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Scanning probe microscopy as a metrology method in micro- and nanostructure investigations

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Abstract. Evolution of many high technologies such as microelectronics, microsystem technology and nanotechnology involves design, application and testing of technical structures, whose size is being decreased continuously. Scanning probe microscopes (SPM) are therefore increasingly used as diagnostic and measurement instruments. Consequently the demand for standardized calibration routines for this kind of equipment rises. Up to now, there has been no in generally accepted guideline on how to perform SPM calibration procedure. In this article we discuss calibration scheme and focus on several critical aspects of SPM characterization e.g. the determination of the static and dynamic physical properties of the cantilever, the influence factors which need to be considered when plotting a scheme for the calibration of the force and displacement sensitivity.

Key words: scanning probe microscopy, micro- and nanostructure investigations.

1. Introduction

Scanning probe microscopy is a group of techniques that provide measurement of surface topography and surface properties on the subnanometer scale. Since its invention in 1982, the number of investigations devoted to technical advances and applications of SPMs has risen rapidly [1]. Contributing factors include the availability of high-quality commercial and laboratory made instrumentation, the wide range of conditions under which high-resolution and high sensitive measurements can be performed (which includes experiments in vacuum, liquid, and air from 4 to over 700 K), and the possibility to observe mechanical, electrical, thermal and optical surface properties. The basic idea of scanning probe microscopy is to observe so called nearfield interactions occurring between a sharp microtip and investigated surface. When the distance between the sample and the microprobe with curvature radius at the end of a few nanometers and with a very steep sidewall angle is in the range of fractions of nanometers tunneling current, force interactions, thermal flux and electromagnetic wave can be monitored and directly used for the surface characterization. It should also be noted that in contrast to commonly used instruments like scanning electron microscopies (SEMs) and optical microscopies SPMs can image surface parameters in all three dimensions: X, Y and Z. In Table 1 we compare the characteristics of these common technologies for imaging and measuring surfaces. It shows that optical microscopes and SPMs are the quickest and easiest to use, with little or no sample preparation and no vacuum required. Optical microscopes and SEMs have larger fields of view but SPMs provide the highest magnifications and resolution. SEMs and SPMs image only the surface and provide larger depth of field, but only SPMs work on nearly all samples with minimal sample preparation.

Table 1
Comparison of various microscopy based methods for micro- and nanostructure investigations

	Optical microscope	Scanning electron microscope	Scanning probe microscope
Operating environment	Ambient, liquid, vacuum	Vacuum	Ambient, liquid, vacuum
Depth of focus	Small	Large	Medium
Resolution XY	1 μm	10 nm	0.1 nm
Three dimensional imaging	–	–	+ Resolution 0.01 nm
Sample preparation	Little	Drying, coating	Little
Characteristics of the investigated sample	Must not be completely transparent to light wave	Conductive surface	Must not exhibit excessive variations in surface height

It should be additionally emphasized that the scanning probe microscopy is one of the newest methods of the high resolution surface metrology. In contrast to optical microscopes and SEMs, SPMS do measure surfaces in all three dimensions: X, Y, and Z. However to achieve quantitative information about the surface of the micro- or nanostructure several factors con-

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cerning the determination of the tip shape, cantilever mechanical properties and microscope scanning system have to be considered. In this case the development of new calibration schemes, which includes fabrication of test samples, design of precise optical and electronical equipment and application of image processing software routines, is needed. In the scanning probe microscopy, nanostructure and nanometrology laboratory of the Microsystem Electronics and Photonics Faculty at the Wroclaw University of Technology we developed several measurement methods and techniques which enable quantitative analysis of the surface properties based on the nearfield observations [2–5]. We will present the procedure for calibration of piezoelectrical scanners utilized in scanning probe microscope system, determination of mechanical parameters of spring beam cantilever and tip shape characterization.

2. Metrological properties of the scanning probe microscopy system

2.1. Piezoelectrical scanner. The heart of the scanning probe microscope microscope is the piezoelectrical scanner. In this case the two basic designs can be distinguished:

— scanning sample systems, where the specimen is moved in X, Y, Z directions and the scanning tip is kept in constant position. In this case the design of the detector head (which is responsible for the tip deflection observation) is simplified but the measurements of big sized samples (e.g. microelectronical integrated circuits or hard disks) are quite difficult. Simultaneously the adjustment of the microscope feedback loop, which maintains the distance between the sample and the microtip, must be performed after every sample change, scanning tip velocity modification etc.

— scanning tip systems, where the tip scans over the investigated surface. In these systems a very smart design of the detector head is needed, so that the setup of the detector head remains mechanically stiff and light. Simultaneously the adjustment of the feedback loop, due to the defined scanner mass can be done more precisely.

To accomplish required subnanometer resolution of the tip or sample scanning movements a piezoelectric tube or piezostack is used. They can be controlled by the precise high voltage electronics to enable measurements of the up to 100 square micrometers sized scannfields. However, it should be noted that the piezoelectric actuators exhibit high nonlinearity and hysteresis while scanning. In Fig. 1 we present the results of the measurement of the static piezotube displacement observed with the optical fiber Fabry-Perot interferometer.

In our experiments we used an interferometric sensor was introduced by Rugar et al in 1989 [6] for detection of cantilever motions in atomic force microscopy. In our design, which includes flat and highly reflective mirror mounted on the piezoactuator parallel to the cleaved fiber end, we have observed that ca.30% of the outgoing light is reflected back into the fiber (Fig. 2). The signal photodiode FD1 detects the interference of the light reflected from the fiber end and from the mirror, which is mounted on the piezoactuator. By recording the interferometric fringes and by interpolation the signal it is possible to calculate the mirror displacement. The wavelength of the semiconductor laser was determined in our laboratory with the accuracy of 10 nm and the resolution of the applied numerical algorithm is 0.5 nm. Using the described system we observed the nonlinearity of the actuator movements of 20 nm, which describes the difference between the true position and the position calculated as a function of applied electrical voltage.

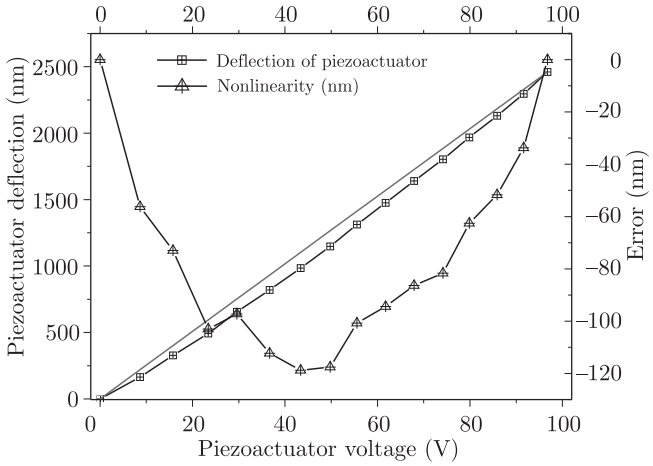

Fig. 1. Piezoelectric actuator deflection measured with the optical fiber interferometer

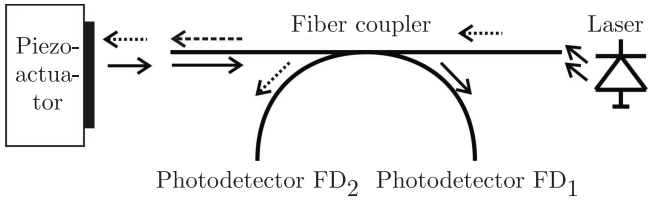

Fig. 2. Schematic diagram of a fiber optical interferometer displacement sensor

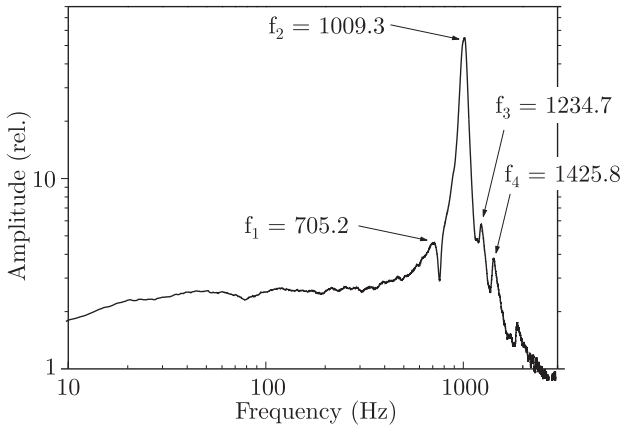

Fig. 3. Piezoelectric actuator deflection as a function of control voltage frequency

Fig. 4. Scanning Probe Microscopy scanning head with integrated 3 interferometers for measurements of piezoscanner deflection in XYZ directions

Additionally, while surface scanning not only the scanner nonlinearity should be taken into consideration but also the dynamic behavior of the loaded actuator. In Fig. 3 we present the piezotube deflection observed when the frequency of the control voltage applied to the piezoceramics was varying. It can be clearly seen that the piezotube exhibits resonance behavior, which means that at some excitation frequencies the displacement of the piezotube increases rapidly. The phenomena presented above show that in the surface metrology using scanning probe microscopy methods the real piezoactuator deflection must be recorded and used for the sample characterization. In the laboratory of scanning probe microscopy, nanostructure and nanometrology we developed optical fiber based instrumentation, which integrates 3 described above Fabry-Perot fiber interferometers for the measurement of the true piezoactuator XYZ deflection (Fig. 4). In our design we record, while surface scanning, signals coming from 3 interferometers [7,8]. Next based on the observed interferometric fringes we are able to calculate the true position of the XYZ scanner. Using this system it is possible to observe the piezoactuator deflection with the accuracy of 10 nm and the resolution of 0.5 nm. In Fig. 5 we present the results of the measurements of the topography of the chromium-quartz line structure observed with the calibrated atomic force microscope. In this case, based on the interferometric fringes analysis, the width and the height of the chromium line are with the uncertainty of 99% 399 nm+/–27 nm and 189.5 nm+/4.8 nm respectively. Future projects performed in the laboratory of scannning probe microscopy, nanostructure and nanometrology at the Faculty of Microsystem Electronics and Photonics of the Wroclaw University of Technology will be connected with the development and application of the metrology scanning probe stage, which will integrate a system for the measurements of the piezoactuator position in XYZ axis with respect to the additional parasitic scanner movements. In this case we will be able to determine dimensions of the micro- and nanostructures integrated with substrates of relatively high mass and volume.

2.2. Mechanical properties of the SPM cantilever. Second key component in the scanning probe microscopy system is the probe (tip) integrated with the micromachined spring beam. Today's advanced semiconductor technology methods enable tips and cantilevers to be produced in large quantities with very sharp tips of well defined geometry. Soft cantilevers are available with spring constants less than interatomic bond strengths (ca. 1 N/m) to allow topographic imaging of surface atomic structure by sliding the tip across the surface and monitoring cantilever deflection (Contact AFM). These cantilevers can be fabricated with resonant frequencies higher than 10kHz to enable fast measurements of surfaces with high spatial frequency roughness.

To enable topography measurements of very sensitive samples, resonance techniques of the scanning probe microscopy (e.g. so Non-Contact or Tapping Mode AFM) are applied. In these techniques the application of stiffer cantilevers and higher resonance frequency (up to 1MHz) is required. However, in all scanning probe microscopy methods the precise calibration of the spring constant and tip shape is needed. In scanning probe microscopy, nanostructure and nanometrology laboratory at the Faculty of Microsystem Electronics and Photonics we tested cantilevers with integrated piezoresistive deflection sensor [9]. In this case piezoresistive tip deflection sensor is integrated with the spring beam. When the cantilever is bent the mechanical stress occurs and as a consequence the resistance of the piezoresistor is changed. In this way it is possible to determine the tip deflection by measuring the electrical signal on the output of the piezoresistive stress detector. The application of the piezoresistive detection scheme simplifies the mechanical setup of the scanning microscope head and is especially suitable for metrological application. Our opinion is that these sensors enable the measurement (which is interpreted as a quantitative process in contrary to detection which allows only qualitative observation) of force acting at the microtip. To perform calibration procedure of the mechanical sensor parameters we developed experimental method which enables determination of the beam spring constant, force and tip displacement sensitivity with accuracy of 5%. The achieved accuracy, which is higher than in case of optical levers should enable application of the piezoresistive sensors in mechanical surface investigations, where cantilever with higher stiffness are recommended. In our experiment we observed the resonance frequencies of the spring beam, which are a function of the geometrical sensor dimensions. based on the observation of the resonance sensor behavior [9].

2.3. Measurement of the microtip geometry. Another very important metrological issue in the tip-cantilever characterization is the determination of the probe radius, which in most applications is in the range of 30 nm. As shown in Fig. 5 while scanning the line structure the recorded profile is wider than the structure width by the tip diameter. This can lead to relatively high uncertainty of the topography measurements especially in measurements of details with dimensions in the range of tens of nanometers (Fig. 6). Application of high resolution SEM systems for measurement of the microprobe ra-

dius is quite difficult and can not be performed for every tested sensor. Therefore there is a need for development of tip calibration procedures, which can be applied just before starting of the sample scanning. In laboratory of scanning probe microscopy, nanostructure and nanometrology we applied for the characterization of the tip shape fabricated with CMOS technology nanostructures, which contain narrow silicon dioxide lines with defined width adjustable in the range from 30 nm up to 100 nm [6,8]. The calibration nano-lines are fabricated at the edge of previously formed silicon steps as a result of a chemical reaction (oxidation) (Fig. 7a) which takes place at the sidewalls of the prepared structure. The line-width is precisely controlled by the progress of the chemical reaction proceeding in lateral direction (Fig. 7b). Removing of the original step by selective plasma etching results in a free standing "fence" consisting of the material produced by the chemical transformation (Fig. 7c). When the SiO_2 nanoline structure is scanned over the shape of the applied microtip can be determined on the basis of the recorded profile. If the width of the structure is defined the tip radius can be calculated "in situ" with high accuracy using scanning probe microscopy system, which will be next used in further experiments. In Fig. 8a we show the 3D view of the topography of the SiO_2 nanolines observed with the standard Contact AFM cantilever, which was used in previous experiments performed in the laboratory. The zoomed image of the single nanoline presented in Fig. 8b shows that the microtip integrated at the cantilever end is flat and that the tip radius of the applied microprobe is 123 nm (Fig. 8c) [10]. Additionally at the tip sidewalls additional step can be observed, which can influence the measurement of the structure geometry. The geometry of the tip which was applied in the performed experiments is presented in Fig. 8d and it is clear that the tip profile corresponds with the shape determined in our experiments. The performed test experiments lead to the conclusion that the investigated sensor is useless in measurements of rough structure and can only be applied in diagnostics of smooth structures deposited on atomic flat substrates.

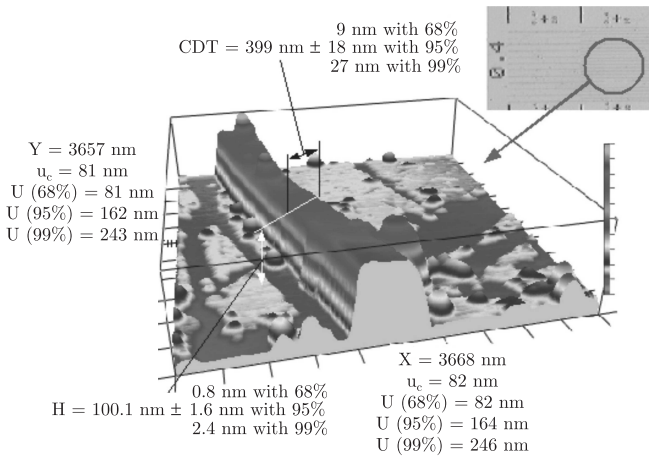

Fig. 5. Topography of the chromium-quartz line structure observed with the calibrated atomic force microscope

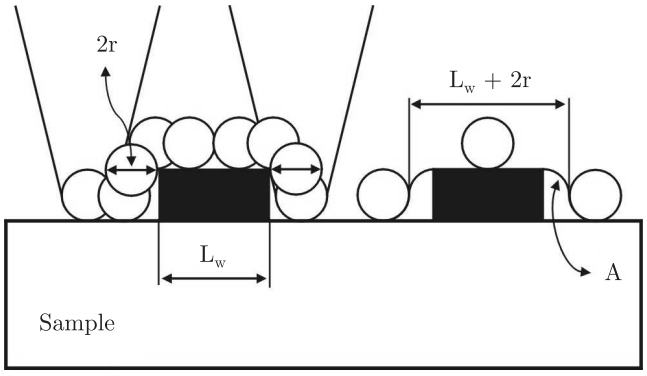

Fig. 6. Measurement of linewidth with the scanning probe microscope

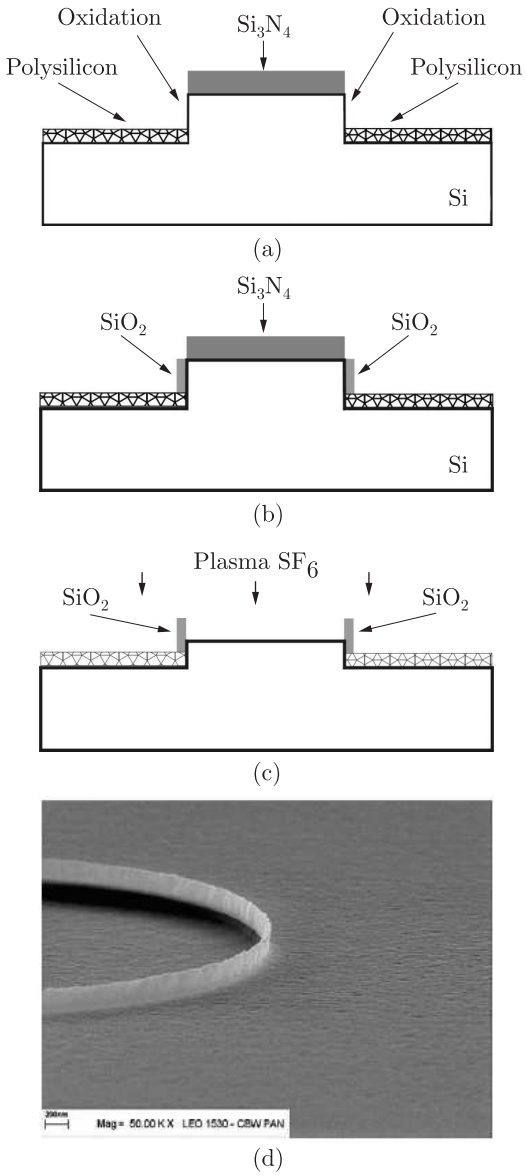

Fig. 7. Fabrication process of the calibration structure for the SPM tip characterization: oxidation of the sidewalls of the Si steps (a), oxidized Si steps before plasma etching of the Si_3N_4 layer (b), plasma etching of the Si_3N_4 layer and Si step (c), SEM image of the final device (d)

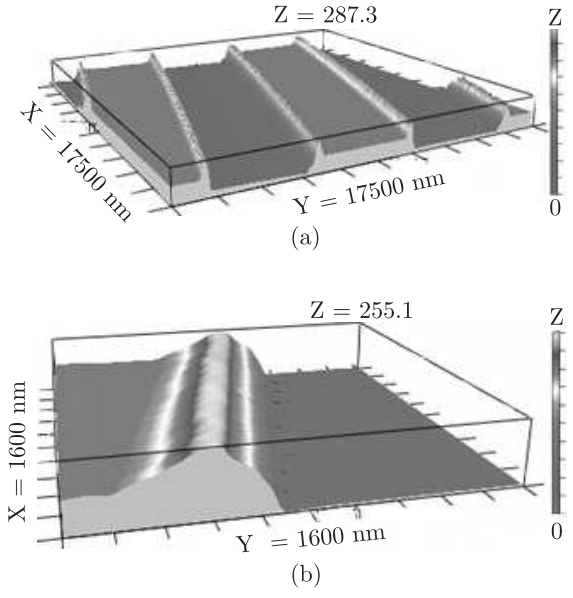

Fig. 8. Characterization of tip shape using SiO_2 nanolines: 3D view of the topography structure a), zoom image of the single nanoline b), crossection of the tip profile c), SEM image of the tested microprobe d)

3. Conclusion

In this paper we presented experimental methods which enable utilization of the scanning probe microscopy in the analysis of micro- and nanostructure geometry. We described the application of the fiber interferometry in the measurements of microscope piezoactuator deflections in XYZ directions. In our setup we achieved the resolution of 0.5 nm and the accuracy of 10 nm of the piezoactuator position measurement. Using this system which was integrated with the scanning probe microscope we measured the topography of the chromium line deposited on the quartz substrate. We determined the width and the height of the structure with the uncertainty of 99%. We also presented the method for the tip shape calibration which was based on the application of microfabricated line structure. In this case we were able to measure *in situ* the tip radius of the microprobe and to asses the quality of the microprobe.

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