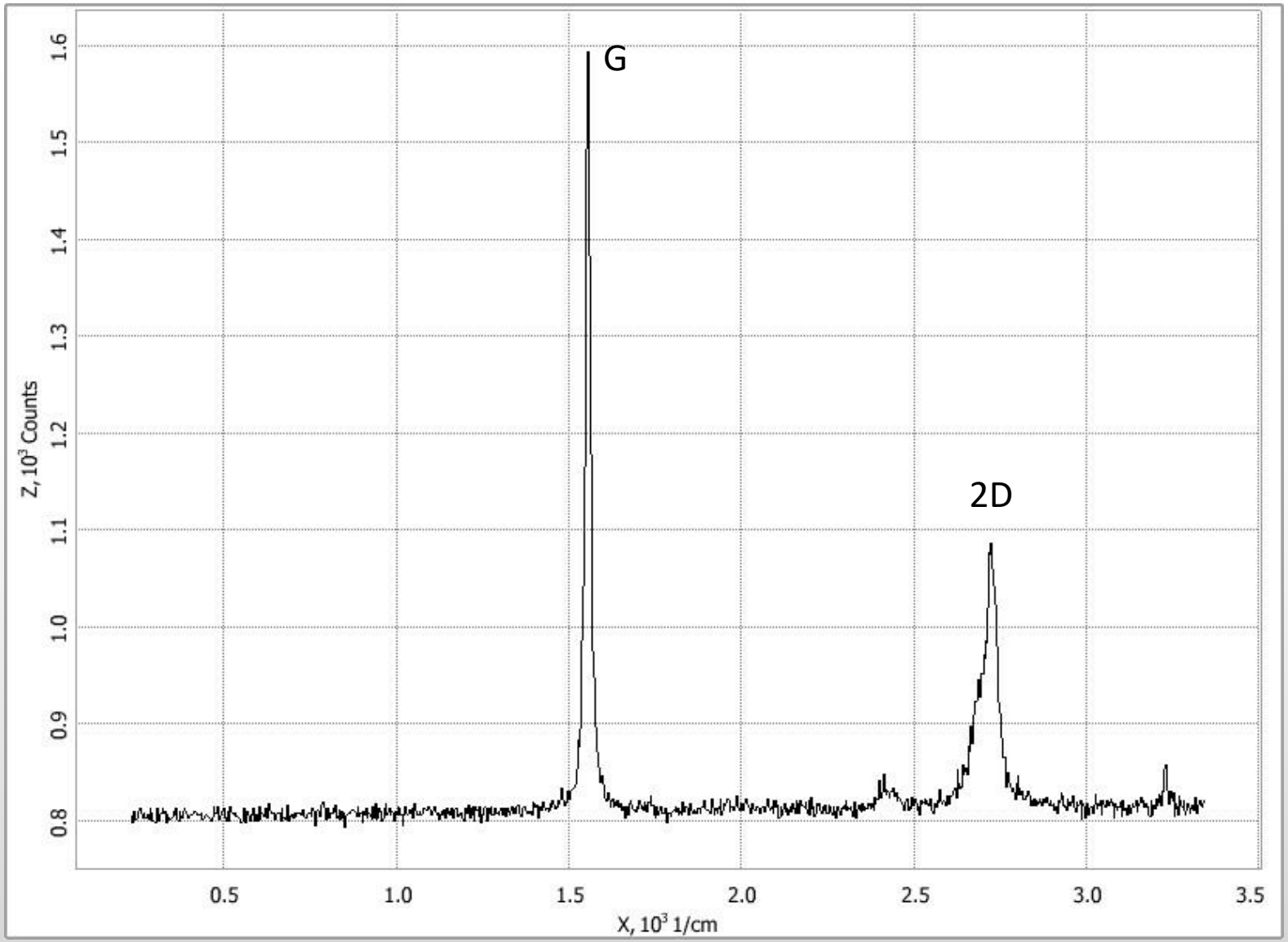
- Flat Sample Holder
- Wave length: Co radiation (~1.789 Å)
- Filter: Fe
- Mask: 10
- Slits: S1=½, S2=¼
- Tension: 40 kV
- Current: 40 mA
- Room temperature (~298K)

XRD patterns were handled using HighScore Plus.

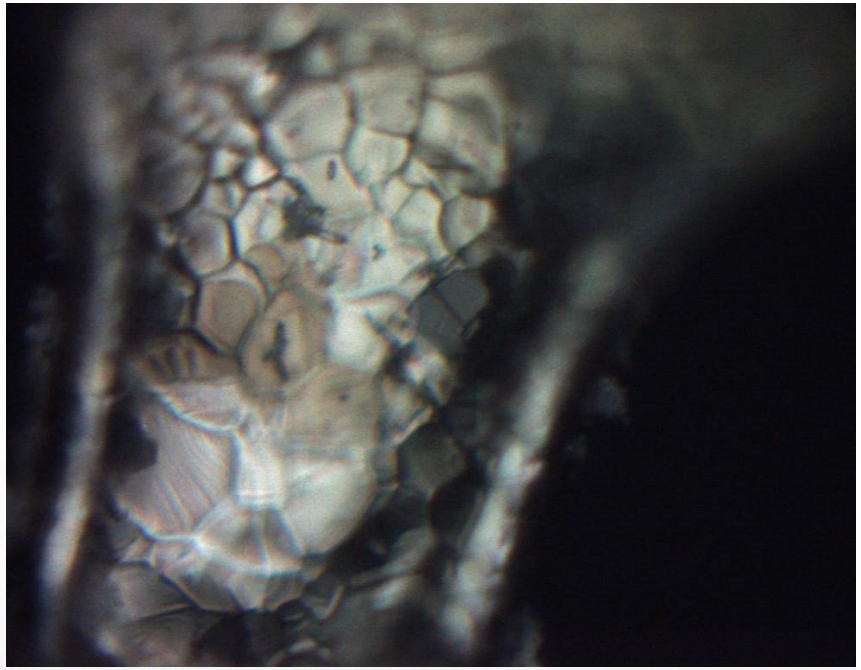
Atomic Force Microscopy and Raman spectroscopy investigation of graphene on Ni foam

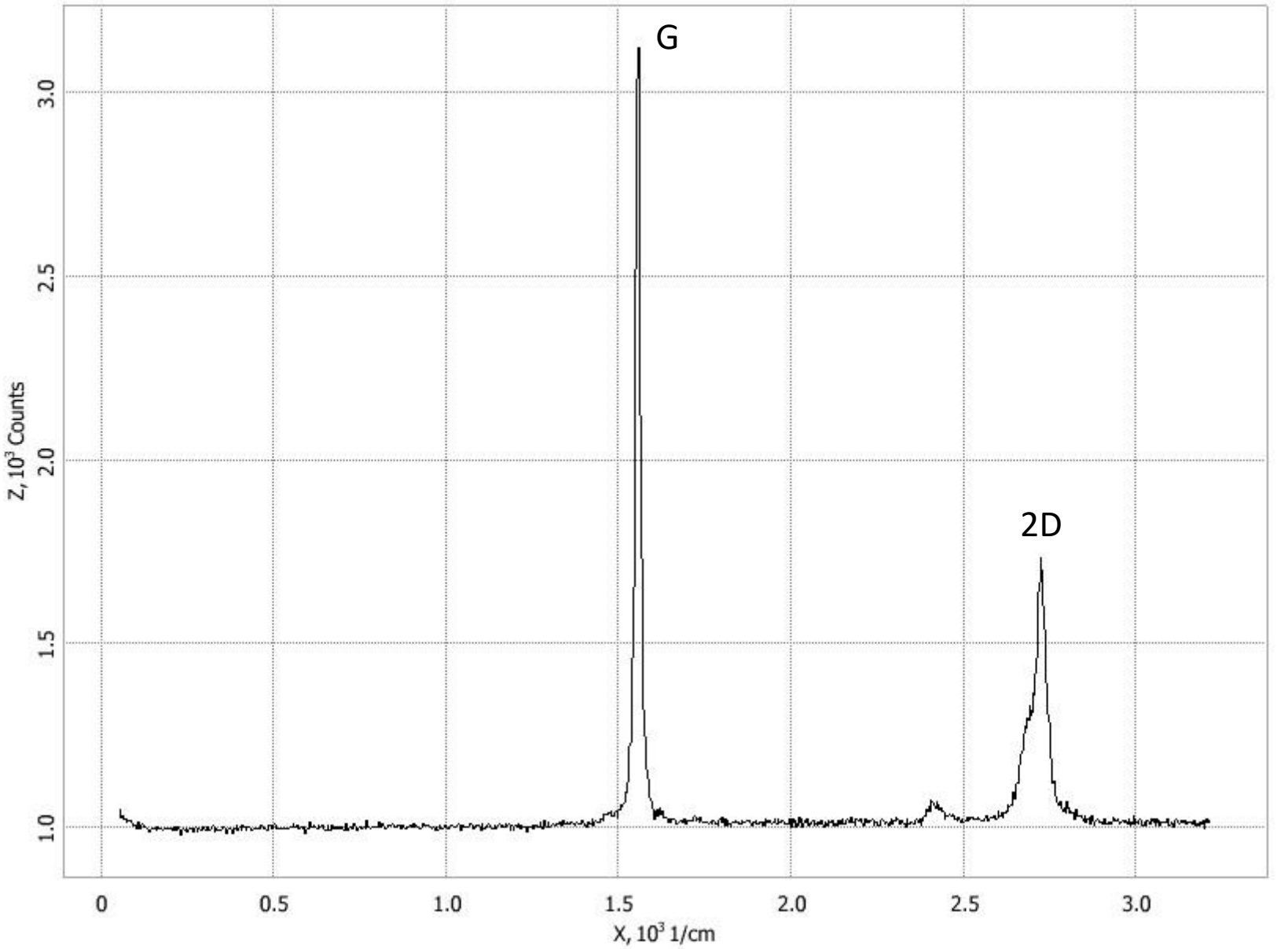






AFM and Raman spectroscopy investigation of graphene/Ni/ZnO composite





AFM and Raman spectroscopy results

- Only the G (1580 cm^{-1}) and 2D (2680 cm^{-1}) bands are observed. The absence of a D band (1350 cm^{-1}) indicates that the graphene foam is of high quality .
- From the analysis of Raman spectra, the number of carbon layers can be determined by determining the FWHM of the 2D band and the ratio of the intensity of the G and 2D bands. In the case of a larger number of layers, the 2D band widens and the ratio of the intensity of the 2D to G band is less than 1. The ratio of I_{2D} / I_G in the case of the single layer is equal to or greater than 2, for the double layer it is between 1 and 2, and for the multi-layer it is smaller than 1.
- Our results show that the FWHM of the 2D band is about 30 cm^{-1} and the ratio of I_{2D} / I_G is less than 1. It means that we have more than two graphene layers.

We have to think about whether we have graphene or graphite?

XRD pattern of 3D graphene

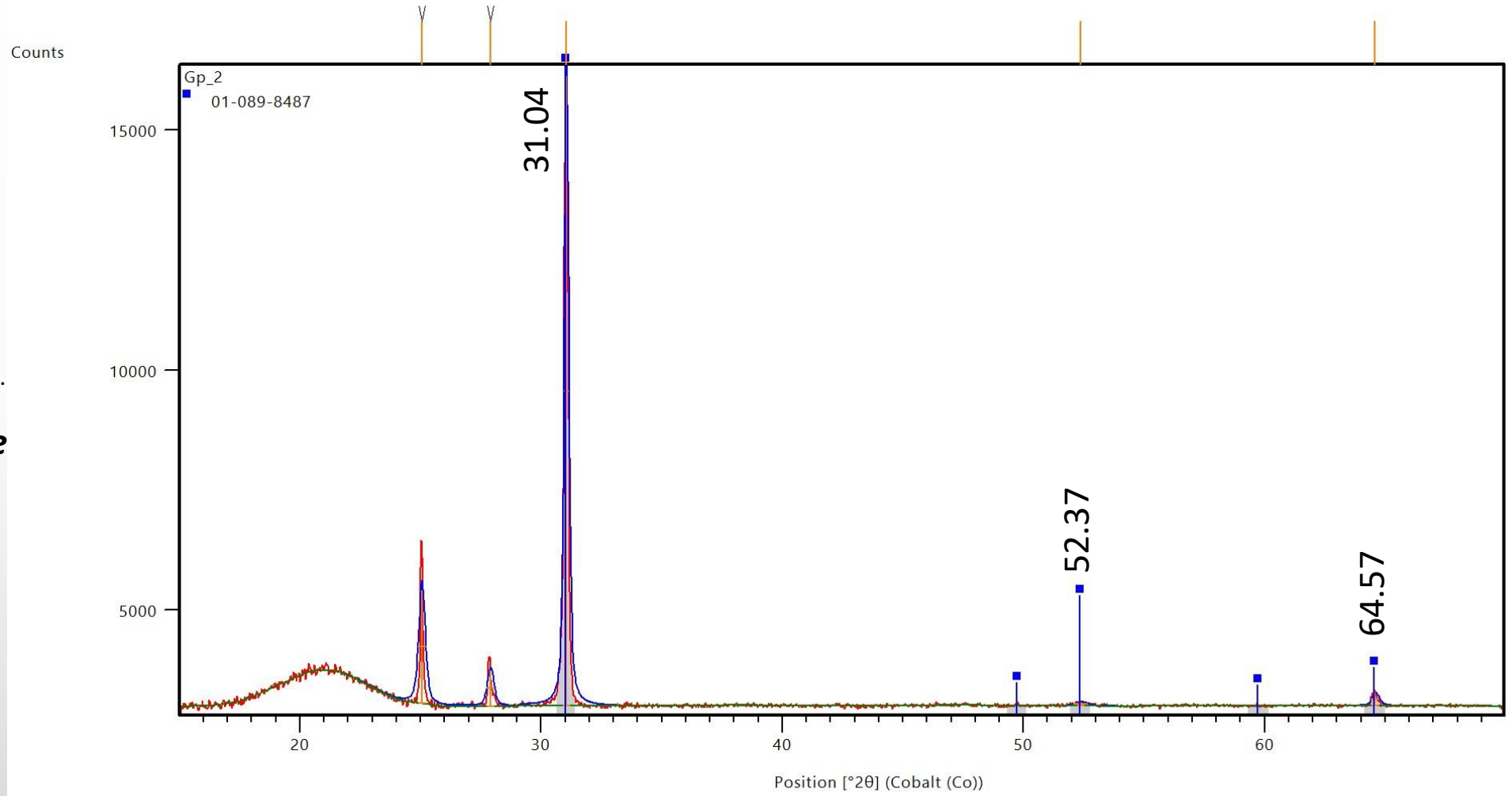
Measured XRD pattern

Empirical formula: C
Chemical formula: C
Crystal system: Hexagonal
a (Å): 2.4610
b (Å): 2.4610
c (Å): 6.7080
Alpha (°): 90.0000
Beta (°): 90.0000
Gamma (°): 120.0000

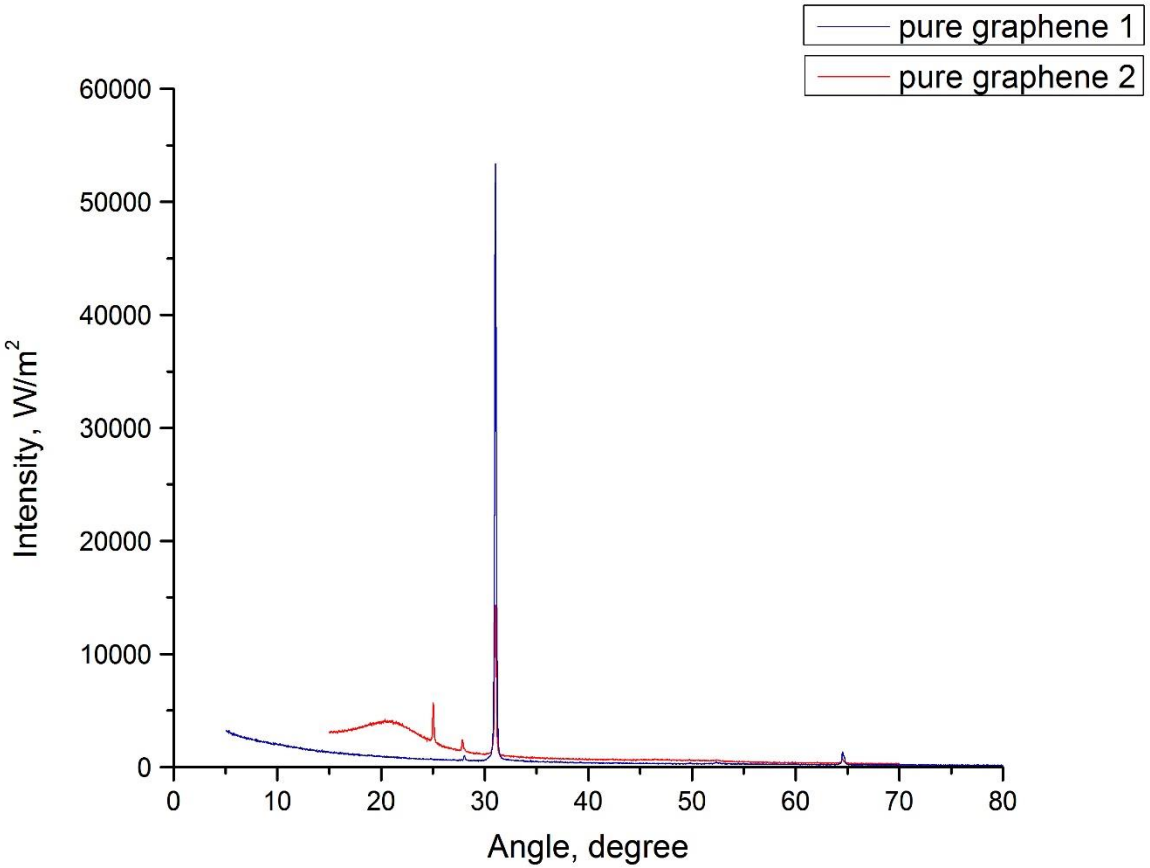
Displayed data is from HighScore Plus database.

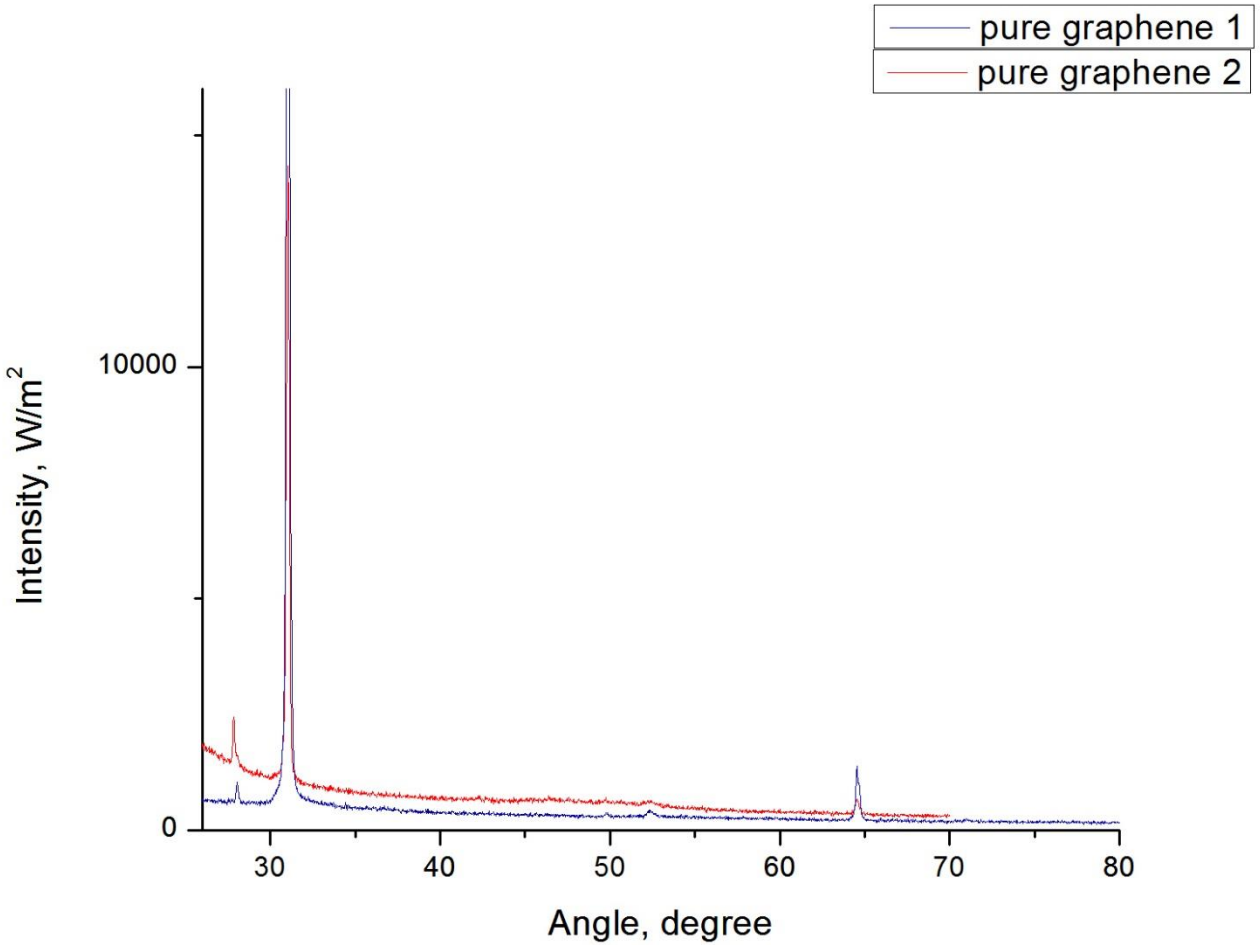
Data from Mincryst database

Formula: C
Crystal system: Hexagonal
a (Å): 2.464
b (Å): 2.464
c (Å): 6.711
Alpha (°): 90.0
Beta (°): 90.0
Gamma (°): 120.0



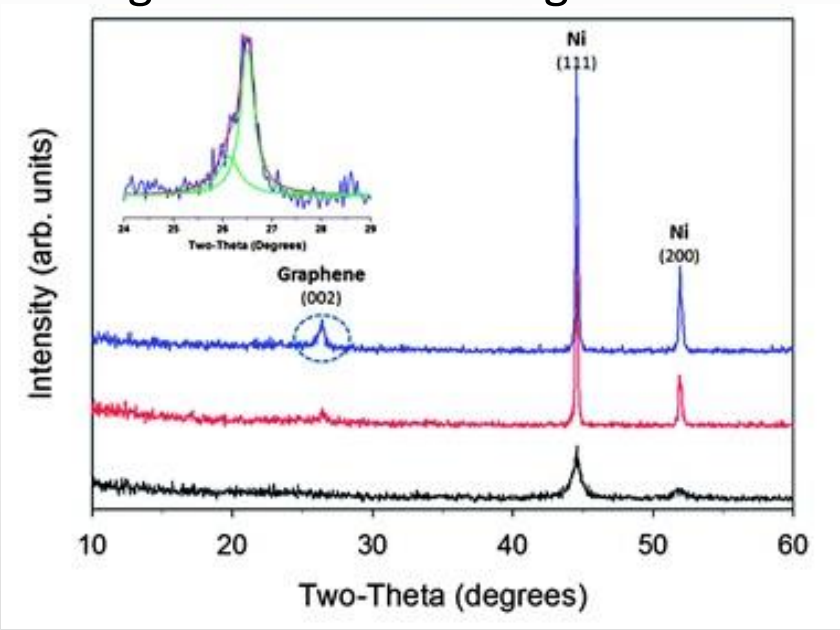
Comparison of the XRD patterns



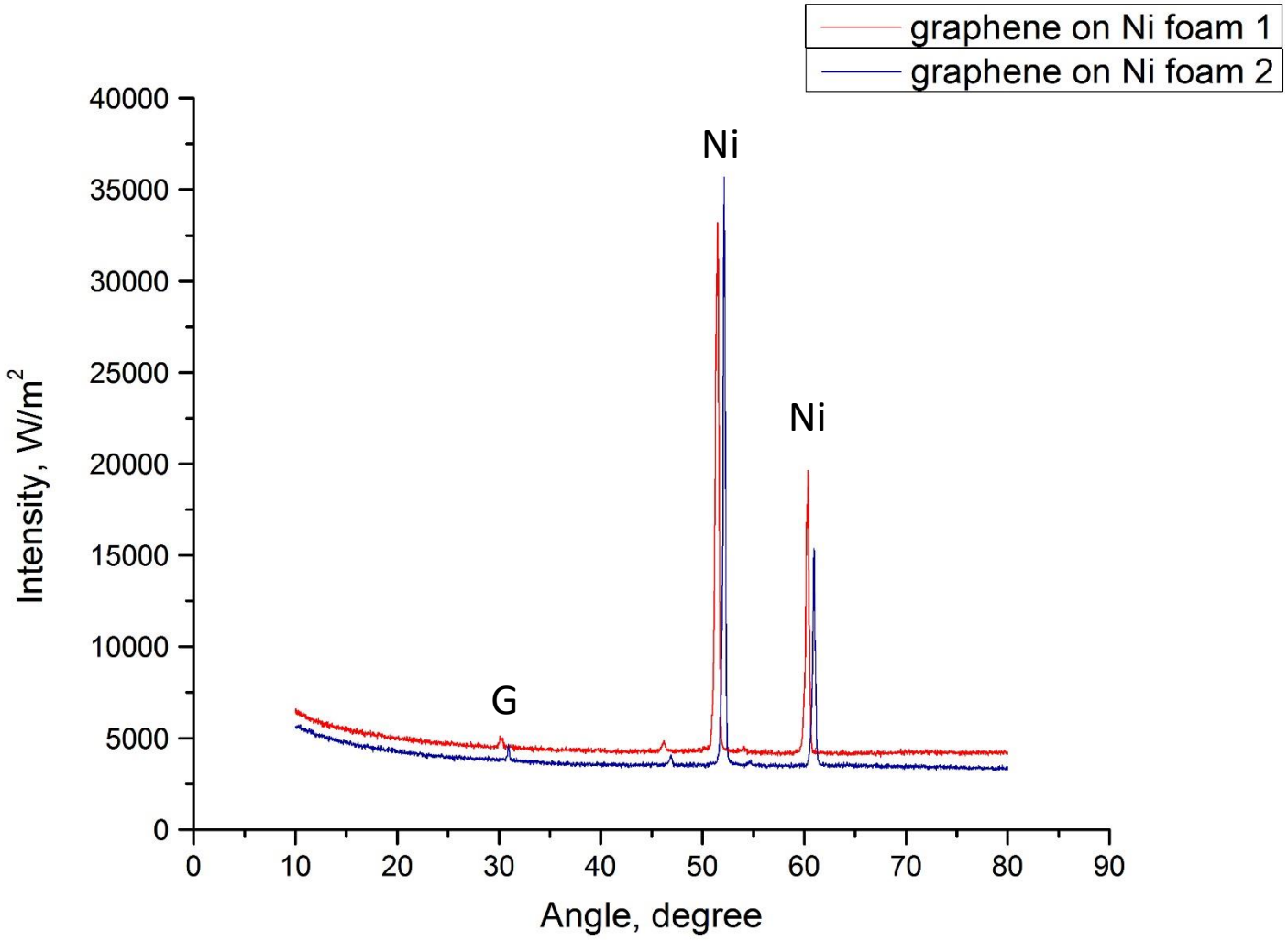


XRD patterns of graphene on Ni foam

- The synthesis method is reproducible
- The peaks from the measurements are comparable with the theoretical results
- Significant peak shift can be caused by using different wavelength of radiation

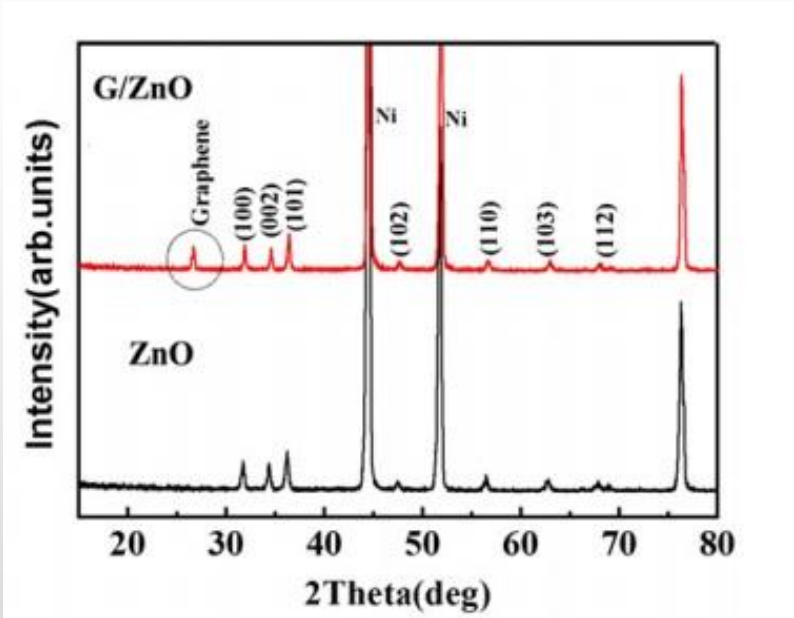


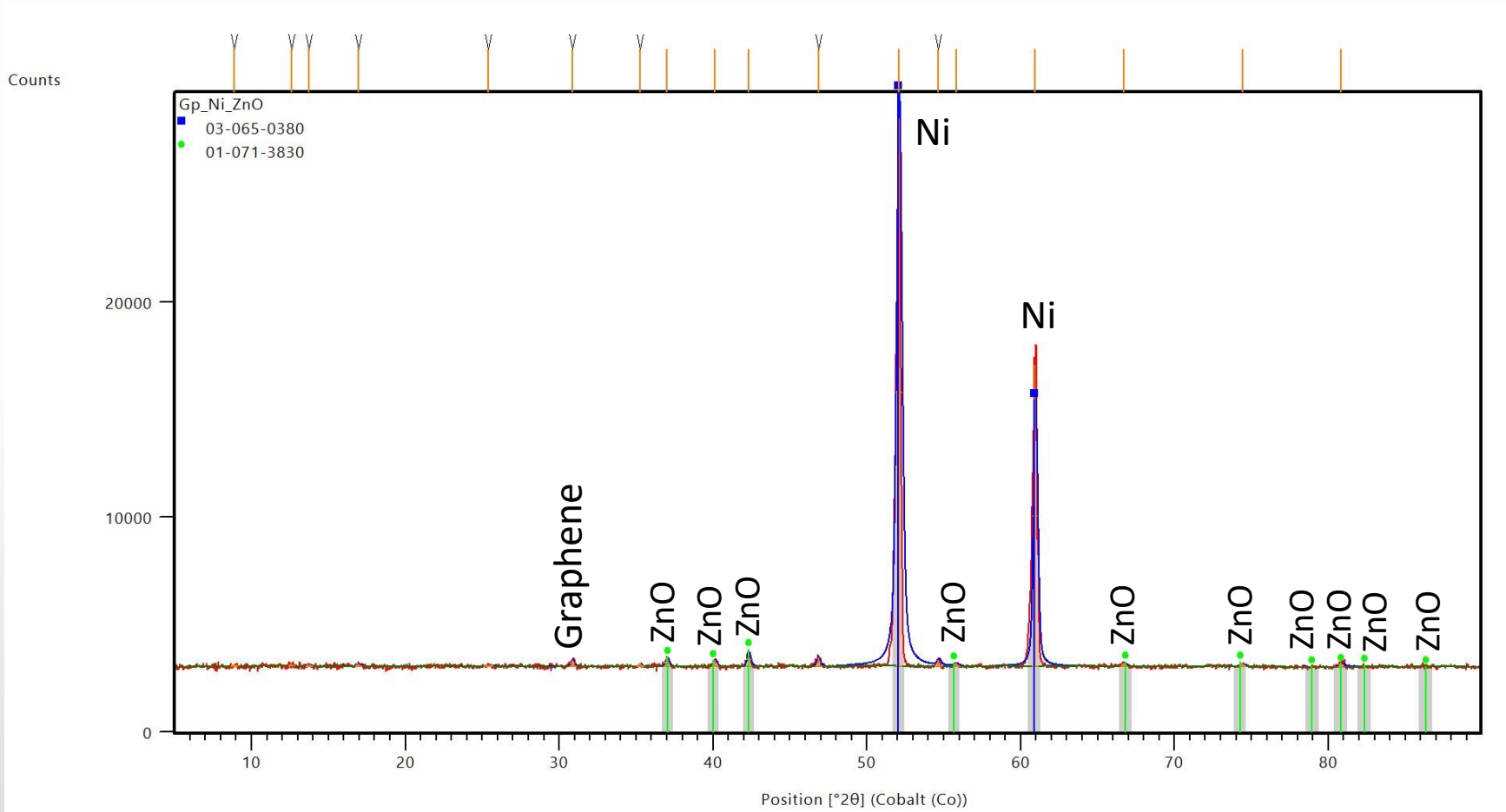
Source: Three dimensional nickel–graphene core–shell electrodes,
Xiaoyin Xiao, Joseph R. Michael, Thomas Beechem, Anthony McDonald, Mark Rodriguez, Michael T. Brumbach, Timothy N. Lambert, Cody M. Washburn, Joseph Wang, Susan M. Brozik, David R. Wheeler, D. Bruce Burckel and Ronen Polsky



XRD pattern of graphene/Ni/ZnO composite

- Identification of graphene is difficult, because the peak intensity of Ni is significantly higher than the peak intensity of graphene

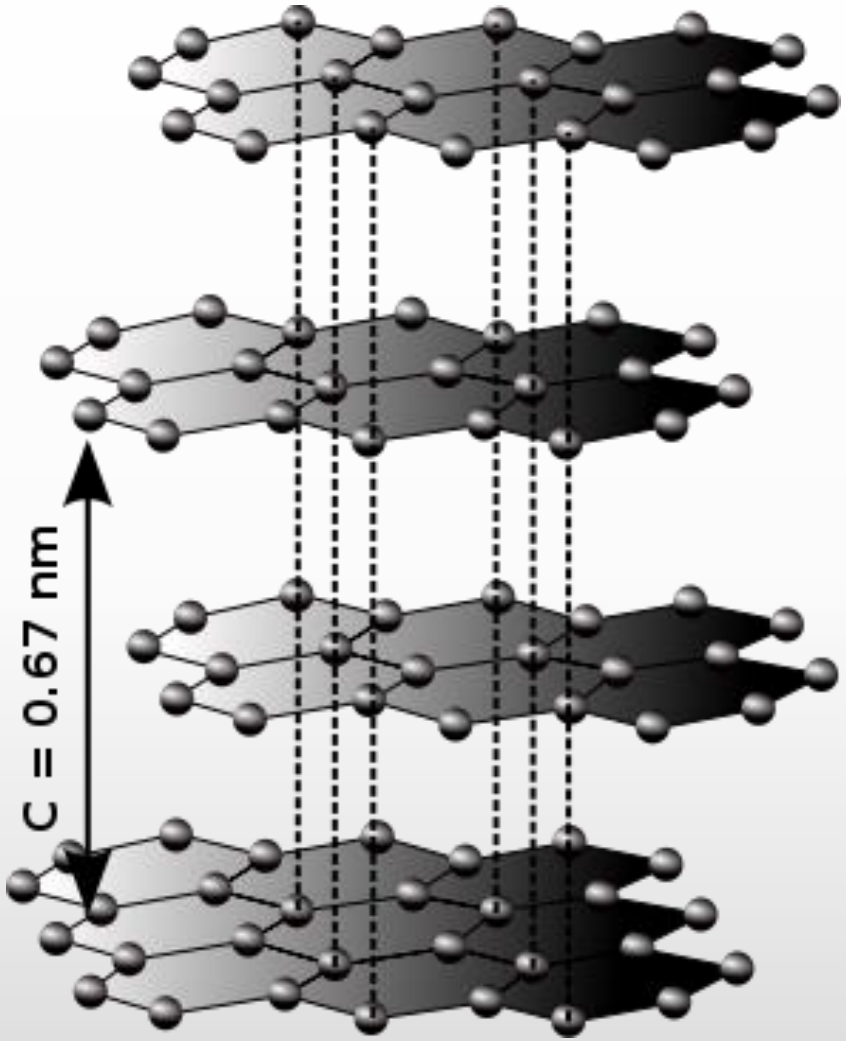




Source: 3D graphene/ZnO composite with enhanced photocatalytic activity, *Ran Cai, Jia-genWu, Li Sun, Yan-jun Liu, Ting Fang, Shan Zhu, Shao-yang Li, YueWang, Li-feng Guo, Cui-e Zhao, Ang Wei*

Conclusions

- The synthesis method is reproducible
- The results of the measurements are comparable with theoretical results
- Considering both techniques, it can be said that the samples are scarcely defective, but they are not single-layer graphene – they are multilayer graphene or simply graphite (more than 10 graphene layers)



Source: www.researchgate.net


PANalytical