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Evaluation of atomic force microscopy measurements of small regular and non-regular particles using Gwyddion software

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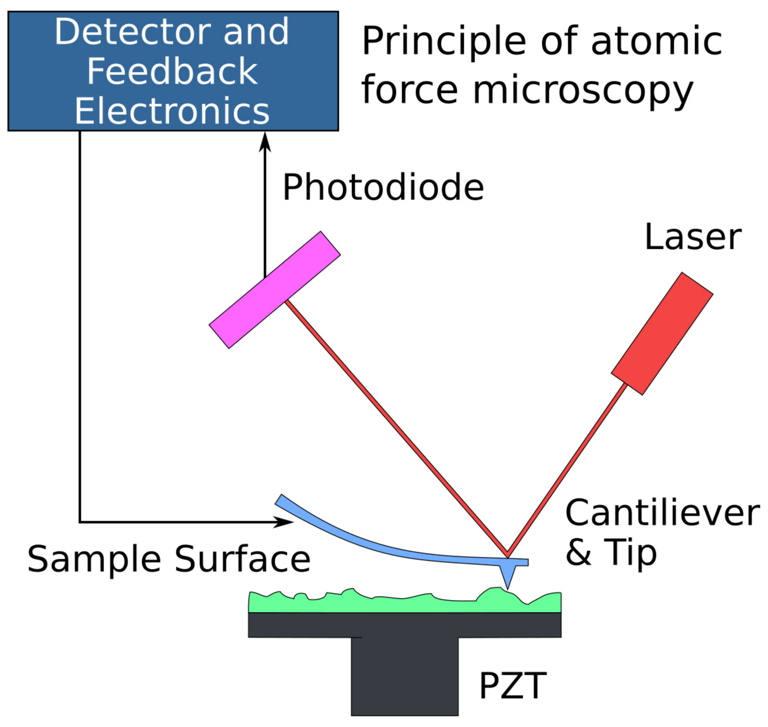
Introduction & Motivation

Atomic Force Microscopy (AFM) measurements are not very often used for characterization of solid state aerosol particles because other techniques, such as light scattering, are usually more suitable for their routine large-scale evaluation. However, when we consider precise characterization of aerosol particles, individual particles, or non-spherical particles then atomic force microscopy can be the right choice for measurement of particle properties.

In real situations, for nanoparticles on rough substrates or for nanoparticles that are not isolated, non-trivial evaluation of AFM measurements in image processing software is usually needed (Klapetek, 2011). The aim of this work is to investigate the different ways of AFM data evalutation and related issues (aspects of different evaluation approaches, influence of particles shape on the measurement, measurement uncertainty, etc.). We present real AFM measurements and results of numerical modelling in Gwyddion open source software, see http://gwyddion.net/ or (Nečas, 2012) for more details.

Measurement



Samples for real AFM measurements were particles dispersed on microscope cover glass in water that was later evaporated. Used particles are following: (i) Nanosphere TM Size Standards (NIST traceable) polymer microspheres, nominated diameter (100±3) nm and (296±6) nm, (ii) gold nanorods Sigma-Aldrich 771651, 25 nm x 47 nm - D x L (10 % uncertainty).

AFM measurements were performed using Bruker Icon instrument, experiment mode: PeakForce QNM in Air, used probe: ScanAssyst-Air.

Algorithms for particle measurement

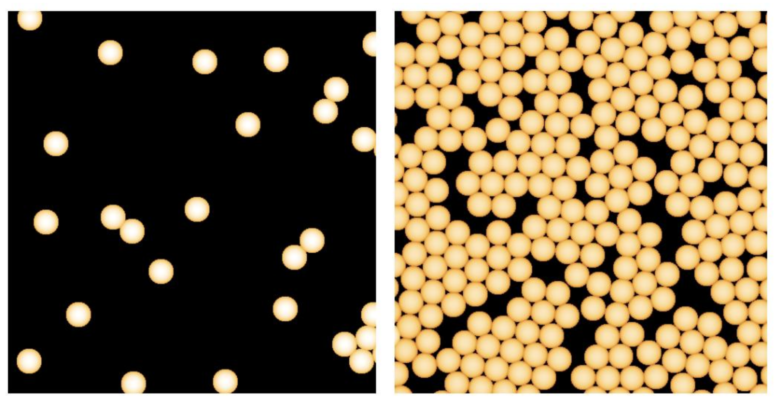
- Manual & individual: human influence, time demanding, low number of particles.
- Image segmentation and statistical analysis: large number of particles analysed, subject to errors related to wrong image segmentation e.g. due to overlapping particles.
- Line profile analysis: suitable for perfectly packed monodisperse particles only, not in this case.
- Fourier analysis: provides mean particle diameter only, best for high surface coverages.

Methodology

To test how different techniques are suitable for polydisperse particles analysis we have used the following steps (all in Gwyddion open source software, with help of data synthesis modules):
1. Simulation of particle deposition on the surface, different surface coverages.
2. (optional) creation of virtual AFM images by tip-sample convolution.
3. nanoparticle statistical analysis using virtual AFM images and data processing software.

Results

Monodisperse particles modelling



Example of simulated virtual images with two different coverages: 0.1 (left) and 0.8 (right).

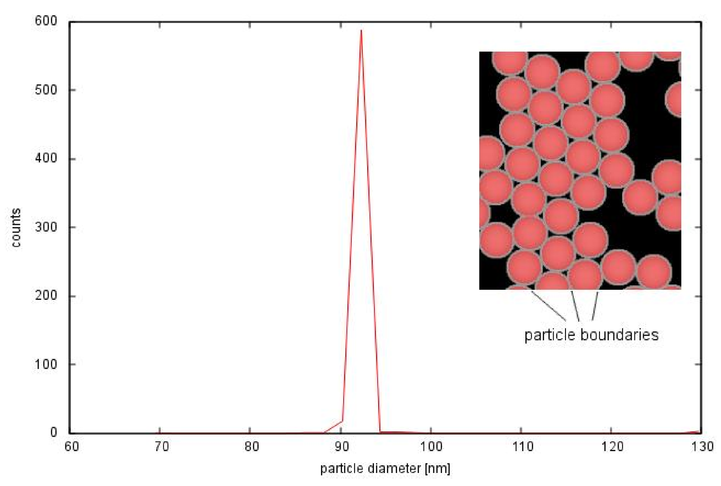
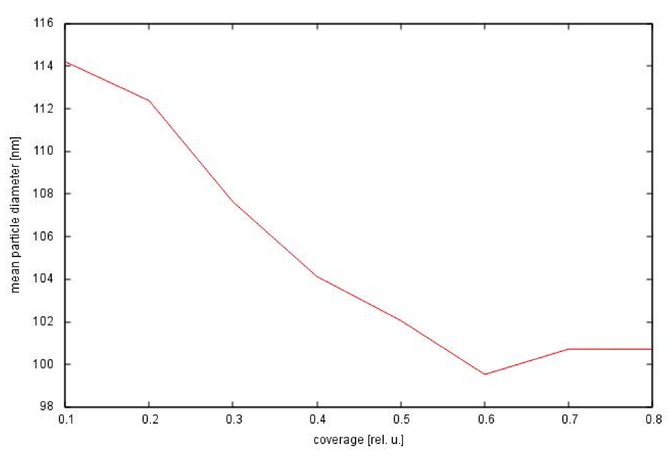
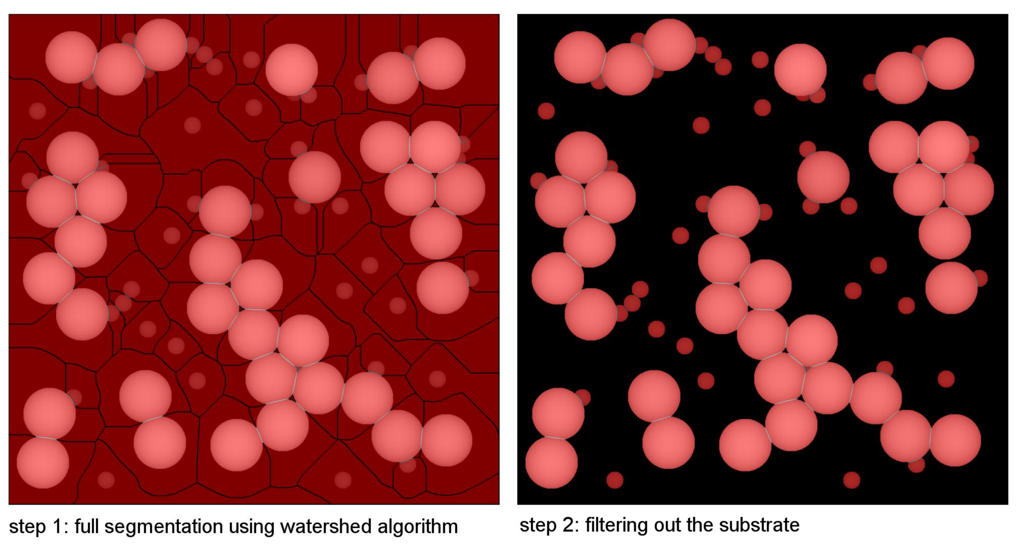


Image segmentation approach using watershed algorithm, this method is almost insensitive on surface coverage, however in general has tendency to underestimate particle size



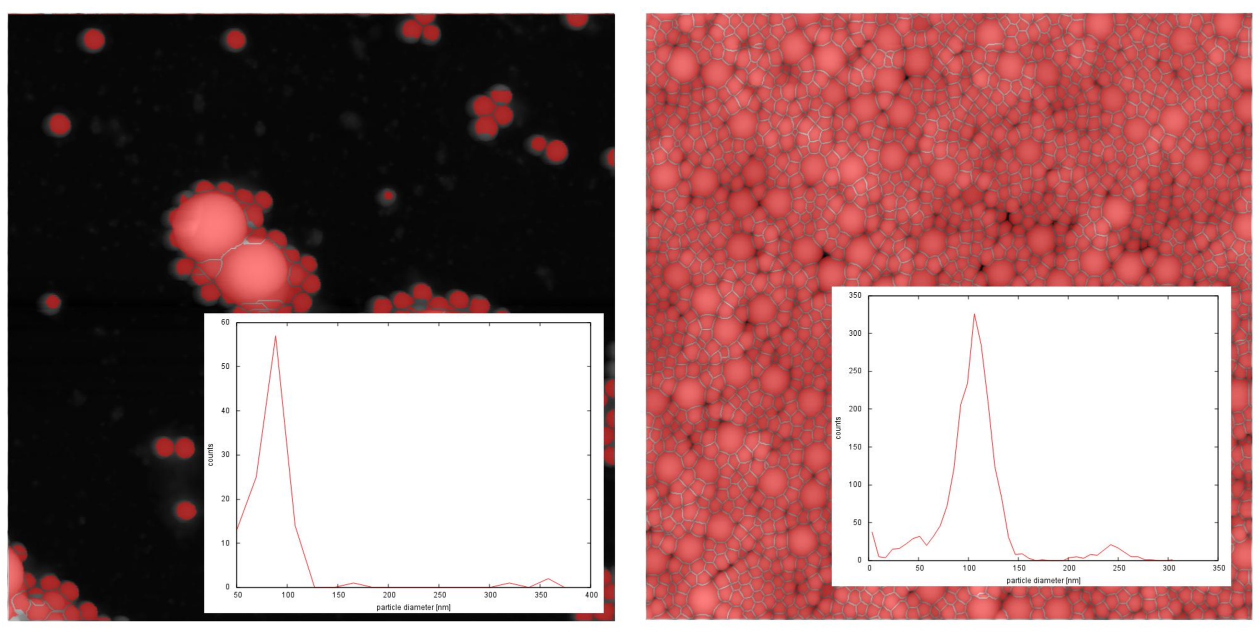
Fourier analysis approach. Method works best for high surface coverages and in general overestimates the particle size.

Polydisperse particles modelling



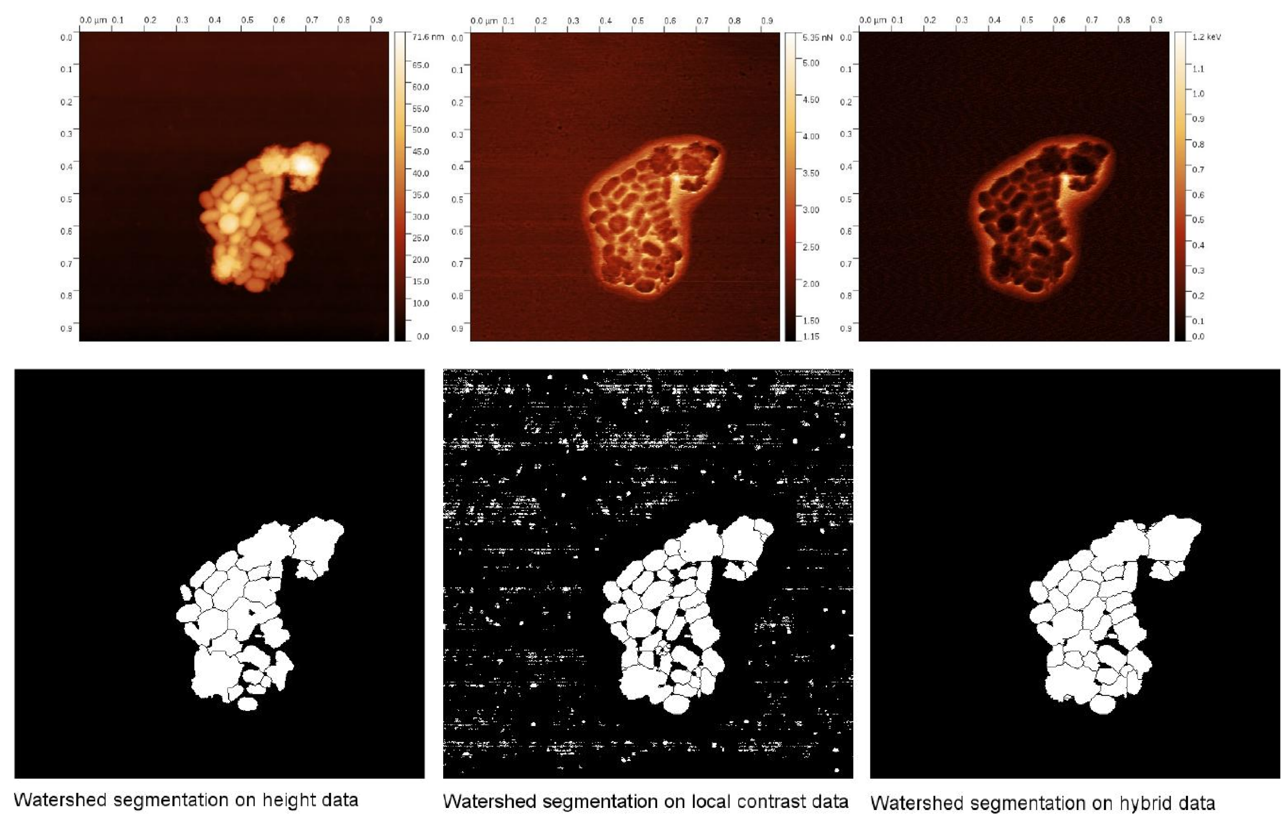
One of multiple options how to segment the data – using full watershed segmentation with thresholding. Standard thresholding does not work for disperse particles and watershed in its simplest form does not work as well.

Application to real AFM data



Analysis of mixture of 100 nm and 296 nm particles. Small coverage (on the left) and high coverage (on the right) results obtained via watershed algorithm segmentation.

Application to real AFM data: non-spherical particles



Analysis of gold nanorod measurement. Segmentation was done manually choosing particles for calculation, this includes human influence, which is not optimal from metrology point of view. Resulting particle size is 40±8 nm for the minor axis and 73±9 nm for the major axis.

Conclusion

- Fourier analysis is not well suited for polydisperse particles analysis.

- Large pixel area of individual grains is necessary to get good data from segmentation based methods, even if this would be obtained artificially by upsampling measured data.

- The polydisperse particle sets can be analysed by segmentation methods.

- Uncertainties in few percents are common, for non-ideal cases tens of percents.

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