

ROCKLEIGH INDUSTRIES

INDUSTRIAL MINERALS & CHEMICALS

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Material Characteristics Report

8 Rockleigh rd, Rockleigh, New Jersey 07647 USA 1 917-825-3806
bariteworld@optonline.net / www.BariteWorld.com



Purpose of the study:

- Investigated the composition, microstructure and morphology of the graphene powder provided by the customer, using proper technique and methodology;
- Provide the next parameter characterizing the graphene material:
 - flakes thickness D , estimated number of layers N and the lateral size of the flakes L ;
- Estimate the potential of the material to be separated into different categories, based on thickness and size.
- Provide assessments on the technical compliance of the material to the existing standards in the field.

Sources/References used:

- *ISO/TS 80004-13:2017(E) Standard "Graphene and related two-dimensional (2D) materials"*.
- *Good Practice Guide #145: Characterization of the Structure of Graphene.*

Methodology

Samples preparation: All the measurements were done on the as-obtained material – dispersed, dissolved in IPA and immediately ultrasonicated for 15 minutes, drop-casted onto Si wafers and dried at 45 C. **No change of the material or intentional/nonintentional separation was made.**

Lateral size of the flakes was measured by Scanning Electron Microscopy. Thus image contrast and instrument resolution allows to estimate the shape and lateral size of the flakes either in ensemble or lonely located over the substrate.

Thickness of the flakes was measured by Atomic Force Microscopy. The height of the steps corresponds to the thickness of the flakes. It should be noticed, we have taken into account only realistic data, assuming agglomeration of the flakes and the vertical step is corresponding to the flake thickness.

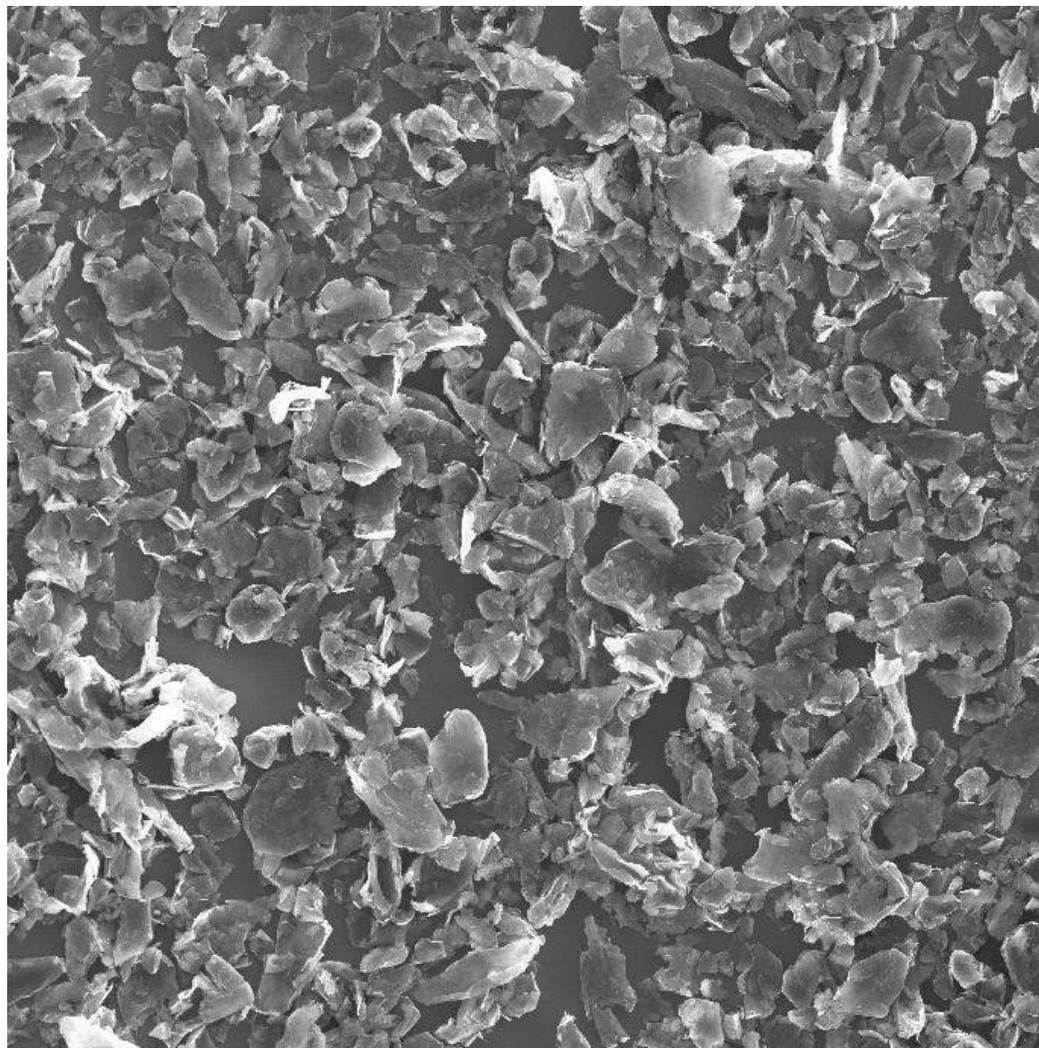
Numbers of layers were calculated assuming the graphene monolayer thickness standard for tapping mode AFM ~ 0.5 nm.

Instruments

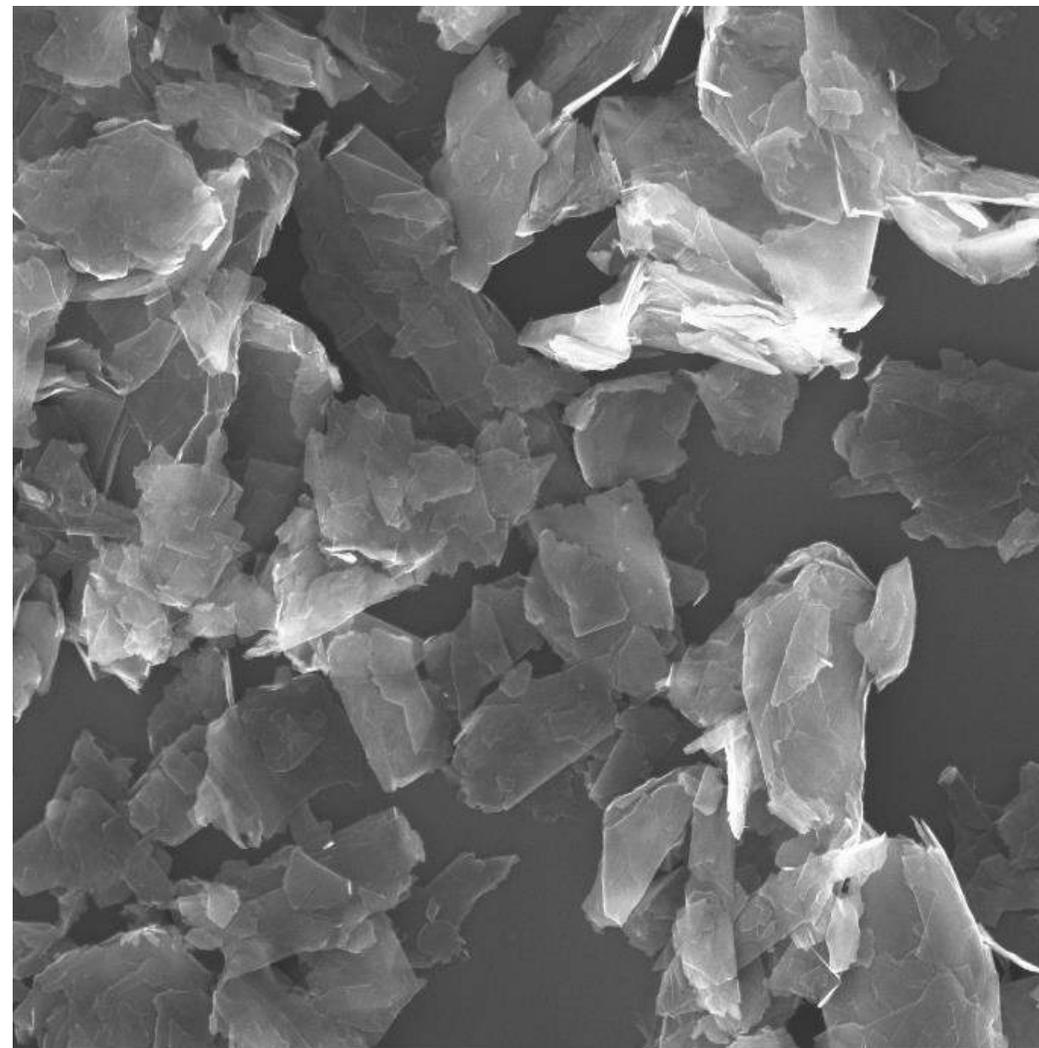
Atomic Force Microscopy (AFM) was measured by Veeco Digital Instruments Nanoscope 3100 in tapping mode. Every 3D scan was cut into respective planes and the step heights were estimated.

Scanning electron microscopy (SEM) was used to characterize the samples microstructure in a Leo 1550 Gemini SEM at operating voltage ranging from 10 kV to 20 kV and standard aperture value 30 mm. Calculations were made both manually and using the adapted software.

Results: Flakes Lateral Size

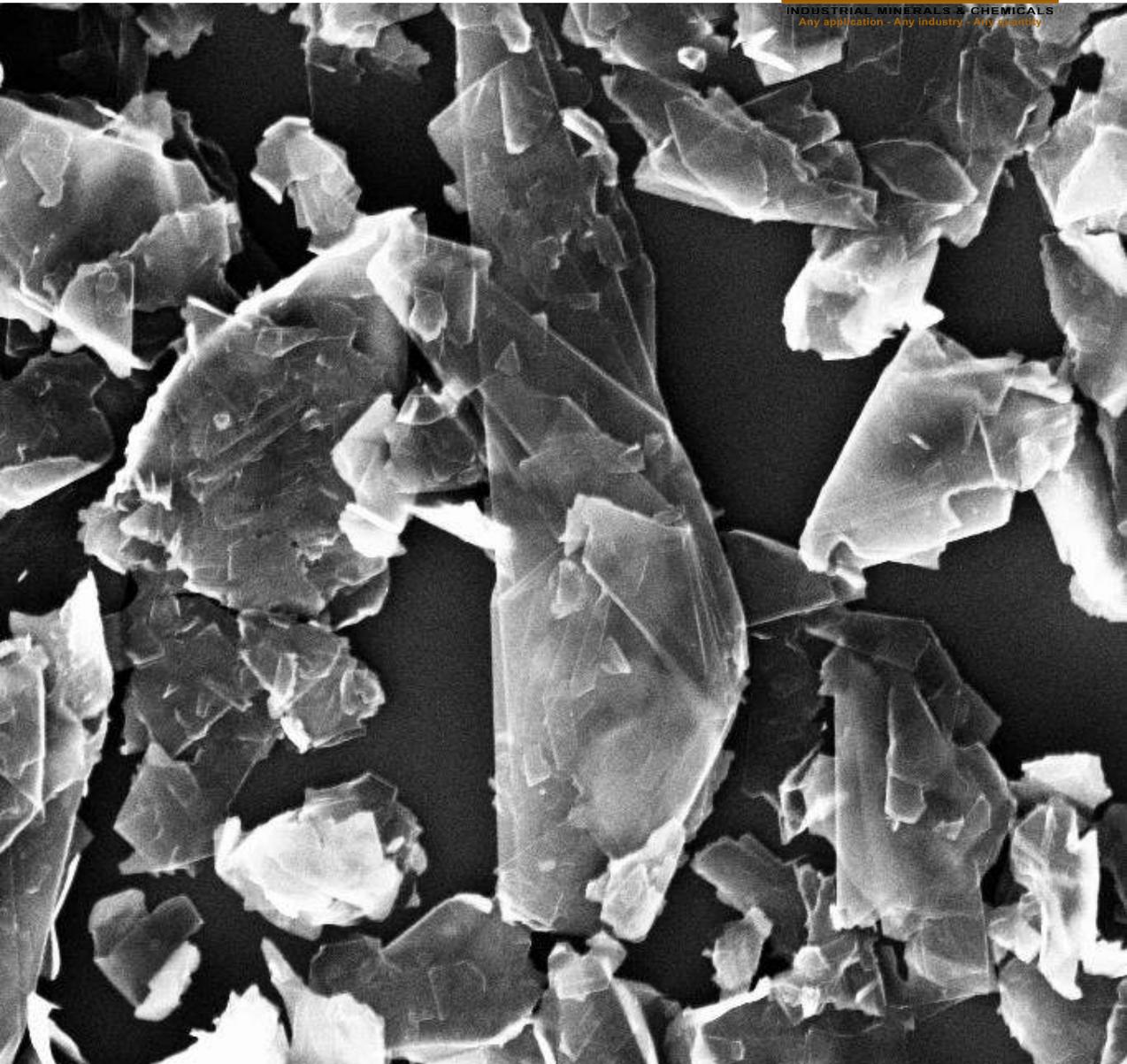
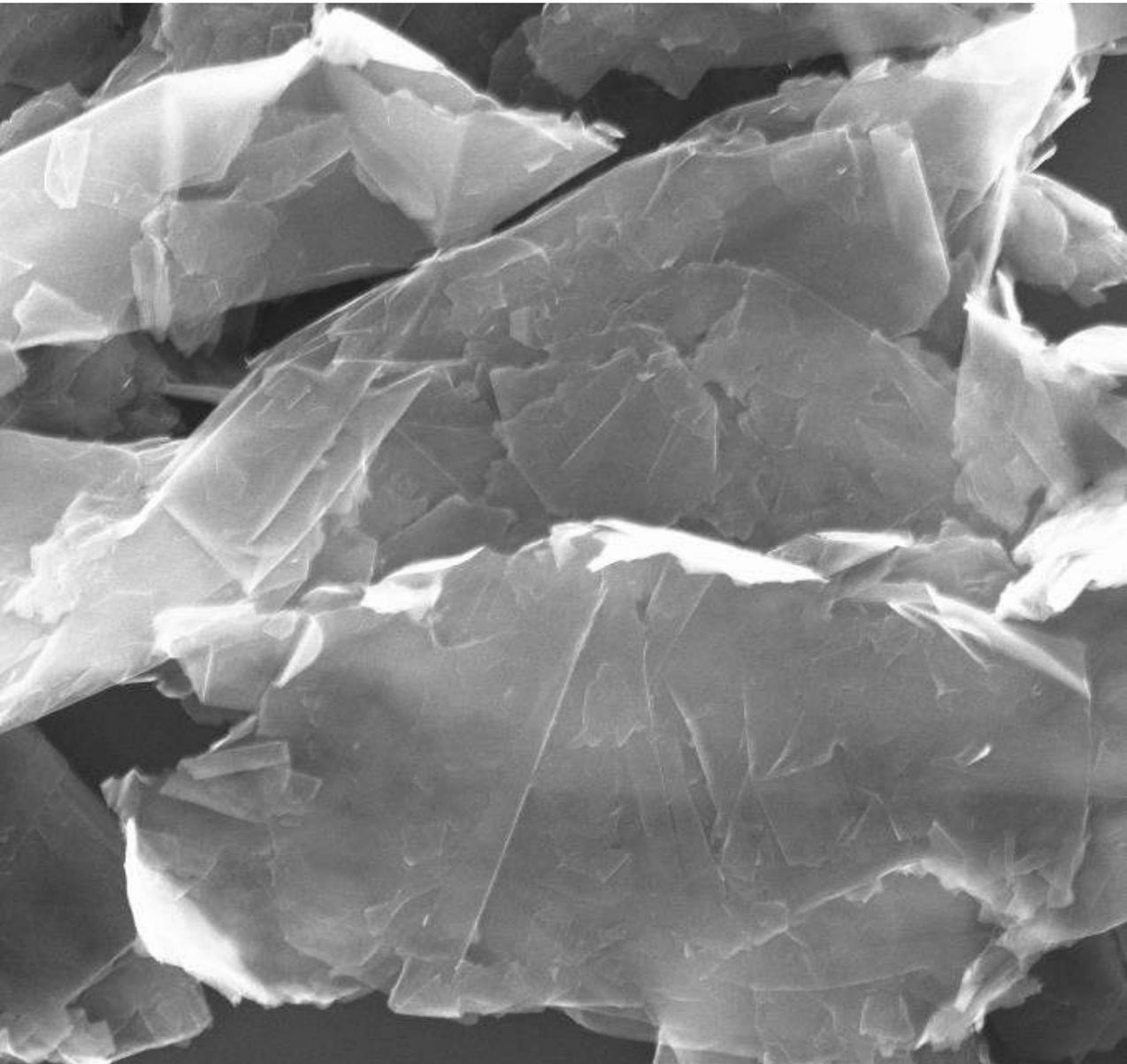


5 K X 10 μ m EHT = 10.00 kV Signal A = InLens Date
WD = 2.6 mm Photo No. = 9175 Time



20 K X 2 μ m EHT = 10.00 kV Signal A = InLens Date :25
WD = 2.9 mm Photo No. = 9180 Time :18

Results: Flakes Lateral Size



3 μ m

EHT = 10.00 kV
WD = 2.6 mm

Signal A = InLens
Photo No. = 9177

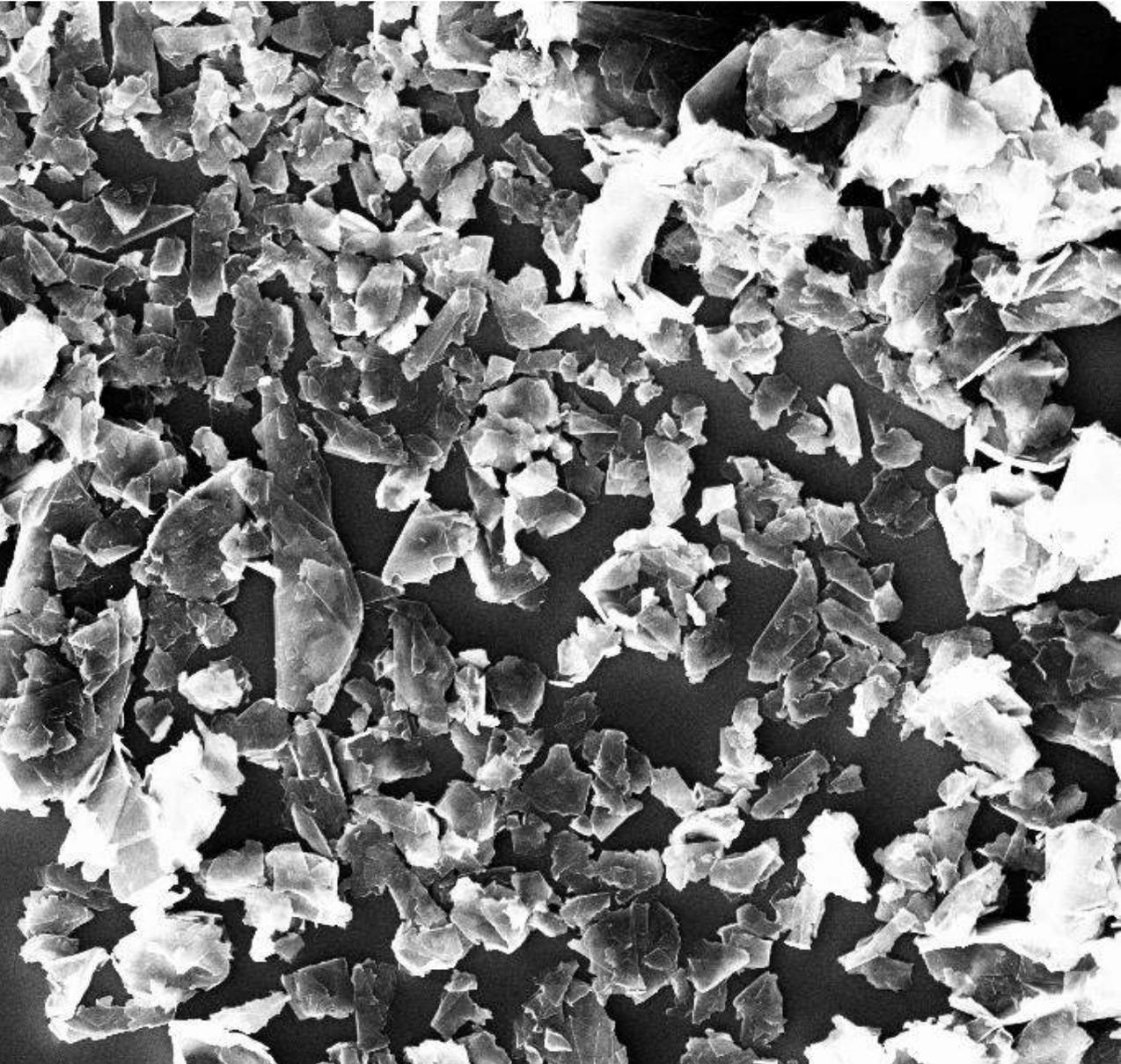
Date :25 Jun
Time :18:1215.82 K X

1 μ m

EHT = 10.00 kV
WD = 2.9 mm

Signal A = InLens
Photo No. = 9185

Results: Flakes Lateral Size



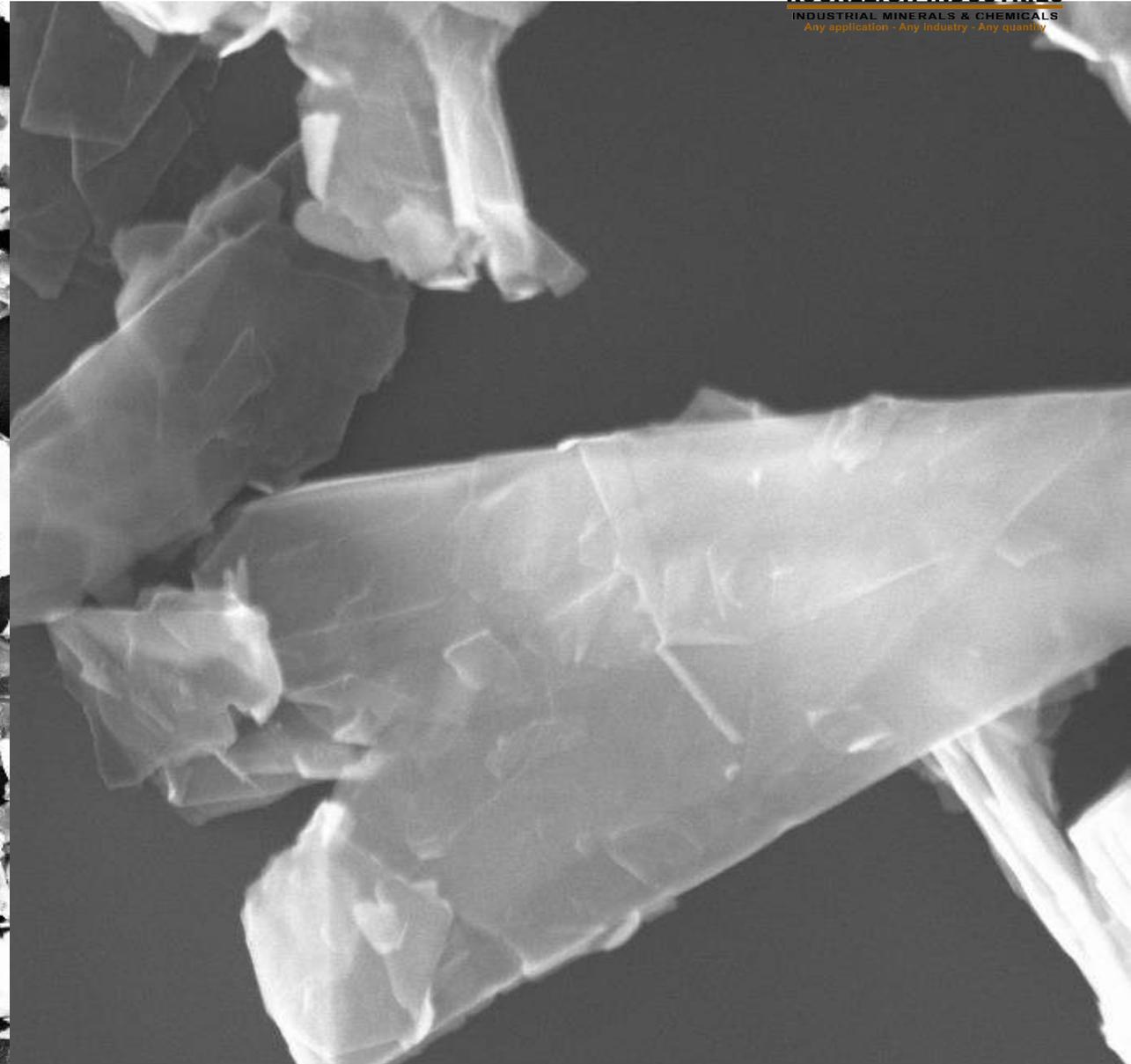
EHT = 10.00 kV
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Signal A = InLens
Photo No. = 9186

Date :25 Jun 2020
Time :18:32:22

X

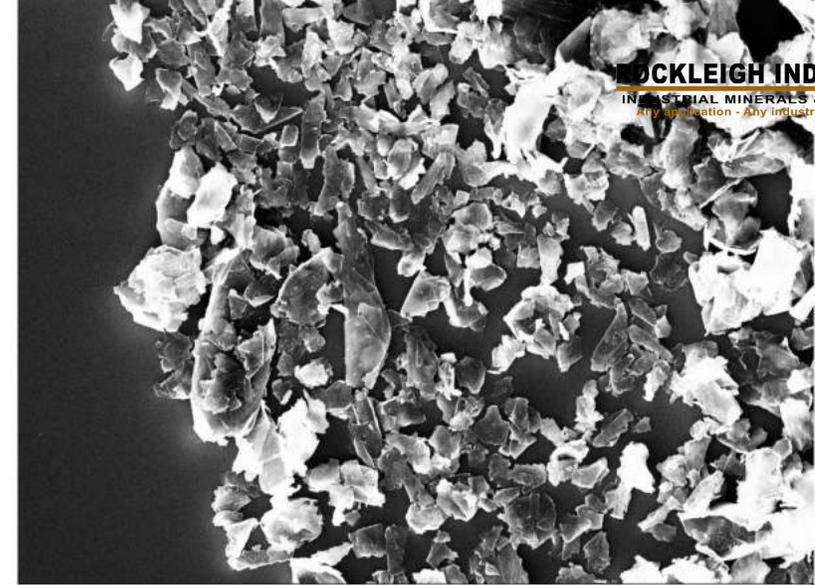
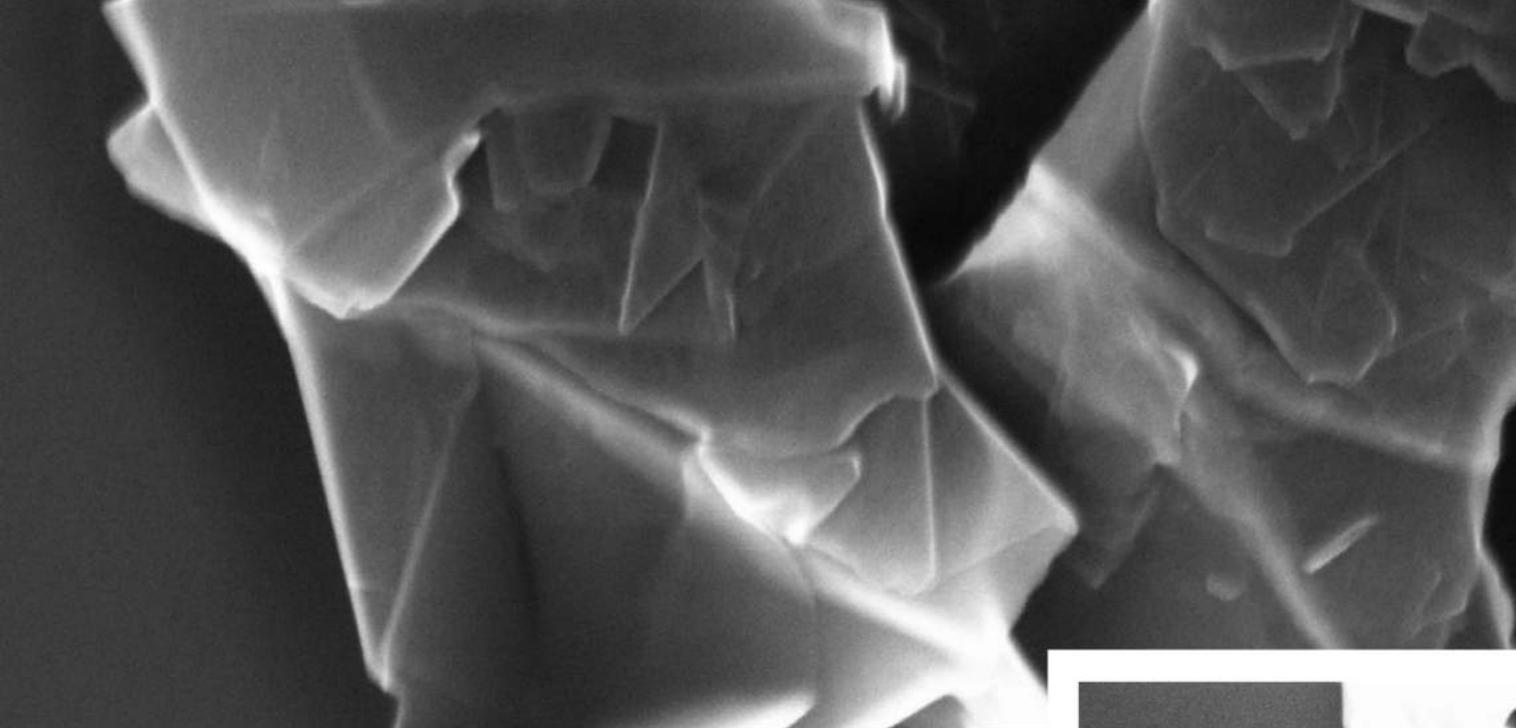
1 μ m



EHT = 10.00 kV
WD = 2.6 mm

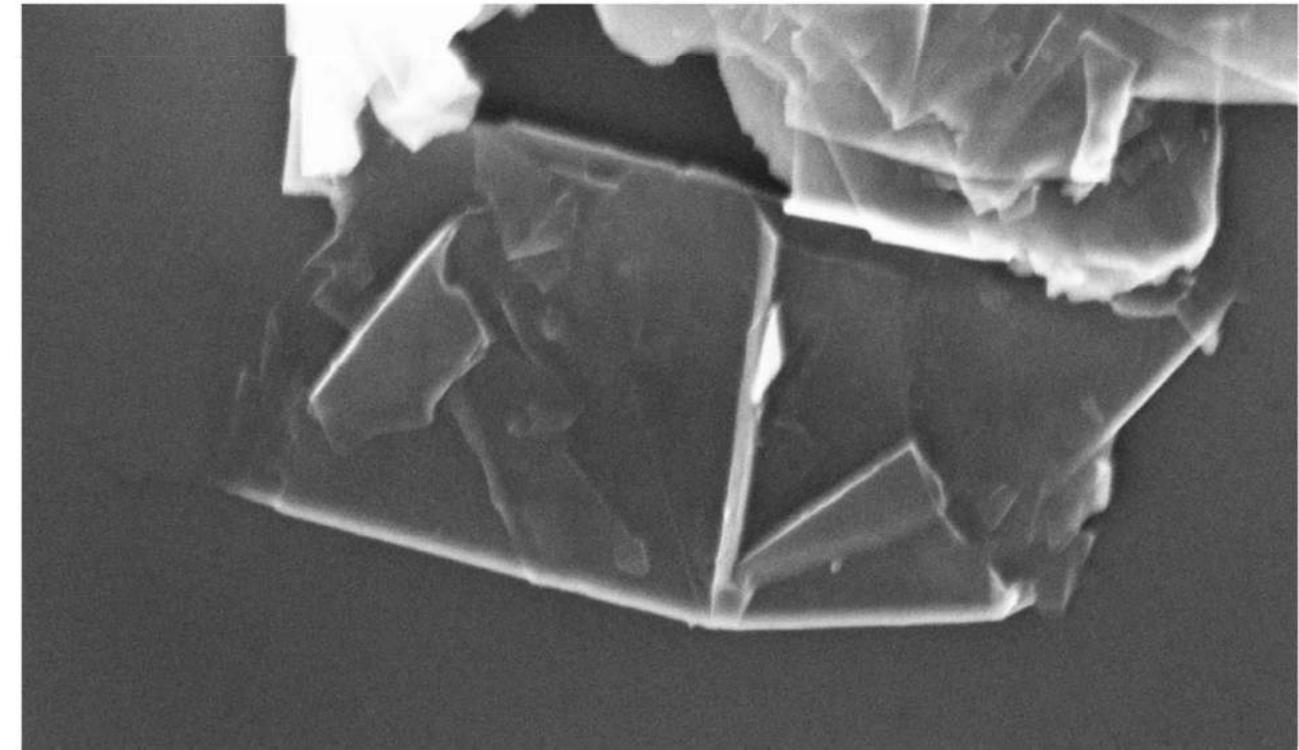
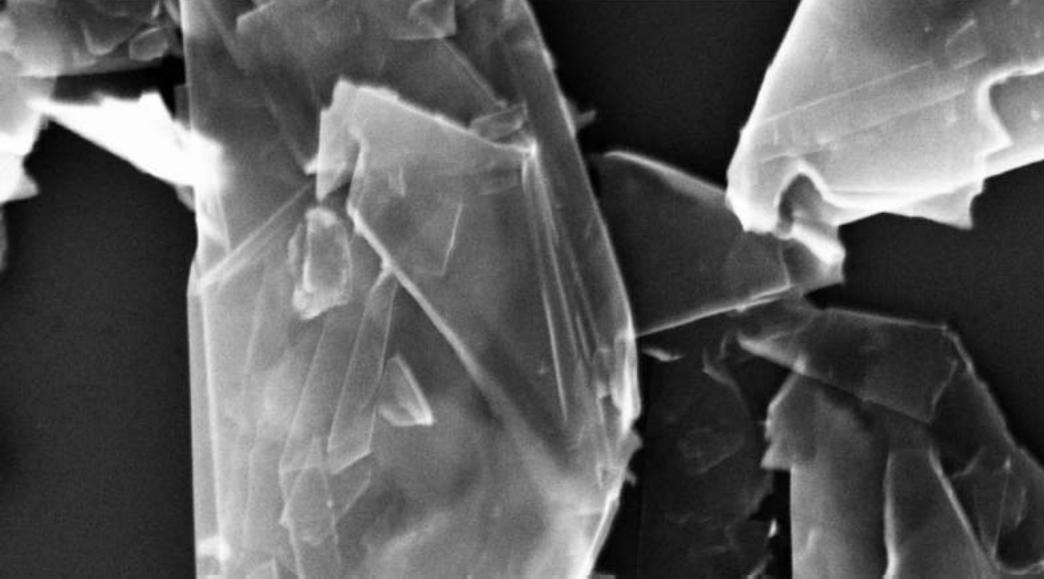
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Date :25
Time :1

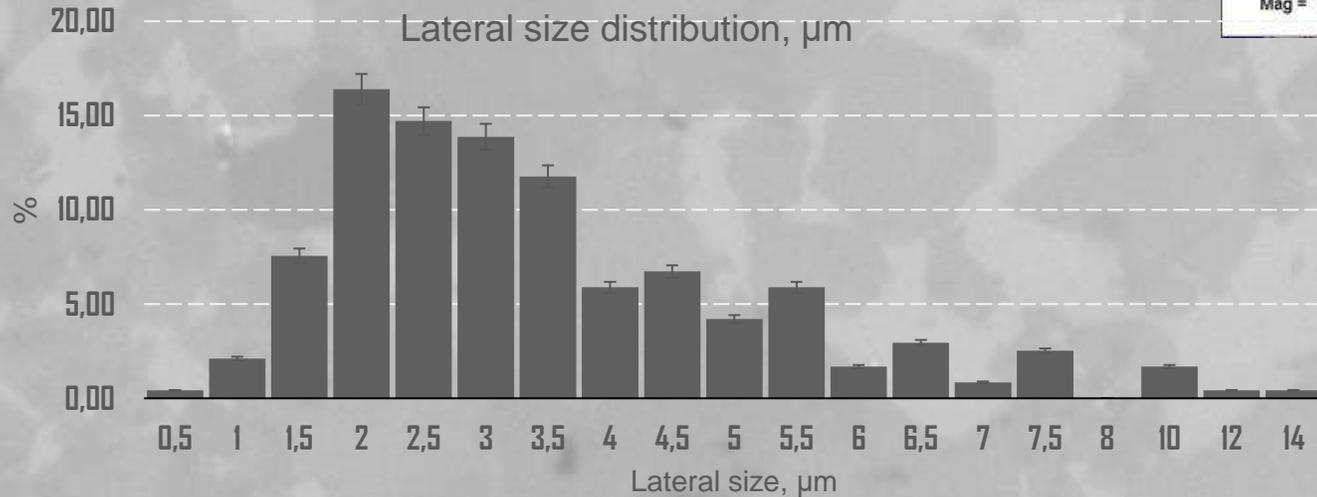
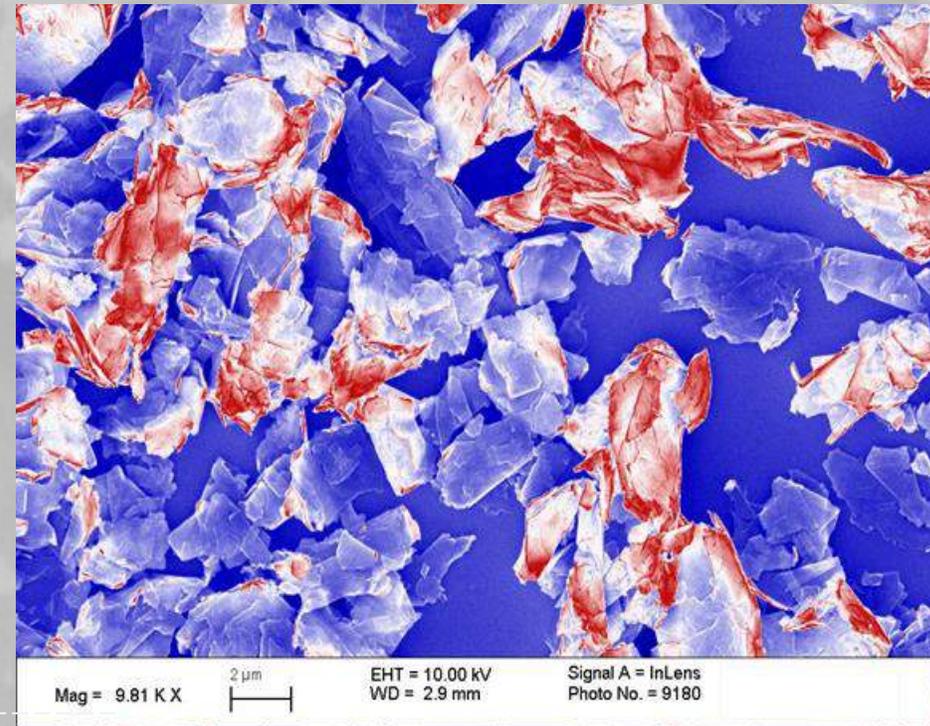
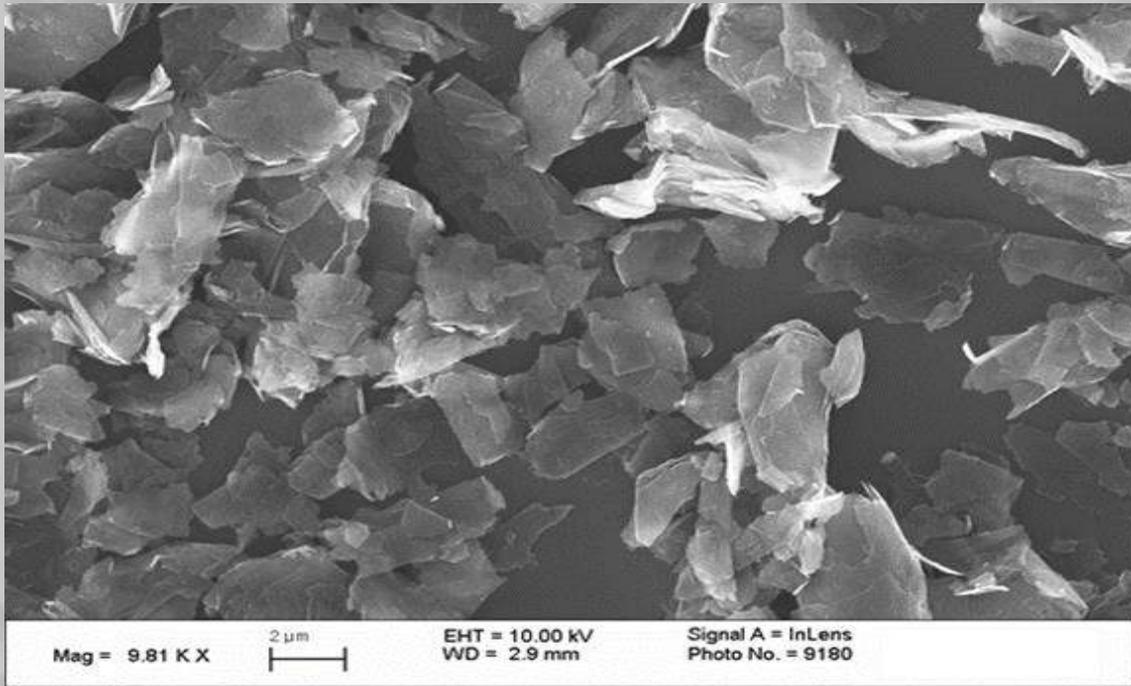


5.54 KX 2 μm EHT = 10.00 kV Signal A = InLens Date :25 Jun 2020
WD = 2.9 mm Photo No. = 9186 Time :18:32:22

Mag= 63.74 KX 300 nm EHT = 10.00 kV Signal A =
WD = 2.9 mm Photo No. =



Statistics: Flakes Lateral Size Analysis

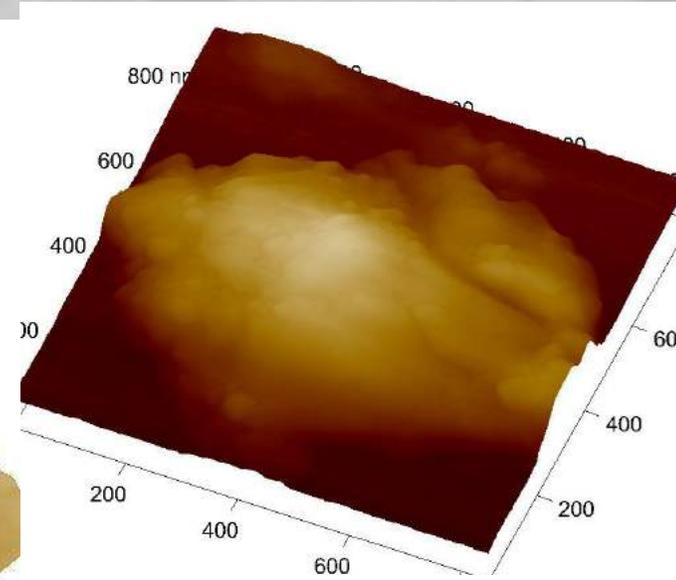
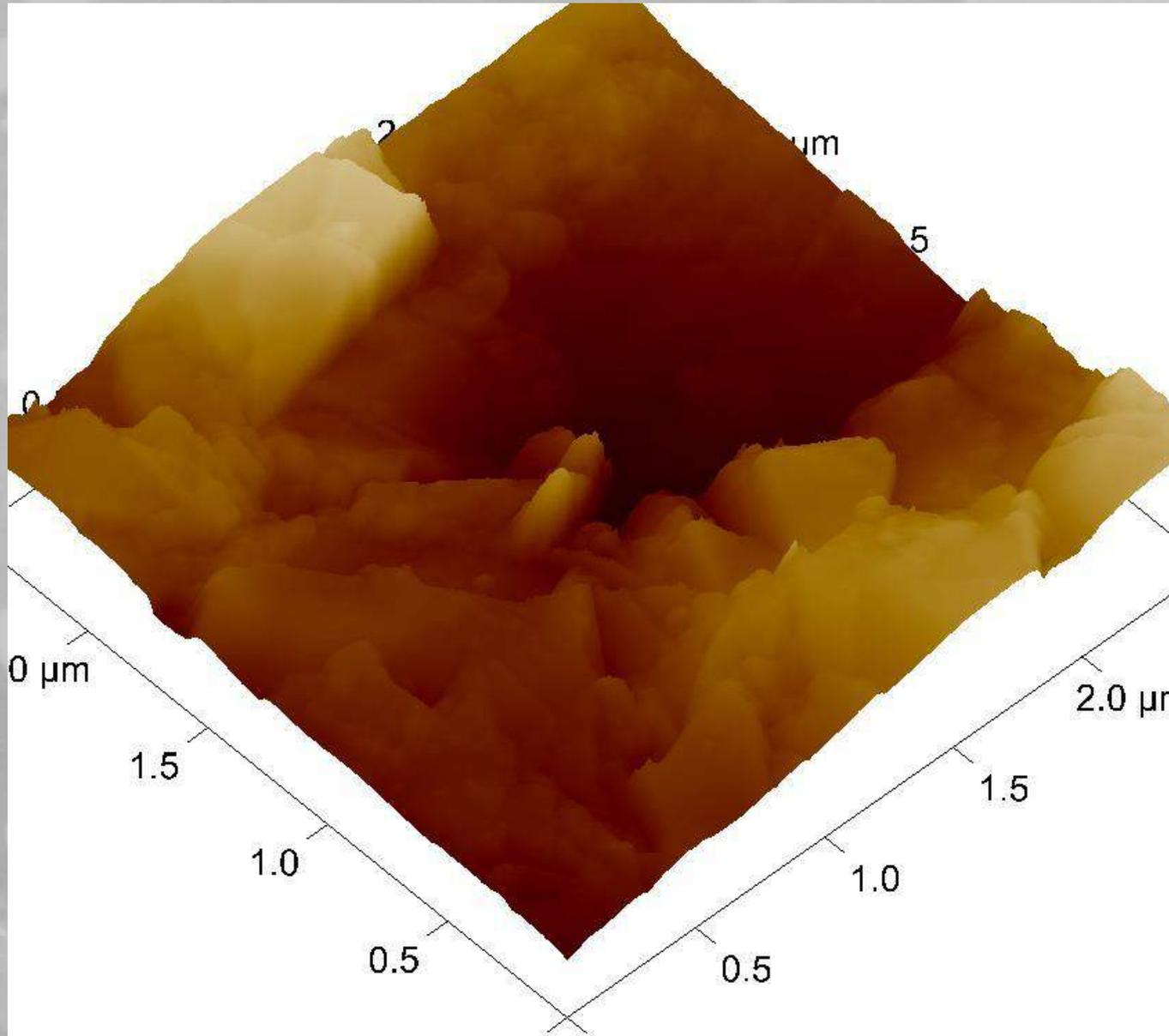


Total number of analyzed graphite flakes $n = 1200$.

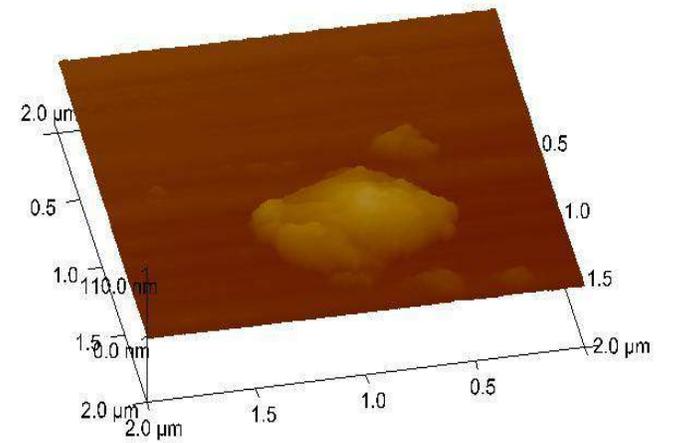
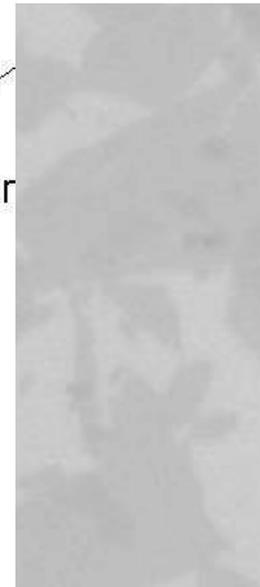
KEY POINTS:

- Flakes size varies from 0.2 to 14 μm .
- 90 wt % of the flakes has the lateral size between 1 and 5 μm .
- 80 wt % of the flakes has the size between 2 and 3.5 μm .
- Dominant lateral flake size is 2 μm .

Flakes Thickness Analysis: AFM study



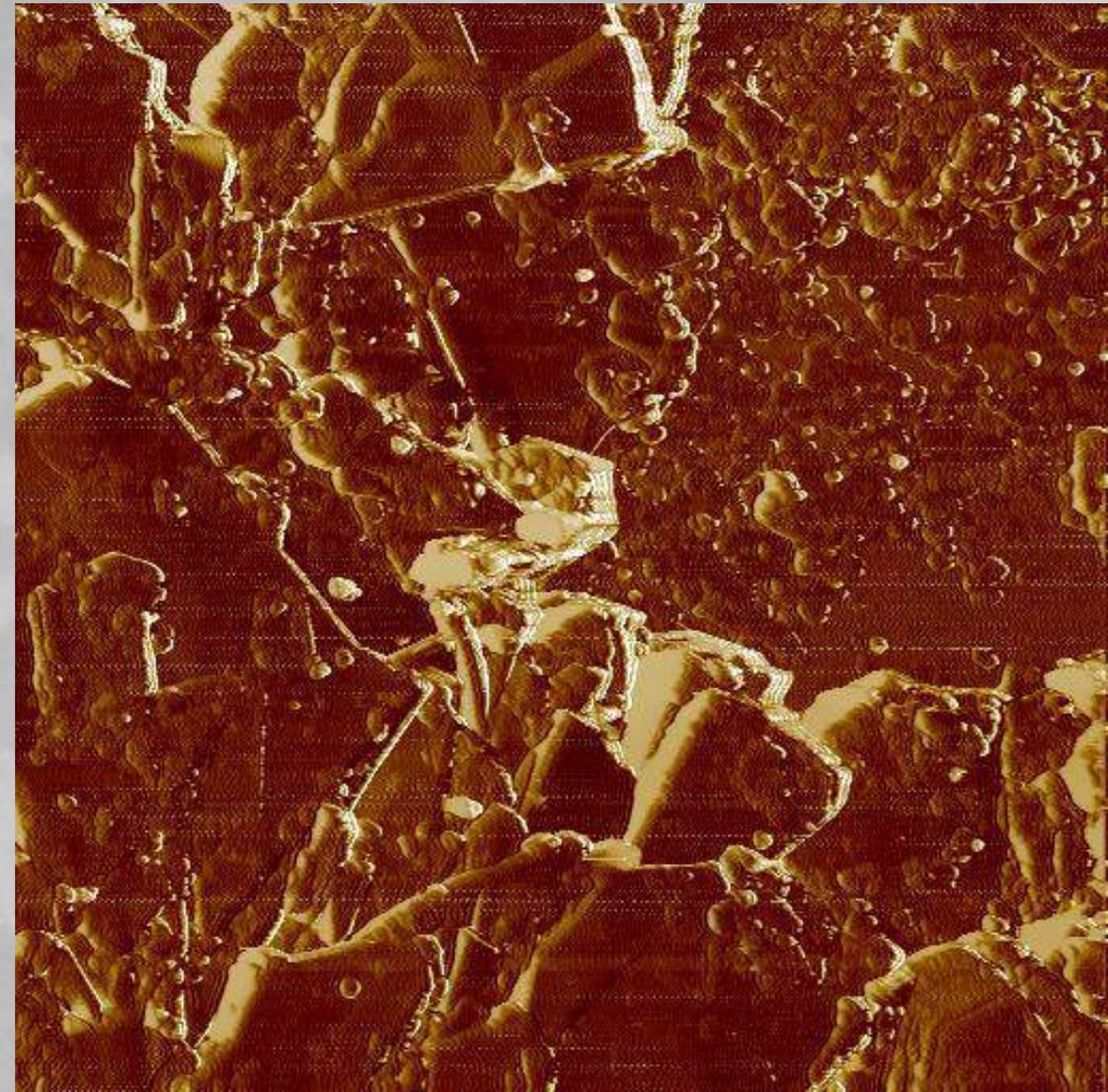
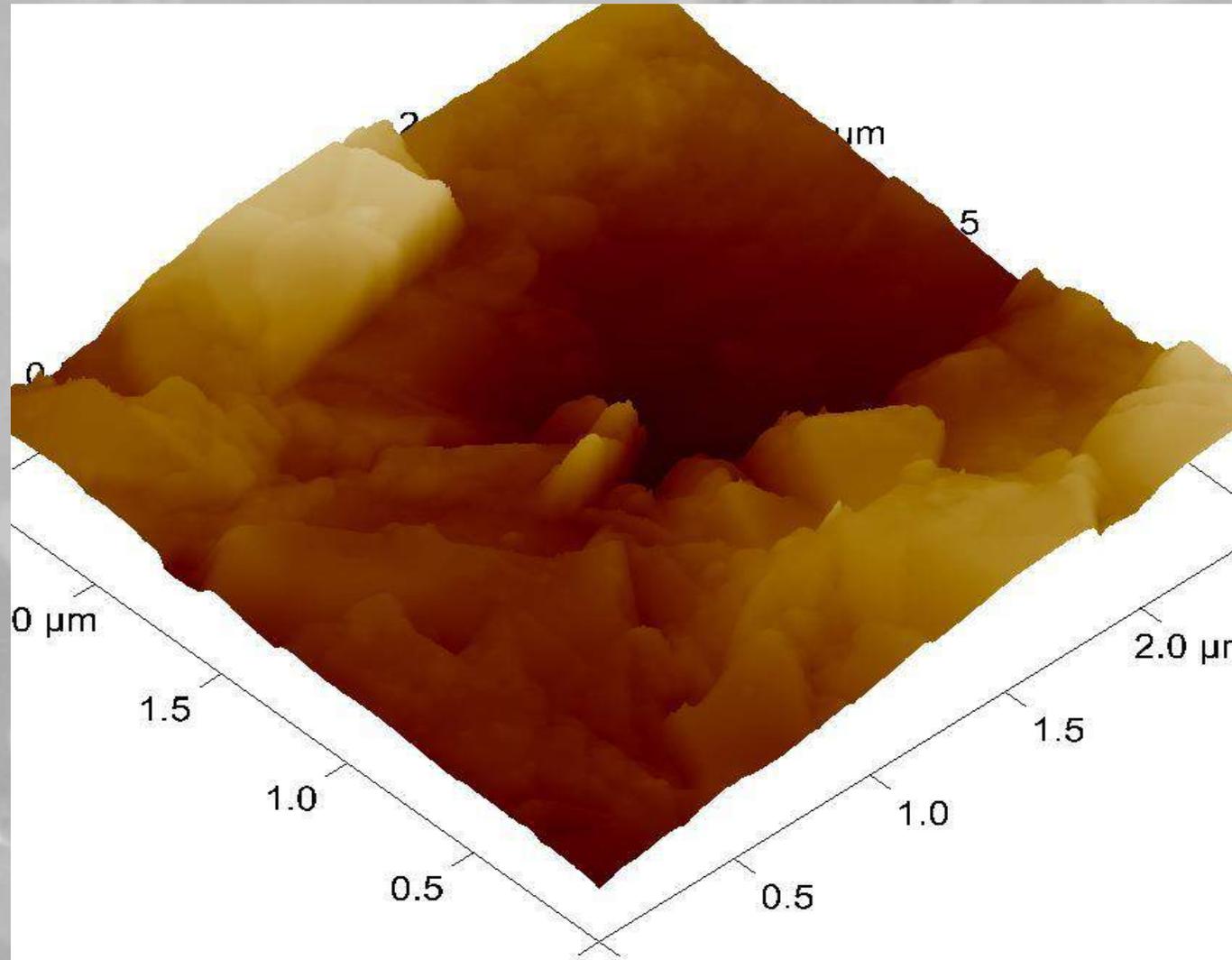
Three 3D scans were taken



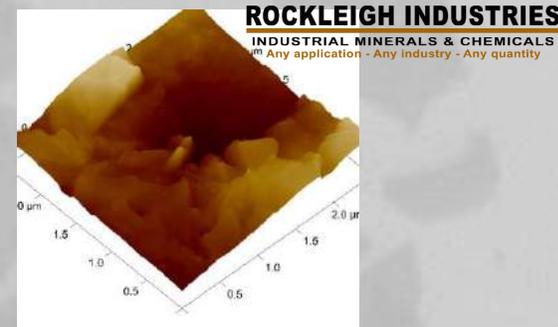
Flakes Thickness Analysis: AFM study

3D scan "Amplitude"

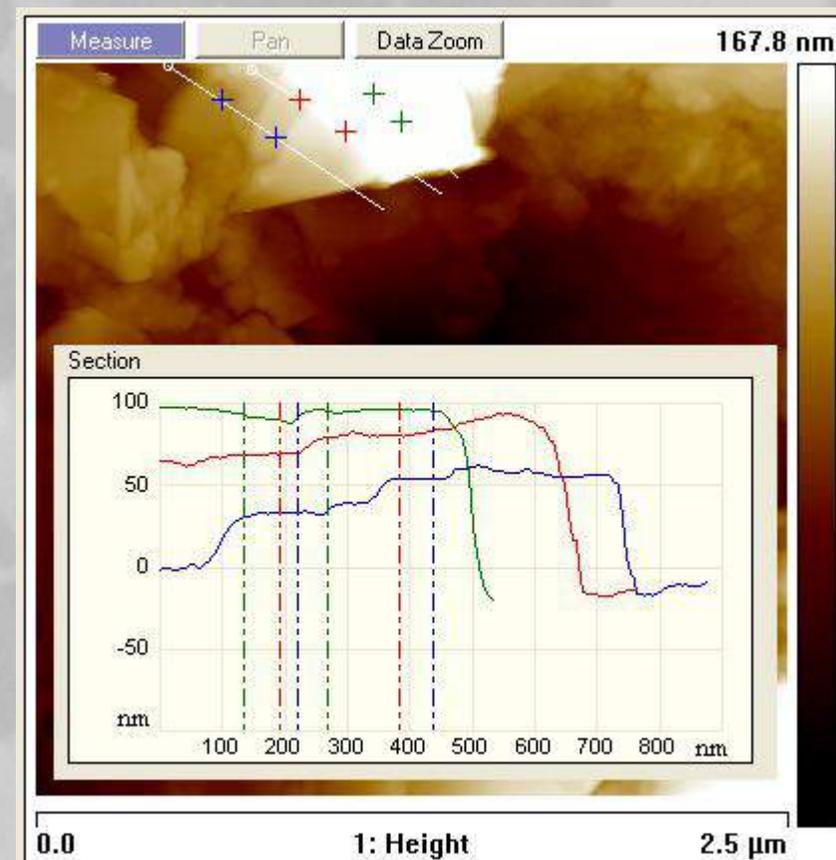
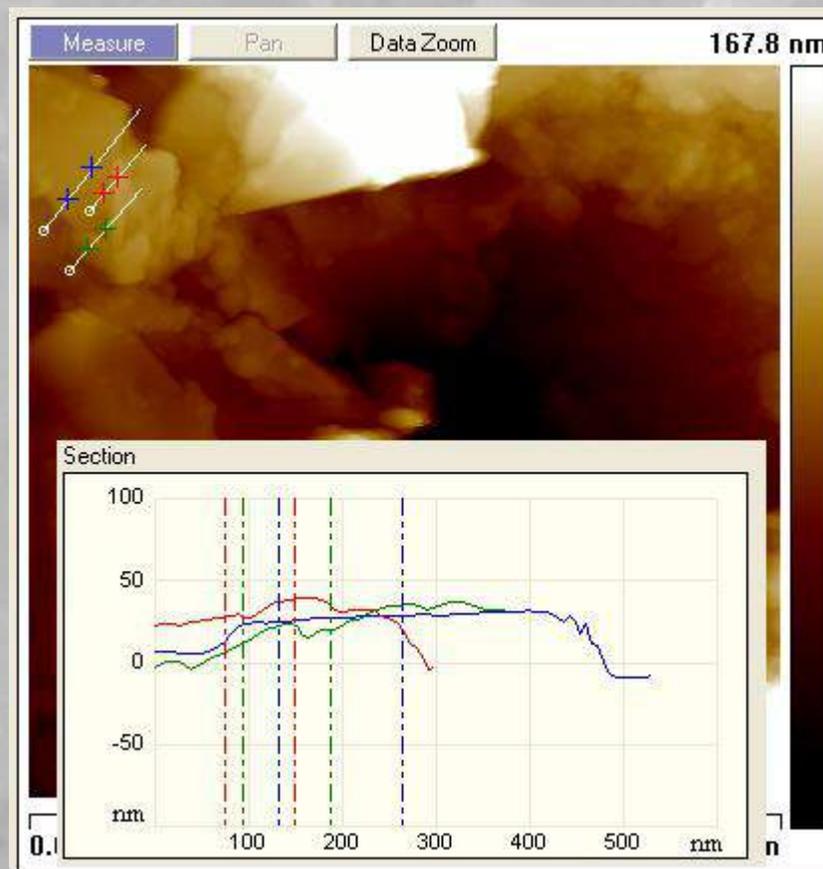
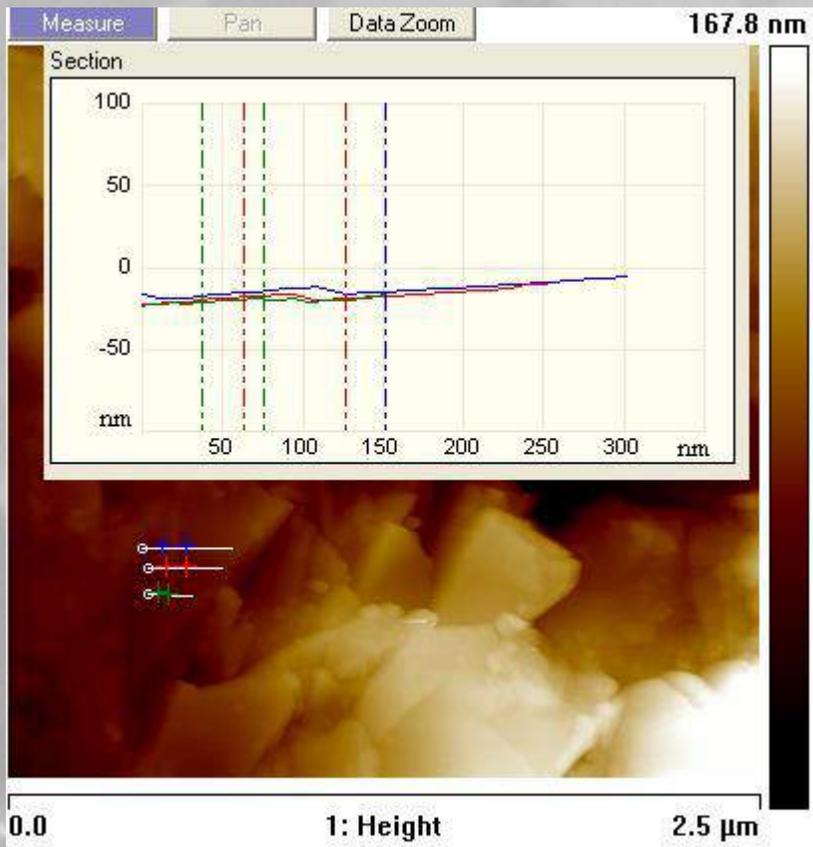
2D scan "Phase"



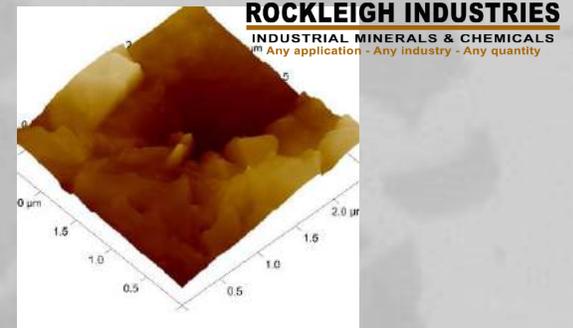
Flakes Thickness Analysis: AFM study



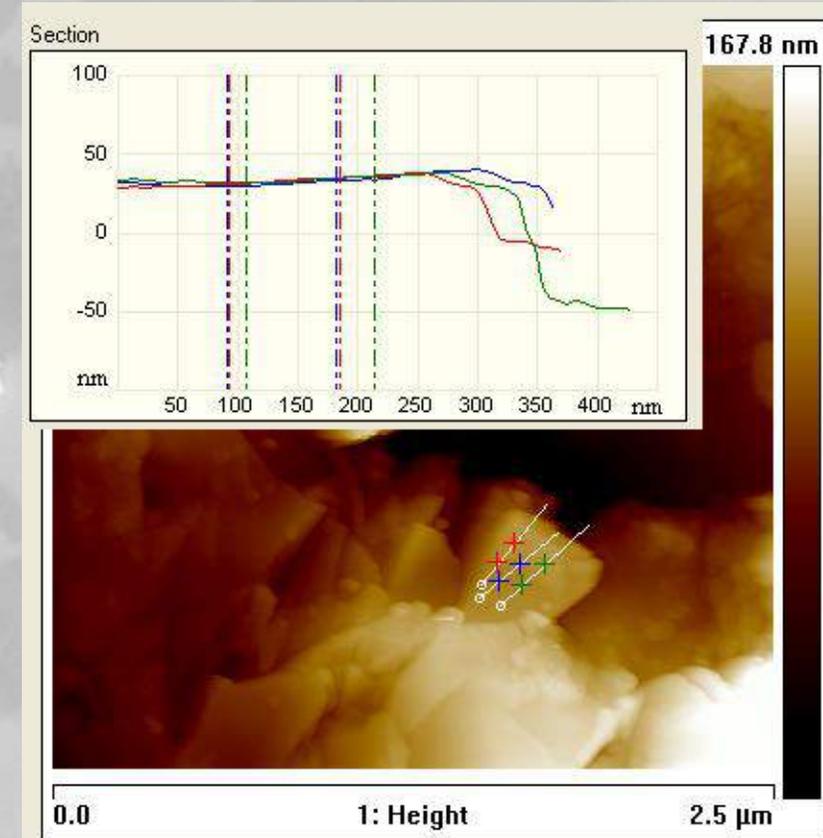
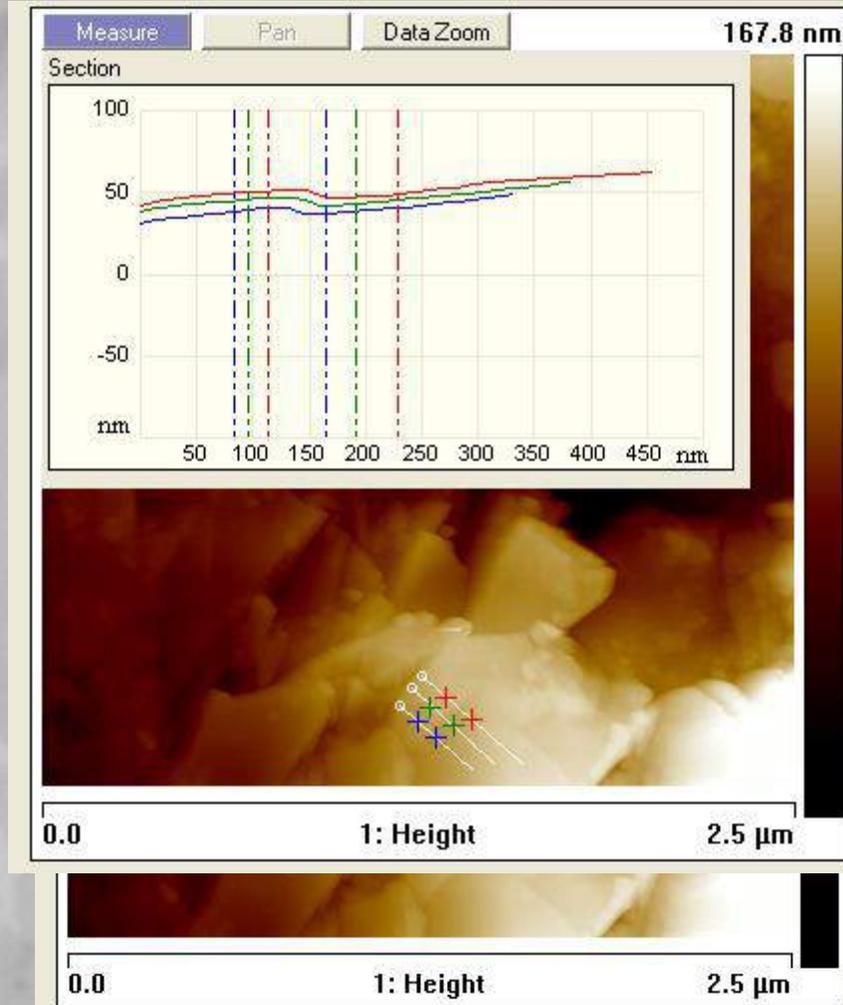
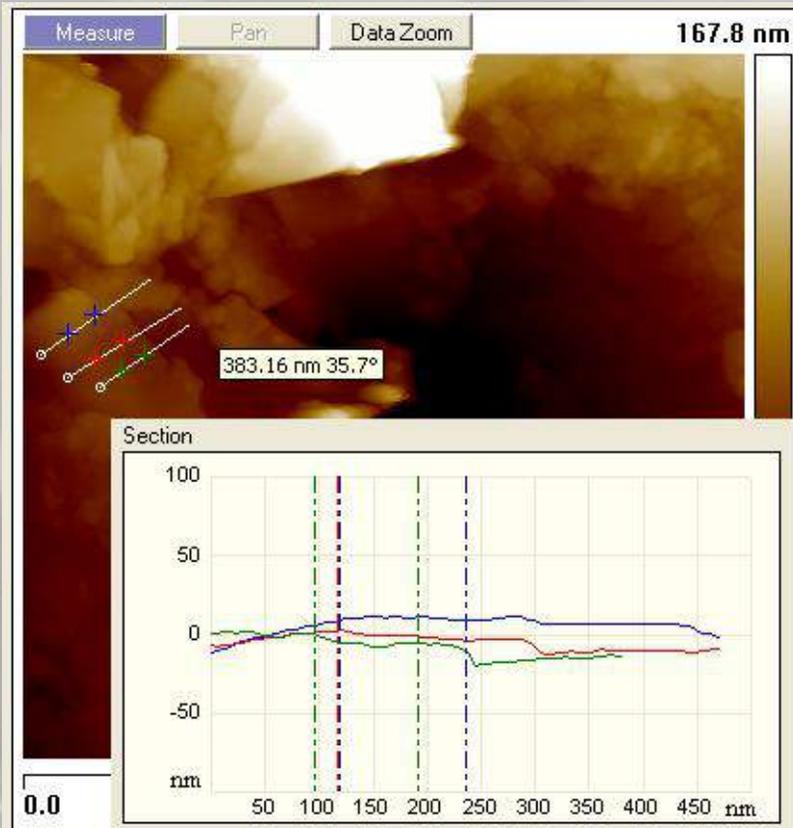
The same 3D scan was used to obtain cross-sections at different locations/flakes.



Flakes Thickness Analysis: AFM study

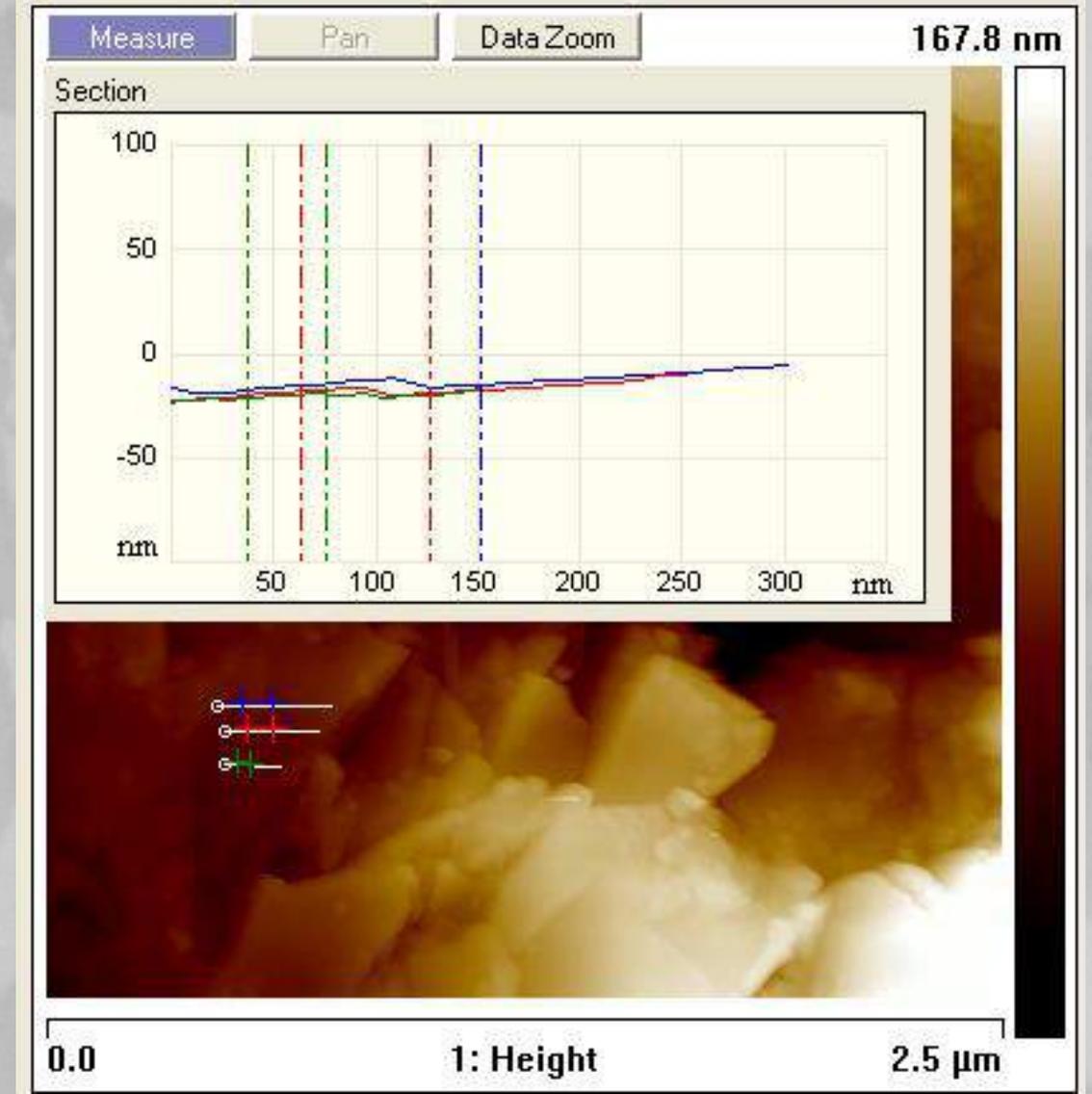
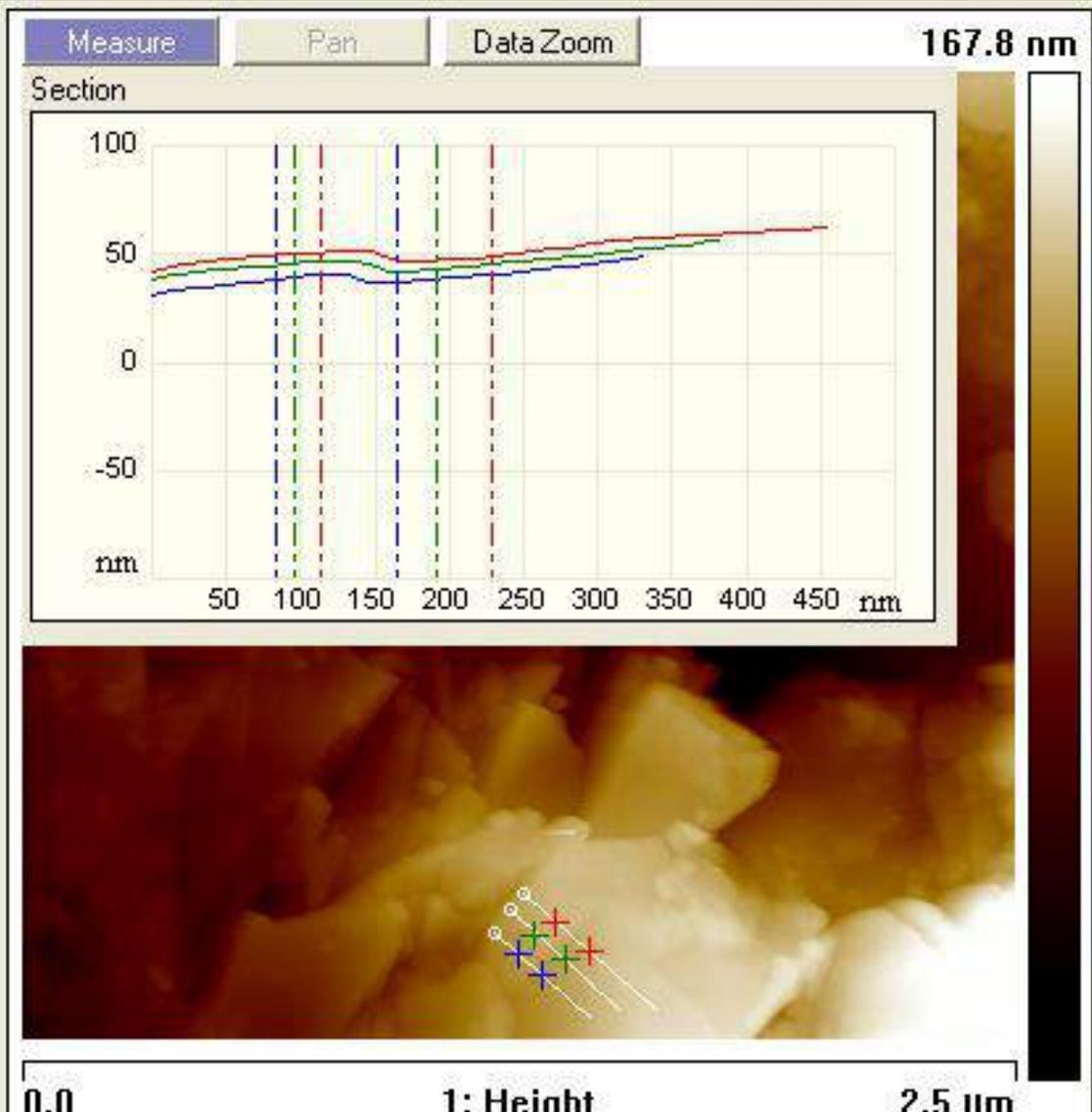


The same 3D scan was used to obtain cross-sections at different locations/flakes.



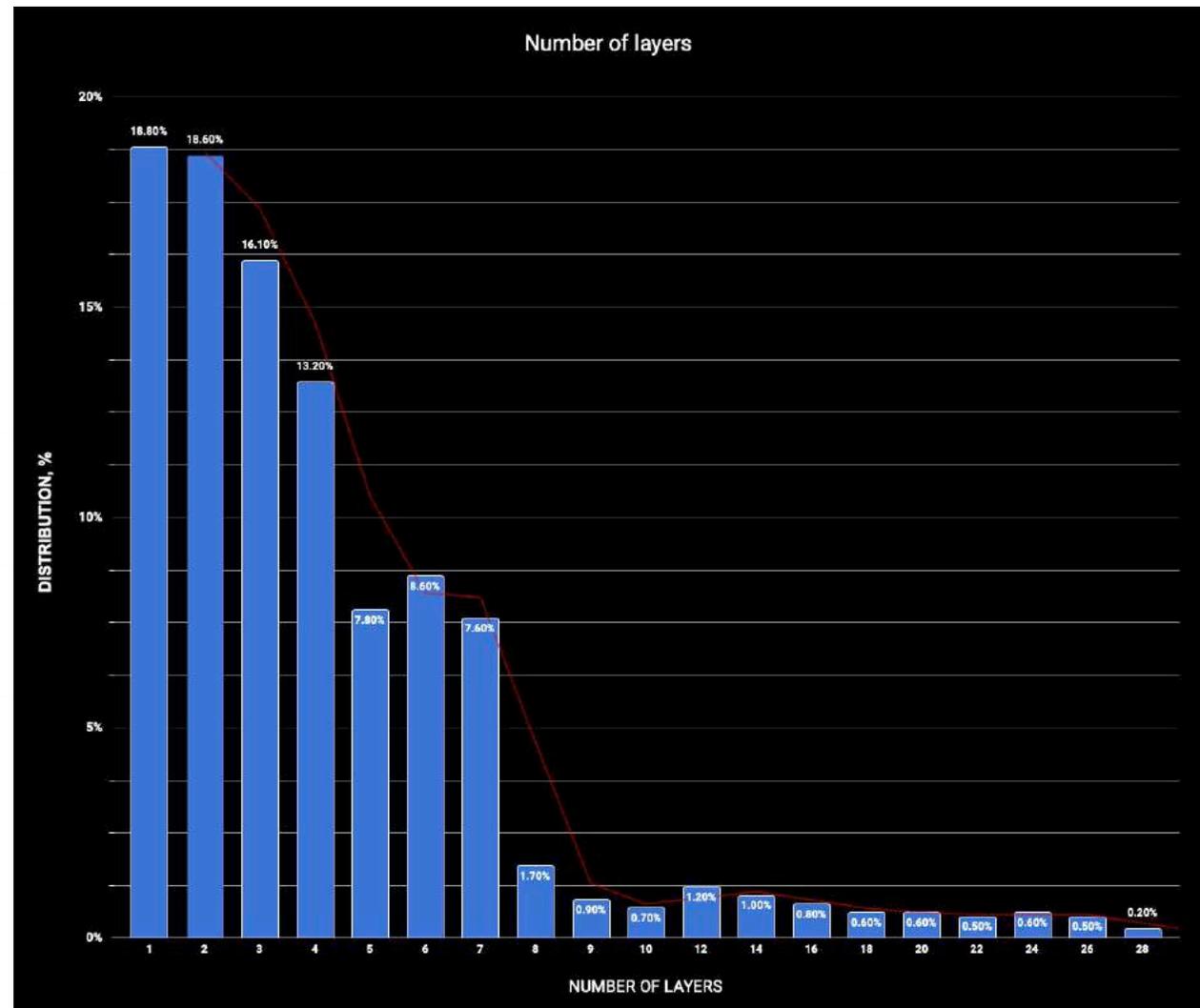
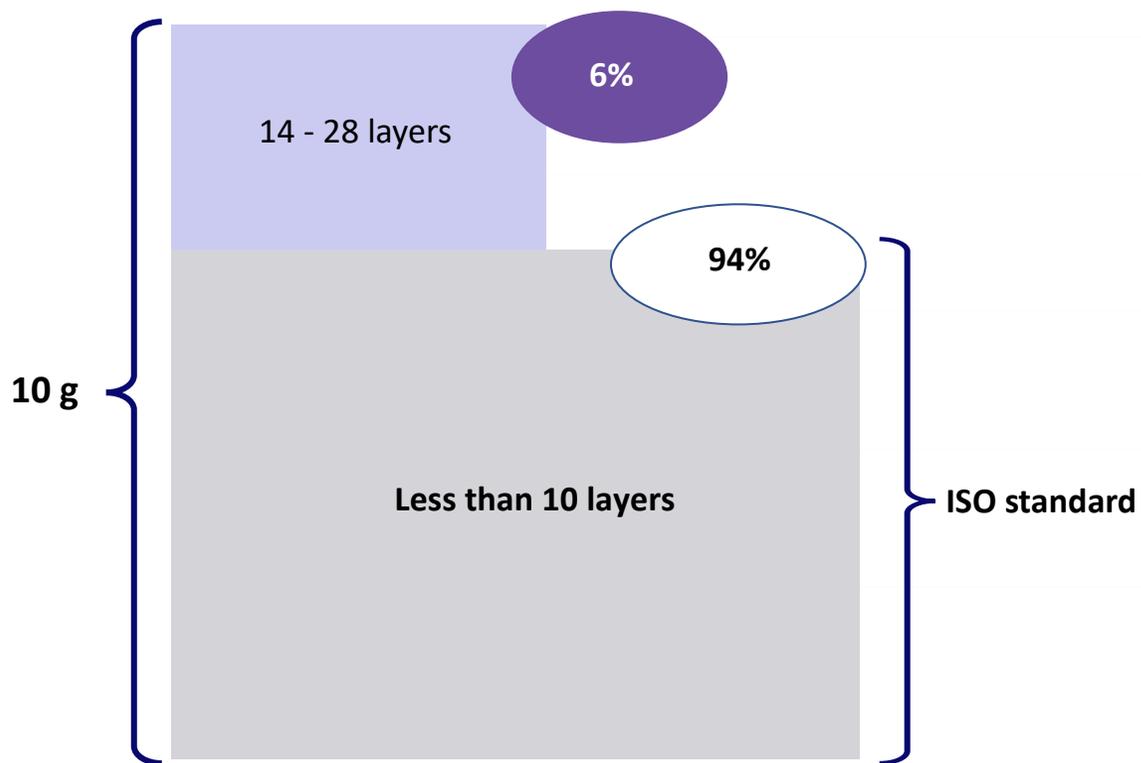
Flakes Thickness Analysis: AFM study

The same 3D scan was used to obtain cross-sections at different locations/flakes.



Summary of Flakes Thickness Results

Composition of incoming material



Potential of the material or what Rockleigh Industries technology can do for you As we demonstrated, 94 wt % of the material has the thickness less then 10 layers. Referring to the existing ISO standard, only this part of material can be called “Graphene”.