Exercise: Measurement of surface roughness using AFM

Tasks:

1.) Set up and calibrate the instrument on a glass sample (slide).
2.) Perform surface measurements on three different samples in contact mode with at least 2 set force values.
3.) Perform measurements on the same samples also in oscillatory mode.
4.) Evaluate the surface roughness using the Gwyddion software.
5.) Compare the obtained roughnesses.
6.) Discuss the results.

Keywords: Surface structure, AFM, roughness, contact mode, oscillation mode**.**

Introduction:

The study of surfaces is an important part of basic and applied research. The surface affects how the object interacts with its surroundings. It may involve frictional or adhesive properties, interaction with water, or other fluids, in biological applications, such as cell and tissue adhesion. The size of the studied structures on the surface can reach only tens of nanometres (in the extreme, even nanometers), at the same time, materials such as metals do not transmit optical radiation, which also limits the possible methods for studying surfaces. AFM is a very advantageous method in this area because it is relatively undemanding experimentally and offers excellent resolution. A method that also provides very good resolution is scanning electron microscopy (SEM), and it is to some extent complementary to AFM. For example, it does not allow for the determination of mechanical properties but it is possible to obtain information about the chemical composition of the surface layer.

Two main modes are used primarily for imaging surfaces using AFM. The first is the contact mode. In contact mode, the instrument scans the tip over the sample and adjusts the height of the tip to maintain a constant force between the tip and the sample. As this force depends on the distance between the tip and the sample, it generates a height map of the sample. The second mode is the oscillatory mode. In this mode, the tip is oscillated by the instrument at a frequency close to the natural oscillation frequency of the cantilever. The force interaction between the tip and the sample then alters the parameters of the induced oscillation, and the instrument adjusts the height again to maintain the oscillation properties at a set value.

In this exercise, we will be studying metallic samples with various applied surface layers. In numerous experimental and developmental applications, different methods are employed to apply a surface (functional) layer onto a substrate material. The macroscopic properties are then determined by the base material, while the surface layer contributes its own characteristics to the surface. This approach can be advantageous, for example, with fragile or expensive materials that possess desired surface properties. AFM is used to evaluate the resulting product, enabling the assessment of factors such as the presence of the applied layer (e.g., nanoparticles), the existence of clusters, impurities, surface homogeneity, and so on.

In our case, we will evaluate the surface roughness. This can be assessed both qualitatively (describing the presence of grooves, protrusions, their shapes, regularity, orientation, etc.) and quantitatively (density, frequency, measured dimensions). In addition, statistical processing of the measured surface heights is used for description. This can be done either on the entire sample (histogram), but typically processing is applied to selected curves. In the data set, one line (or curve) is chosen, and its height profile is processed.

The roughness parameter R_a is used to describe the roughness on the selected curve:

$$R_a = \frac{1}{N}\sum_{i=1}^{N}|z_i - \bar{z}_i|,$$

where N is number of points, z_i is the height in given point and $\bar{z}_i$ s the average height in the given dataset. There are also other roughness parameters, for example, R_q, defined as the square root of the average of the squared values:

$$R_q = \sqrt{\frac{1}{N}\sum_{i=1}^{N}|z_i - \bar{z}_i|^2}\,.$$

From the definition, it is apparent that these quantities may suffer from systematic error. If the sample is not orientated parallel to the measurement plane and the entire curve is slanted, the surface roughness will be falsely higher. At the same time, in the case where irregularities on the sample exhibit a certain orientation (e.g., regular long grooves), the choice of the curve can significantly influence the determined roughness. In the calculation of the roughness, a common approach involves subtracting a curve determined as the background before actual height processing. The choice of determining the background can again fundamentally affect the calculated value.

For a more detailed data processing, please refer to the Appendix.

Required equipment: AFM (Atomic Force Microscope), Inverted Microscope, CCD Camera, Samples.

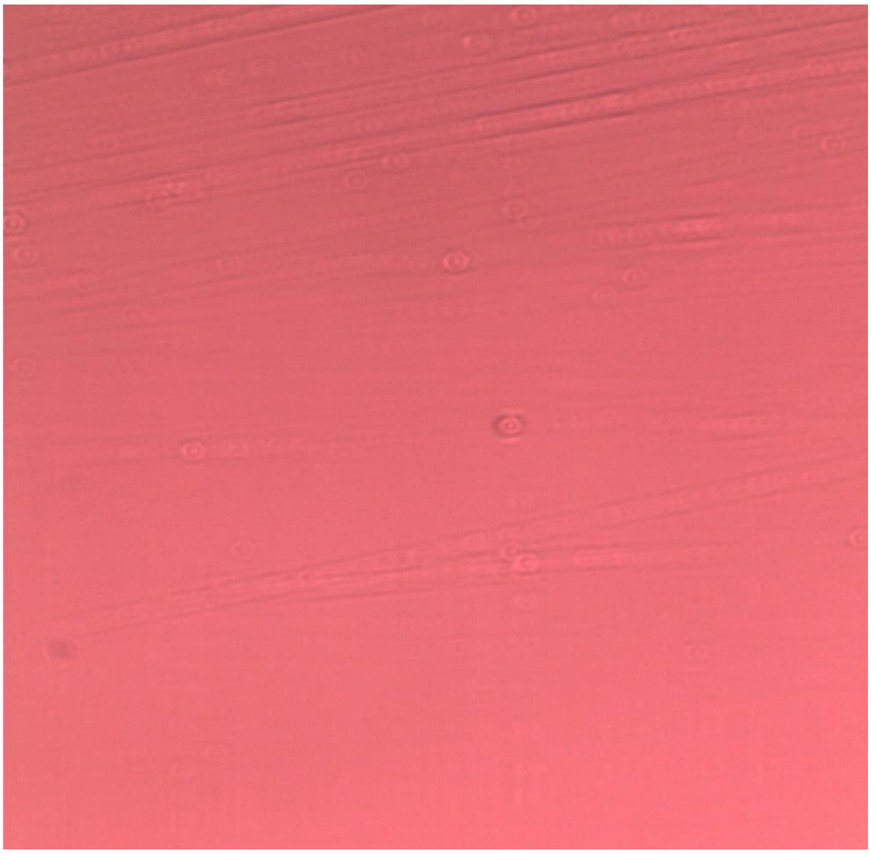
Instructions:

1. Prepare AFM.
    - Launch the SPM software on the measurement PC and the QuickPHOTO program on the microscope PC.
    - Install the cantilever with the tip into the holder.
        - Insert the holder from the cantilever installation kit, rotate it 90 degrees so that the upper surface of the holder is horizontal, and secure the holder.
        - Use tweezers to remove the cantilever and place it in the holder, securing it with a cross screw.
        - Proceed carefully following the instructions and guidance provided by the instructors.
        - Ensure that the cantilever does not fall out and that you do not touch it with anything, eliminating the risk of damage.
        - Before this procedure, practice the process using the damaged cantilever provided. Surface properties measurement using AFM.
2. Insert the holder into the measuring head.
    - Connect all cables.
    - Exercise caution to avoid damaging the tip
3. Perform cantilever calibration.
    - Place a glass slide in the measurement space.
    - Insert the AFM measuring head.
    - Adjust the laser beam to aim at the cantilever in the tip area.
        - Follow the manual.

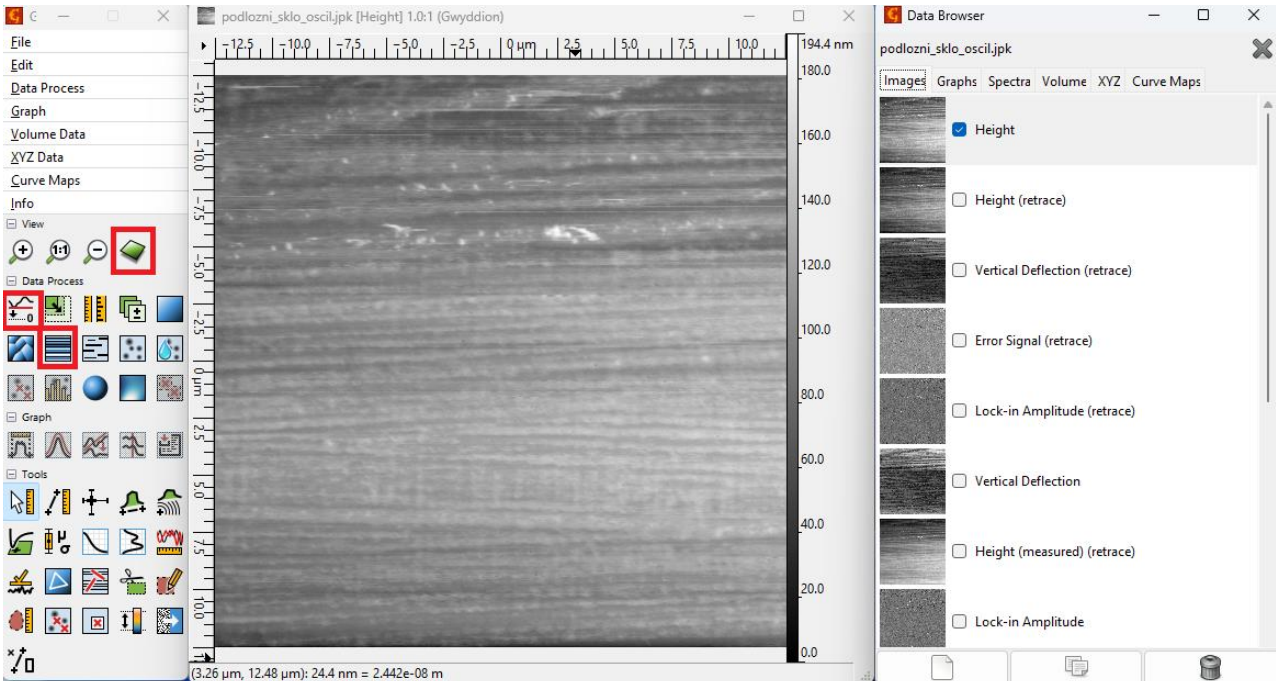
        - Monitor the beam on the microscope PC screen.
    - Set the detector and the mirror of the optical path to align the laser with the centre of the detector.
        - Follow the manual.
        - Monitor on the PC measurement screen.
4. Insert the sample either directly into the holder or attached to the glass slide.
5. If the saple is transparent, capture an image of the sample surface with the microscope.
6. Move the measuring head close to the surface. For transparent samples, do it under the microscope's control. For non-transparent ones, proceed very slowly under visual guidenace and initiate automatic descent in time.
7. Perform surface measurements using the appropriate mode in at least two different areas of the sample. In the case of contact mode, measure the same area with different force settings.
8. Save the measured data
9. Before leaving, turn off the PC and light sources
10. Work out a measurement protocol.
    - Choose an appropriate way to represent the measured data (height map, gradient map, 3D image) and discuss their advantages and disadvantages.
    - Analyze the surface structure based on the images (character, roughness, possible pattern).
    - Determine the roughness of the measured surfaces from the height maps of the samples in the Gwyddion software (see the appendix).
    - Results (Summary of experimental results).
    - Discussion and interpretation of results (Discuss how the results of individual samples differ from each other, any potential influence on the results, etc.)
    - Conclusion (Summarise the entire experiment)

Appendix: : Data processing in the Gwyddion software

Gwyddion is open-source software developed for data processing, including AFM data, and can be obtained at gwyddion.net. We will demonstrate its functions using an example of processing data from the measurement of the glass slide using the oscillatory mode. With the glass substrate, you can also examine the sample with an optical microscope (the microscope at the device is inverted, and non-transparent samples cannot be observed). The image shows a certain optically distinguishable pattern.

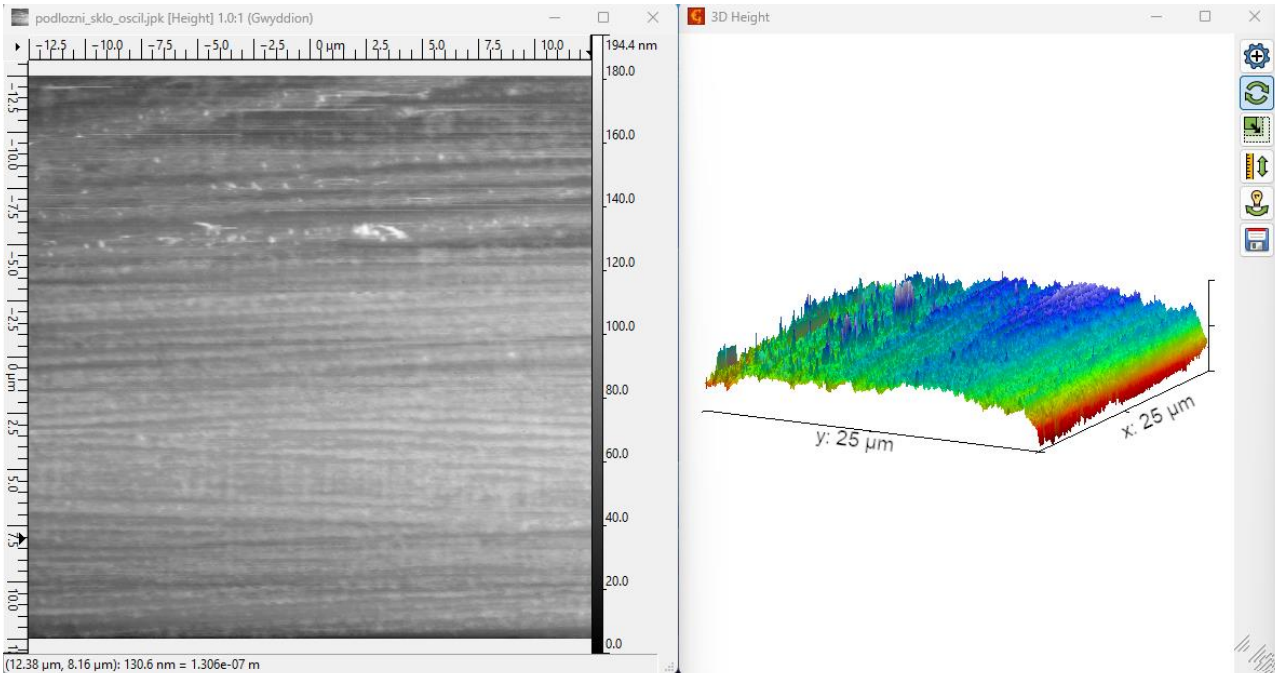


After launching the software, it is necessary to open a file with data (File > Open), and the height data will be automatically loaded.

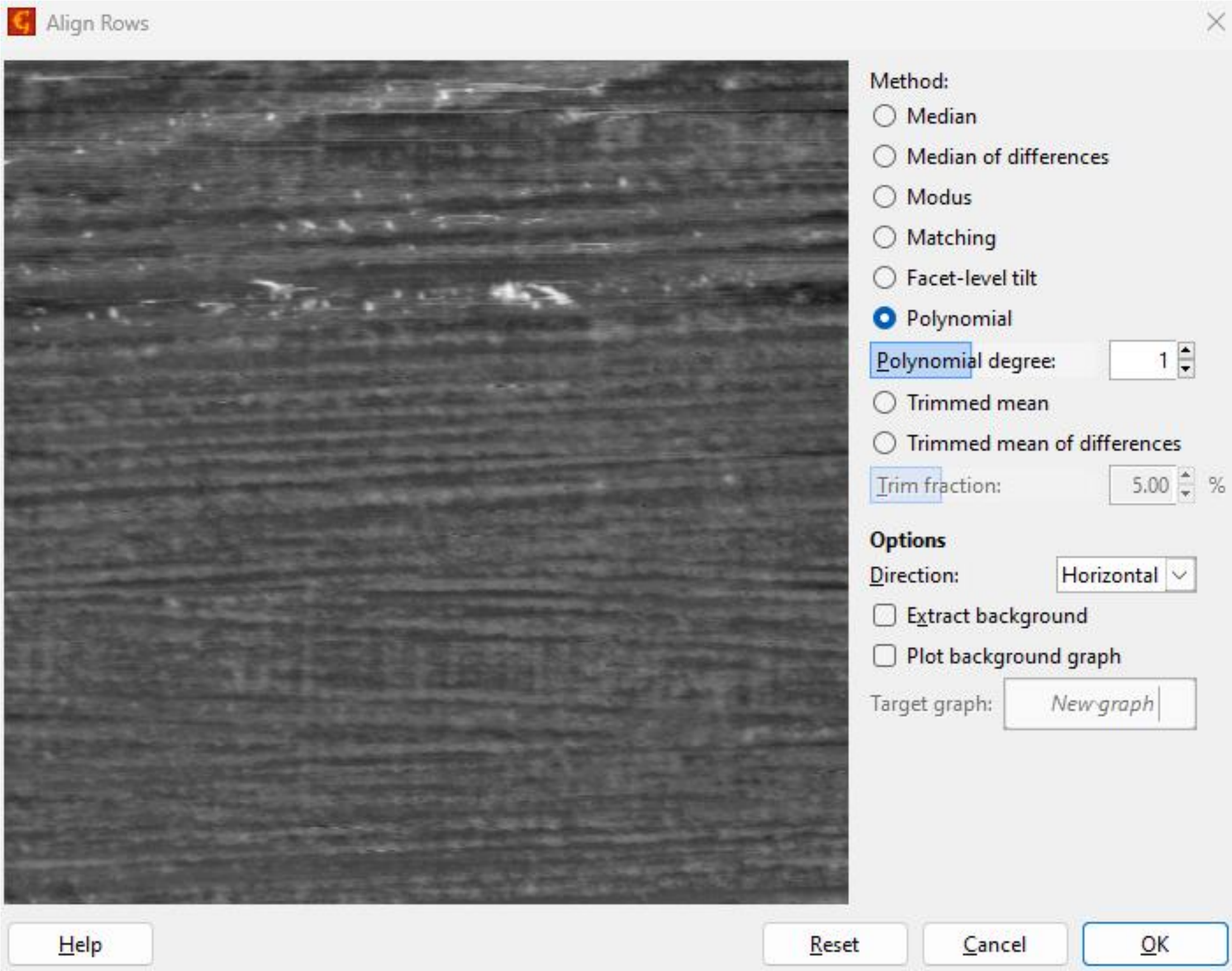


After opening the file, a surface map will appear in the middle, and on the left a list of recorded traces will open, which, however, will not be utilised in this task. The button allows displaying a 3D representation of the surface (after right-clicking on the image, it is possible to change colours and lighting and shading types).

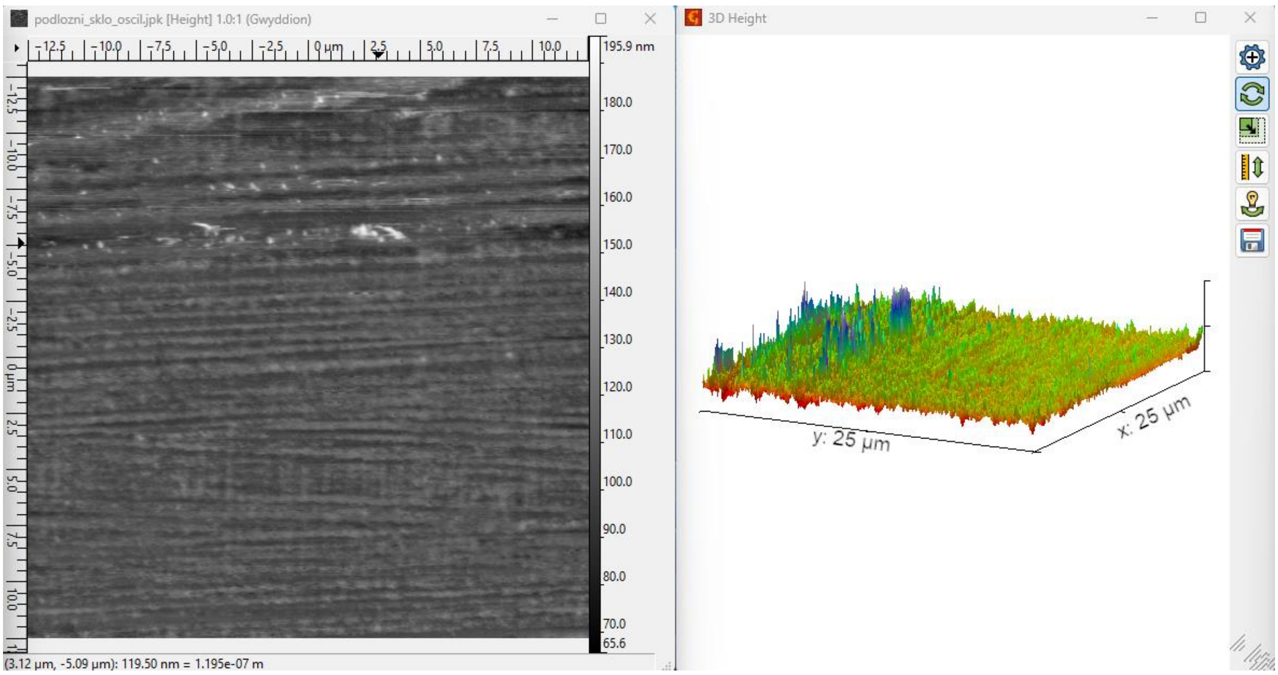
After displaying the 3D model (and adjusting the colour scale and lighting), we observe the following.



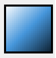
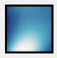
It is noticeable that on the height map at the bottom and on the 3D representation on the right, the surface of the sample sharply drops. Another parallel undulation continues further in the obtained image. This undulation is parallel to the scanning direction of the microscope and does not represent real shape of the sample surface, but rather a changing offset in individual measurement lines. This error needs to be compensated for (if the undulation did not run parallel to the data collection, it would be more indicative of actual surface roughness). Compensation for this phenomenon is carried out using .]. After pressing, a window with method settings will open. It is advisable to try different variants, and the image represents the data after adjustment.



After applying the filter that levels the offset of individual lines, the previous image looks like this:

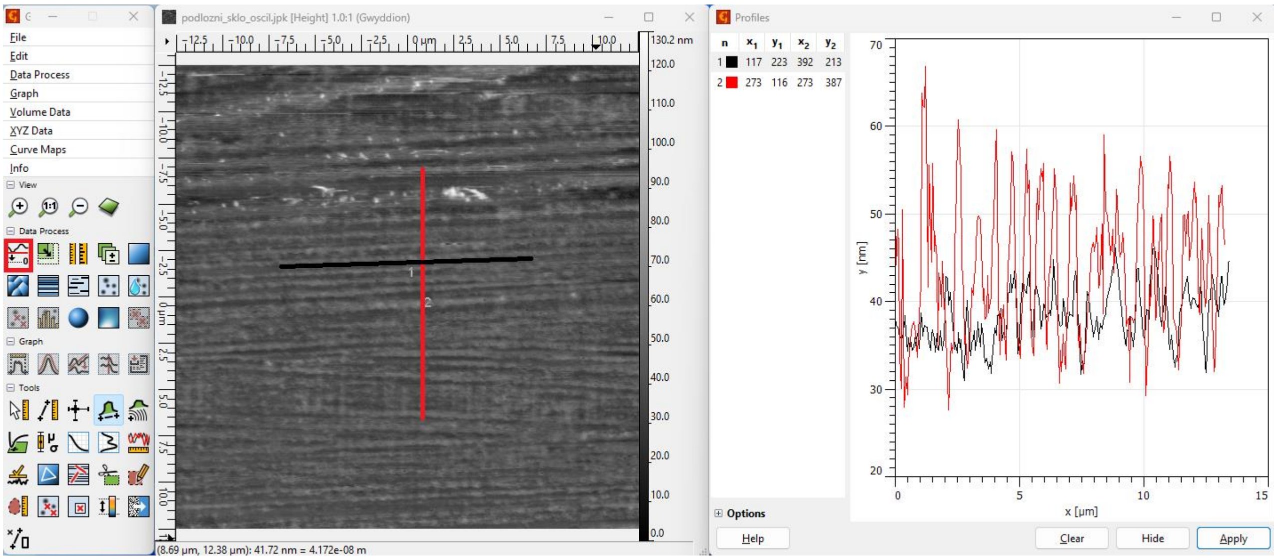


It can be observed that the surface undulation has been smoothed. The difference in the surface map is not as pronounced. In general, the surface map is not very sensitive to various changes, and it is better to use the 3D model.

It may happen that even after aligning the lines, surface undulation still occurs. This situation arises if the measured surface is genuinely undulated or is not parallel to the measurement plane. This can be further compensated using tools  (Level data by mean plane substraction), which automatically fits and subtracts a plane, or  (Remove polynomial background where a polynomial of a selected order is subtracted.

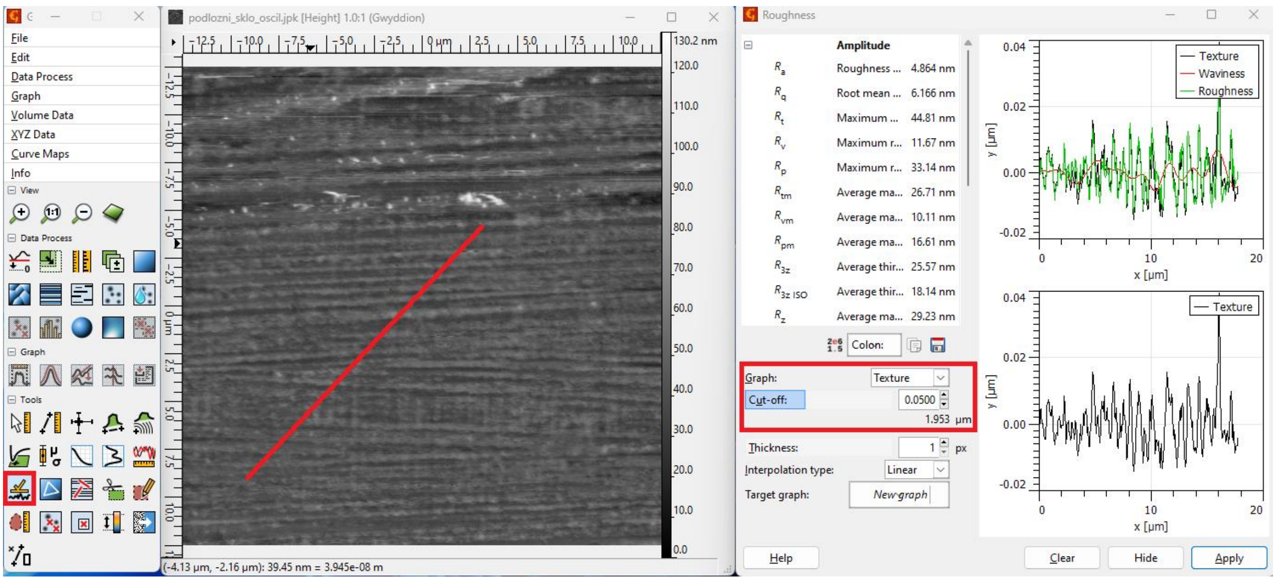
Once we have aligned the data, it is also advisable to use the , "Shift minimum to zero" function, which shifts the lowest value to 0 (so far, the value was measured by the instrument), for better orientation.

Now, using the function  we can look at the slices of our image.

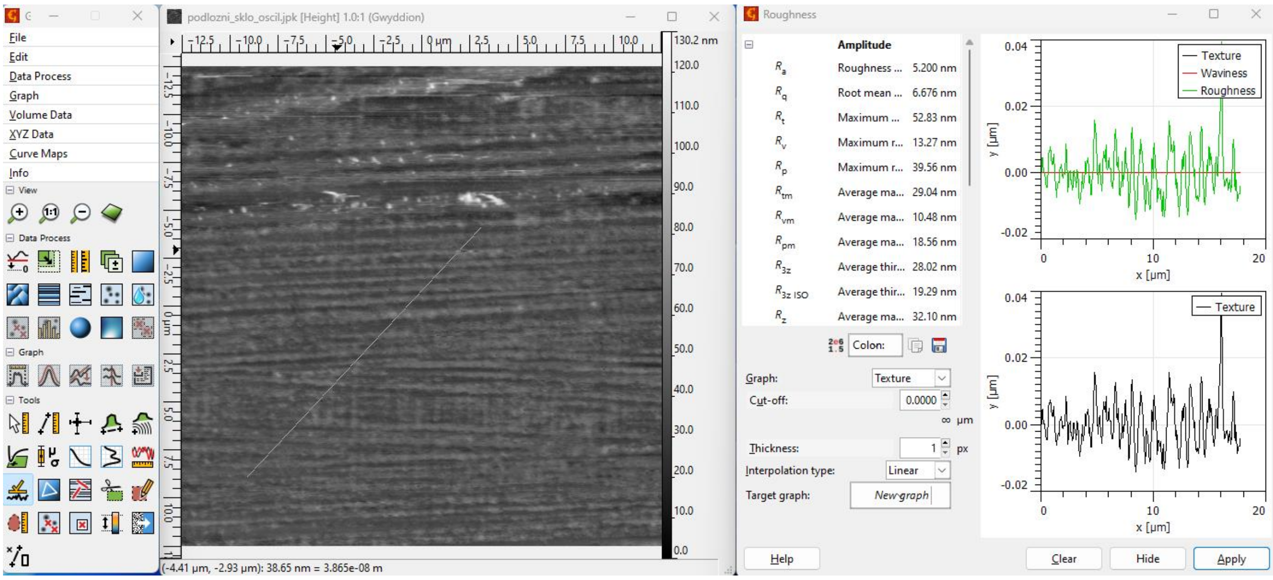


We select the line by a simple marking on the image, and its profile is displayed in the second window. In the image, it can be seen that the red line has a much more rugged profile than the black one, which is oriented in the direction of surface roughness (colors are used for clarity; otherwise, the lines are marked with numbers). This, of course, will affect the roughness analysis. The selected image can be exported by choosing the "Apply" option.

For the roughness analysis, we will use the function .



Once again, it displays and analyses the selected curve. The calculated roughness parameters are shown in the dialogue. On the upper graph, the surface (black), the subtracted background (red), and the surface after background subtraction, from which the roughness is calculated (green), are visible. However, in our case, we already know that the surface is compensated for roughness, and we do not want to subtract the background because it is the studied structure of the sample. In such a case, it is necessary to set the cut-off value to 0.



In the image, it can be seen that the subtracted background is now zero. It is evident that the data processing procedure can significantly influence the result obtained. Therefore, it is necessary to consider what adjustments to make and to process the samples consistently to enable comparisons of results.
The described functions are the minimum necessary to process the protocol. Gwyddion offers a wide range of statistical and other functions. For further information, you can visit the project's website and refer to the documentation.