LODZ UNIVERSITY OF TECHNOLOGY

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Abstract of PhD Dissertation

Algorithms for characterization of tip-sample force in atomic force microscope

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Since its introduction in 1986, atomic force microscope has become an essential tool for the examination of surfaces in the nanoscale. The AFM relies on the interaction between the sample and a sharp probe attached to a flexible cantilever placed over the sample surface. The most widely used mode of operation of the AFM is the tapping mode. In this mode, the lateral interaction forces between the tip and the sample are minimized. In the tapping mode, the cantilever is excited to vibrate at its resonant frequency or close to it. Then it is brought close to the studied sample surface so that the tip makes intermittent contacts with the surface once in every oscillation period. The contact with the surface alters the amplitude and phase of the oscillations. The vibrations are detected with an optical system where a laser beam reflects from the back of the cantilever and then falls onto a position-sensitive photodiode. As the cantilever is scanned across the surface, the amplitude of oscillation is maintained at a set-point value through a feedback loop. Therefore, the feedback signal reflects the topography of the sample surface. The tip-sample interaction is in fact a non-linear one. This results in the appearance of higher harmonics of the fundamental oscillation of the cantilever.

The phase shift between excitation and response of the vibrating cantilever depends on the energy dissipation during the tip–sample contact. The amount of power dissipated in a sample is related to the mechanical properties of the sample such as viscosity and elasticity. According to theoretical considerations, for a given sample elastic properties one can determine approximately the sample damping constant by measuring the average power dissipation. A theoretical analysis showed that information on the elastic properties of the sample surface is contained in higher harmonics of the fundamental oscillation signal in tapping-mode AFM. The higher harmonics offer the potential for imaging and sensing material properties at the nanoscale. It was pointed out that resonantly enhanced higher harmonics are sensitive to the stiffness of the material under investigation.

The measurement techniques used commonly in commercial AFM systems allow to control only the amplitude and phase of the fundamental mode of the cantilever. The information on the higher-order Fourier components, related to the nonlinear interaction between the tip and the sample, is lost. The main focus of this work was on a new measurement techniques capable of recovering full information about cantilever oscillations. As a result, information on the non-linear tip-sample interaction, and, in particular, an insight into the mechanical properties of the sample could be obtained.

The theses of this dissertation are formulated as follows:

1. The proposed sliding discrete Fourier transform (SDFT) algorithm requires less than half of the multiplication operations than other known algorithms with comparable accuracy.

2. The analysis of the higher harmonics of the fundamental oscillation of the atomic force microscope (AFM) cantilever allows detecting changes in mechanical properties of heterogeneous samples at the nanoscale.

One useful method of detecting simultaneously the amplitude and phase of the cantilever deflection signal is based on the Fourier method. The major difficulty to implement this method as the amplitude and phase detector for the tapping mode AFM is related to the phenomenon of spectral leakage, which occurs when the sampling frequency is not exactly an integer multiple of the investigated signal frequency. Spectral leakage distorts the measurement in such a way that energy from a given frequency component is spread over adjacent frequency bins. In the case of signal amplitude, the effect of spectral leakage can be reduced by the standard concept of windowing. However, windowing does not allow recovering the correct signal phase. To eliminate negative effects of spectral leakage, a new, patented method was proposed. This method, called synchronous detection, guarantees that sampling frequency is always an integer multiply of the excitation frequency.

The computational complexity of the conventional discrete Fourier transform (DFT) algorithm is $O(N^2)$. To reduce the computational time, most frequently the fast Fourier transform (FFT) algorithm is used, for which the computational complexity is $O[N \log_2(N)]$. When only knowledge of the part of the frequency spectrum is desired, the recursive DFT algorithm called the sliding DFT (SDFT) can be used. This algorithm performs an N point DFT on time samples within a sliding window. The SDFT initially computes the DFT of the N time samples. Then, the time window is advanced one sample and a new N point DFT is calculated. The largest advantage of this process is that each new DFT is efficiently computed directly from the results of the previous DFT. This work presents a new algorithm for calculating the sliding DFT. The algorithm is based on the digital waveguide resonator. The proposed algorithm is guaranteed to be stable because it always has z-domain poles located on the unit circle. In contrast to the standard algorithm, the waveguide resonator does not suffer exponential amplitude drift due to round-off errors caused by the quantization. Coefficient quantization can therefore only cause quantization in the frequency of the oscillation.

Furthermore, the algorithm was also designed to be numerically stable for full frequency range. In the simplest form, the proposed algorithm requires only two multiplications per new sample. In comparison to other SDFT algorithms, it means that it is necessary to do only half of the multiplications to obtain a new value of DFT.

Known SDFT algorithms were compared with developed algorithms to evaluate their numerical stability. The results of the numerical simulations confirmed that the only algorithm with comparable accuracy is the modulated SDFT. However, for real valued input signal, modulated SDFT requires ten real multiplications, while the proposed algorithm requires only three.

The developed SDFT algorithm is a part of the synchronous detection method capable of measuring the full frequency spectrum of cantilever oscillations. To test the usability of the applied method of synchronous detection to simultaneously detect higher harmonics of the fundamental oscillation signal, a polymer blend sample was used (PS-LDPE). Mechanically heterogeneous regions of the sample surface were revealed in all the images recorded using the amplitudes of higher harmonics. The results show that the total higher harmonics content (i.e. the sum of the average amplitudes of the higher harmonics) for the PS region is almost three times larger than that for the LDPE region. Material contrast was also present in all the images recorded using the phases of higher harmonics. For most of these images, the average phase differences between the regions with different elastic properties were larger than that of the traditionally used phase image of the first harmonic. The obtained images of the investigated surface prove that the imaging at higher harmonics can be used to distinguish between different materials, even when a conventional cantilever is used.

The spatial resolution, sensitivity and precision of the detection system of the AFM was also demonstrated on a hexagonal crystal 6H-SiC(0001) sample from NT-MDT. The sample possesses uniformly distributed half-monolayer high (0.75 nm) steps on the sample surface demonstrating chemical and mechanical stability. The images obtained with AFM equipped with the proposed detector clearly reveal the presence of more or less uniformly distributed steps on the sample surface. The peaks in the obtained height distribution correspond to particular steps on the sample surface. Taking into account the heights of particular steps, the average step height was determined to be (0.75 ±0.03) nm. This result is in agreement with the data given by the sample producer.

When scans are performed with a T-shaped cantilevers, the method of synchronous detection could be also used to control the peak force. The peak force is defined as the maximum force acting between the cantilever tip and the surface. The insufficient signal-to-noise ratio of the standard (rectangular) cantilever enables access only to time-averaged values of the tip-sample interaction forces. Torsional deflections move the laser spot horizontally on the photodetector. The tip of a typical cantilever is located on the longitudinal axis, preventing tip–sample forces from creating torque on the cantilever when tapping on a sample. On the other hand, T-shaped cantilever known also as a torsional harmonic cantilever (THC) has a tip that is offset from the long axis of the cantilever. When it is vibrated in the tapping mode, tip–sample interaction forces generate a torque around the long axis of the cantilever and excite the torsional modes. As in the case of the conventional tapping-mode AFM, flexural vibrations are used as the amplitude signal to follow the sample topography. Simultaneously, torsional vibrations are used for the calculation of the time-resolved tip–sample interaction forces. The analysis of which could provide information about the mechanical properties of the sample. Current methods enable the visualization of stiffness, adhesion, peak force, and energy dissipation on the tip-sample separation and energy dissipation on the deformation of the surface. However, the algorithms used to recover this information from the cantilever deflection signal are computationally intensive. So far, there was no known method to measure peak force fast enough to use it as input signal for the feedback loop without significantly increasing the time of the acquisition.

The proposed algorithm uses the advantages of the synchronous detection method to reduce the number and complexity of steps needed to recover the value of peak force. Sensitivity of the proposed detection technique permits to reduce the number of data points required to reconstruct the force spectroscopy curve. Thanks to adjustable sampling frequency, it is also possible to obtain interesting components of the frequency spectrum without the calculation of the full Fourier transform. This task is performed with the help of modified digital waveguide resonators. Curve-fitting procedure used to estimate and to substract the crosstalk signal was also replaced by the spectrum analysis. In consequence, the information about the peak force is available within a few oscillation cycles. This means that the value of the maximum force the tip has on the surface during every oscillation cycle could replace amplitude of the first harmonic as the *Z*-axis feedback signal without slowing down the scanning process. Therefore, the risk of damage to the surface of the soft samples could be reduced.

In comparison with other detection methods, good signal-to-noise ratio and high detection speed are specific and significant advantages of the applied method of synchronous detection. As a consequence, this method was found to be very useful and well suited to applications in which oscillation modes are used.