Scale-depended Choice of Scanning Rate for AFM Measurements

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Abstract. Three samples with different high of topography features were measured at different scanning rate. We also presented statistical and fractal analyses for definition of the surface morphometrics. They can be used for calculation and evaluation of the images' distortion that takes place during scanning rate and proved to be helpful while controlling the measurement. Basing on the results we came to the conclusion that fractal analysis, the statistical surface roughness parameters and AFM may provide us with a deeper understanding of the physical phenomena taking place in the sample-tip interface. This is why, fractal analysis and statistical surface roughness parameters are useful information for the further improvement of calibration system. This approach can be applied for choosing scanning parameters properly, taking into consideration the geometry of the sample and for the microscope calibration by geometrical sizes of features.

Introduction

The accurate estimation of geometric sizes is one of the problems of nanometrics [1-3]. Chemical, mechanical and electrical properties of the elements are affected by surface condition [4-8]. This is why the accurate surface examination is an important task at number engineering areas [9-14].

Surface topography can be studied by many methods and microscopy techniques [15-19]. But none of these methods is free of its own set of artifacts and that must be taken into consideration during data evaluation [20-24]. The reasons of the occurrence of the measurement artifacts may differ: external and internal noises, wrong choice of measurement parameters, improper sample preparation etc. [25-30]. In order to achieve the right results interpretation on the following solutions may be applied: using of filter, careful data processing, fitting techniques etc [31-34].

Atomic force microscope (AFM) is a modern solution which grants additional advantages to the geometrical data with true values of surface features in Z-coordinate and it is one of the substantial tools for nanotechnology. It allows us to obtain the real information about geometrical sizes of the surface features [35-37]. The surface with nanoscale resolution can be also investigated by using AFM. Moreover, it gives us a possibility to study chemical (surface imperfections) and physical (mechanical, electrical) properties. It also enables us to investigate processes of changing of domain structures, self-assembly, chemical adsorption. In addition, we can modify the surface of the sample and prepare nano-structures.

There are other modern techniques for imaging (SEM, etc.) and they too demand compromises between rate of imaging and data results quality. We define here that during the scanning rate
selection the shape of the surface features should also be considered. It is crucial to take into account the slope of the sample. Furthermore, it is necessary to consider geometrical properties of surface features taking into account all mentioned problems. We can investigate the contribution of sample shape in order to improve the reliability of data with following increase in speed of samples studying. A complete evaluation of surface can't be realized basing on average value and divergence of heights.

Fractal and 3D (three dimensional) statistical parameters of the surface, however, can give us more information than 2D (two dimensional) image of the surface form [38-40].

Materials and Methods

We did all the measurements on former calibration grades. They are impaired by time and treatment during measurements. Nevertheless they represent periodical structures with predefined characteristics of topography. According to producer [41], the characters of the structures are following: TGZ1 – 20.0±1.5 nm TGZ2 - 110±2 nm TGZ3 - 520±3 nm.

The measurements were carried out by AFM Ntegra Prima (NT-MDT, Moscow, Russia). HA-NC probes were used of the same producer. The measurements were performed on scanning areas of 30 x 30 μm². Every measurement was done with similar image size and resolution and at two different scan rates. The experiment was implemented with no changes in microscopy setup and at the same ambient conditions so as to maintain the same conditions of measurements. In order to stabilize laser signal the microscope had been turned on for an hour. Closed loop scanner was used for the scanning. Any filtering was also avoided (Figure 1).

Figure 1. The representative 3-D topographic AFM images, for scanning square area of 30 x 30 μm².
Surface Roughness and 3-D Texture

The purpose of all experiments was to come up with parameters more suitable for describing the artifacts induced by scanning rate. Before computing the surface parameters we didn't make any corrections. The statistical parameters for all three measurements were computed at two measurement rates (Table 1) [42]. The base of computations is the matrix of the image. Registration of interaction between nanosized tip and the surface result in data matrix. Interaction is also influenced by the shape of the surface.

| The basic properties of the height values distribution of the surface samples | TGZ1-30x30-61 um per sec | TGZ1-30x30-314 um per sec | TGZ2-30x30-61 um per sec |
|---|---|---|---|
| Ra (Sa) [nm] | 0.401 | 0.414 | 0.225 |
| Rms (Sq) [nm] | 0.464 | 0.479 | 0.272 |
| Skew (Ssk) [-] | -0.114 | -0.113 | -0.092 |
| Kurtosis (Sku) [-] | -1.16 | -1.18 | -0.618 |
| Inclination θ [°] | 3.1 | 3.0 | 1.8 |
| Inclination φ [°] | 84.4 | 92.9 | 148.3 |

| The basic properties of the height values distribution of the surface samples | TGZ2-30x30-314 um per sec | TGZ3-30x30-61 um per sec | TGZ3-30x30-314 um per sec |
|---|---|---|---|
| Ra (Sa) [nm] | 0.184 | 0.225 | 0.184 |
| Rms (Sq) [nm] | 0.226 | 0.272 | 0.226 |
| Skew (Ssk) [-] | -0.21 | -0.092 | -0.21 |
| Kurtosis (Sku) [-] | -0.464 | -0.618 | -0.464 |
| Inclination θ [°] | 1.4 | 1.8 | 1.4 |
| Inclination φ [°] | 135.2 | 148.3 | 135.2 |

Table 1. The basic properties of the height values distribution (including its variance, skewness and kurtosis) of the surface samples.

| Parameters | TGZ1-30x30-61 um per sec | TGZ1-30x30-314 um per sec | TGZ2-30x30-61 um per sec |
|---|---|---|---|
| D | 2.06 | 2.02 | 2.10 |
| R² | 0.991 | 0.991 | 0.993 |

| Parameters | TGZ2-30x30-314 um per sec | TGZ3-30x30-61 um per sec | TGZ3-30x30-314 um per sec |
|---|---|---|---|
| D | 2.10 | 2.10 | 2.10 |
| R² | 0.992 | 0.993 | 0.992 |

Resolution of image could add the error component and is a significant factor for computation of statistical parameters. There’s no dependence between fractal parameters and data resolution. So, for extraction more values about the surface and its distortion both approaches could be applied.

Computation of the surface statistical parameters and fractal analysis aids to characterize the damage of geometrical data of nanoscale texture. Due to existence of a large number of components contributing to the image distortion by using scanning rate (such as drift) the investigation becomes rather practical (because of prediction difficulties). The time for tool calibration and further measurements can be greatly reduced by using the given approach.

Conclusion

In this paper the dependence of AFM-data reliability on scanning rate was studied. Also, we carried out researches on three-dimensional (3-D) surface topography of the samples with micro-motifs of different high. The surface metrics analysis for estimation of artifacts that occur due to inappropriate scanning rate is given in this article as well.

Cube counting method was utilized to perform fractal analysis and AFM-data were used for implementing the evaluation of statistical metrics. Thus, the distortion of the images against scanning...
rate could be characterized with this approach which subsequently can be also applied for
dependences of the other parameters of measurement.

The article expounds the relevance and comparison of fractal and statistical surface parameters for
characterization of data distortion caused by inappropriate choice of scanning rate.

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Appendix

Statistical analysis was carried out with the software SPSS 14 for Windows (Chicago, Illinois, USA). One-way analysis of variance was applied for verification of results with Scheffé post-hoc tests. Statistically significant differences are assumed to be 0.05 or less. The statistical parameters were expressed by $Ra$ (average roughness), $Rq$ (root-mean-square deviation), $Ssk$ (skew), $Sku$ (kurtosis), angles ($\theta$, $\phi$) (inclination).

In detail, these parameters have following meaning [42]:

- **$RMS$ value of the height irregularities**: this quantity is computed from data variance.
- **$Ra$ value of the height irregularities**: this quantity is similar to $RMS$ value with the only difference in exponent (power) within the data variance sum. As for the $RMS$ this exponent is $q = 2$, the $Ra$ value is computed with exponent $q = 1$ and absolute values of the data (zero mean).
- Height distribution skewness: computed from 3rd central moment of data values.
- Height distribution kurtosis: computed from 4th central moment of data values.
- Mean inclination of facets in area: computed by averaging normalized facet direction vectors.
- Variation, which is calculated as the integral of the absolute value of the local gradient.