Invited paper

Diagnostics of micro- and nanostructure using the scanning probe microscopy

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Abstract—In this paper we summarize the results of our research concerning the diagnostics of micro- and nanostructure with scanning probe microscopy (SPM). We describe the experiments performed with one of the scanning probe microscopy techniques enabling also insulating surfaces to be investigated, i.e., atomic force microscopy (AFM). We present the results of topography measurements using both contact and non-contact AFM modes, investigations of the friction forces that appear between the microtip and the surface, and experiments connected with the thermal behaviour of integrated circuits, carried out with the local resolution of 20 nm.

Keywords*— *scannig probe microscopy, microsystem, nanofabrication.

1. Introduction

The development of the scanning probe microscopy (SPM) investigation methods that began with the invention of the scanning tunnelling microscope in 1982 [1] made it possible to observe the structure of conductive and insulating surfaces in the air, in a nanometer scale for the first time. The so-called nearfield interactions that occur between the microtip and the surface at a distance of several nanometers (e.g., tunnelling current or heat flux flowing between the microprobe and the sample, force interaction between atoms of the tip and the surface) are observed in these measurement techniques and utilized for versatile structure characterization.

The SPM instruments have been used successfully at the universities and research institutes with for the last few years to carry out research in the nanotechnology area. Simultaneously the SPM methods were introduced in the semiconductor industry to measure nanometer-sized structures. This trend was driven, of course, by the fact that no other experimental method could provide as much information (e.g., surface roughness, elasticity, semiconductor dopant profiling, line width) on the fabricated devices.

The proper analysis of the acquired images requires however the knowledge of physical phenomena that are observed between the microtip and the sample.

2. Scanning probe microscopy experiments

For closely spaced atoms of the microtip and the surface the interaction energy is described by the Lennard-Jones potential [2]. The resulting force is illustrated in Fig. 1. There are two regions corresponding to the basic atomic force microscopy (AFM) measurement modes. In a contact AFM (C AFM) instrument the microtip touches the investigated surface. In this case the force between the microtip and the sample, which ranges typically from 1 nN up to 100 nN, is repulsive and causes the static cantilever deflection. The C AFM methods are mostly applied for the measurements of mechanical surface parameters. This technique has been developed to probe the viscoelastic and anelastic properties of submicron phases of inhomogenous materials. The measurement yields the information related to the internal friction and to the variations of the dynamic modulus of nanometer-sized volumes.

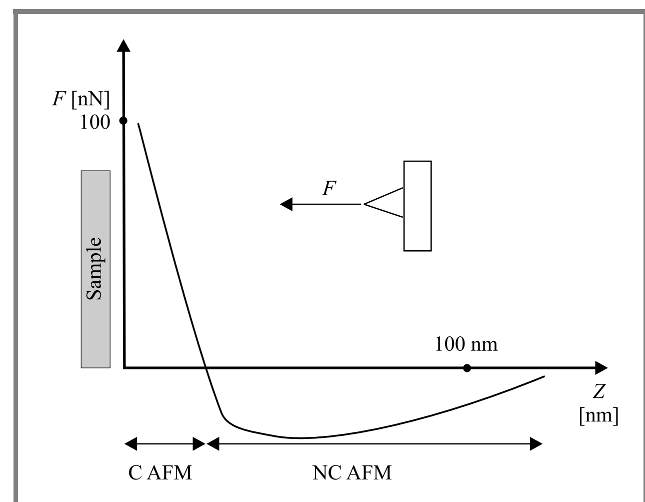

Fig. 1. Atomic force microscopy forces between the microtip and the sample.

The other C AFM method is the so-called lateral force microscopy (LFM), in which not only the vertical beam deflection but also the beam torsion are monitored. It should be noted that in classical tribology, all the investigations are carried out under heavily-loaded conditions. Therefore bulk

properties of the investigated sample dominate its tribological characteristics. In contrast to the macrotribological systems, all micro- and nanosystems operate under very light loads (few μN). In this case friction and wear are influenced only by the interactions of a few surface atom layers, therefore nanometer resolution of the investigations is required. Consequently, C AFM methods with LFM techniques are the appropriate tools for surface characterization.

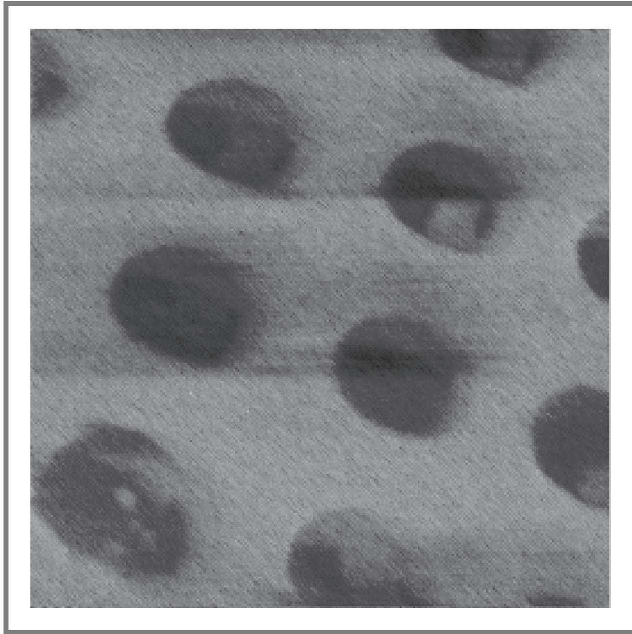

Fig. 2. Topography of a quartz/chromium point structure (scanfield 7×7 μm, structure height 120 nm, scanfield 2 lines/s).

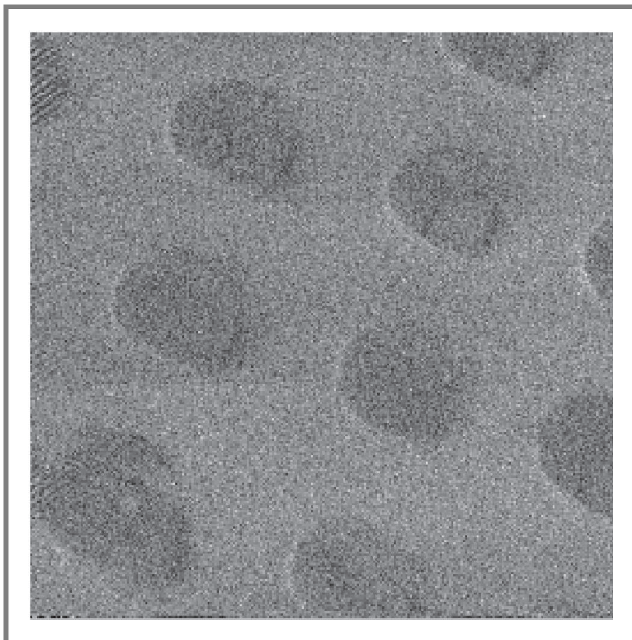

Fig. 3. Friction forces observed on a quartz/chromium point structure (load force 1 μN, maximum friction force 100 nN).

The LFM methods using the piezoresistive cantilevers were applied in the investigation of a gridlike structure of chromium dots on a quartz substrate [3]. The dots were about 120 nm high (Fig. 1). In order to perform simultaneous AFM and LFM measurements the signals corresponding to the topography and friction forces acting on the microtip were recorded. The lateral force image (Fig. 2) shows that friction forces vary on the sample surface. Higher friction was detected on the chromium dots, as shown by brighter locations in the LFM image (Fig. 3). The C AFM methods are also utilized for precise, high resolution, quantitative topography investigations. The quantitative measurement of the structure line width (the so-called critical dimension (CD) measurements) requires however the integration of the C AFM machine with the detection system of the cantilever or sample scanning movements. There are several methods to observe the deflection of the microscope piezoelectrical actuators that move the sample or cantilever in the range from 100 nm up to 100 μm with the resolution of 20 nm and the accuracy of 10 nm. In our experiments we applied the optical fiber interferometry to measure the scanning movements of the sample. Based on the observed interferometric fringes we can determine the piezoactuator deflections with the resolution of 25 nm. In Fig. 4 we show the topography of a structure consisting of chromium lines deposited on a glass substrate. The determined line width estimated based on the interferometric measurements is 1030 nm.

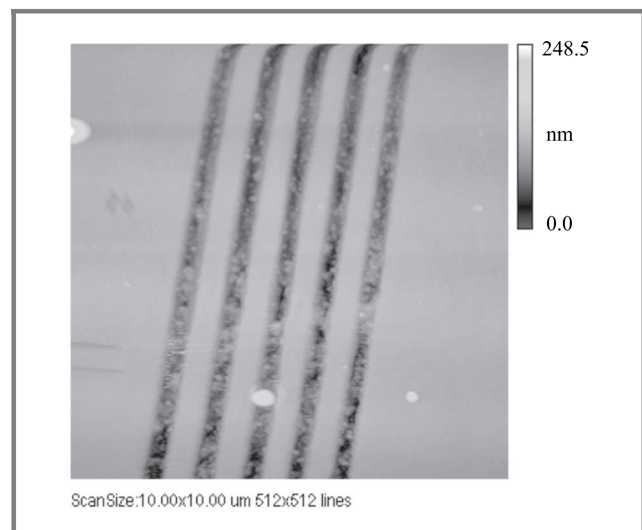

Fig. 4. Topography measurements using calibrated C AFM-quartz etched with reactive ion etching (linewidth 1 μm).

Scanning thermal microscopy (SThM), based on C AFM, is widely applied in the investigations of thermal behaviour of micro- and nanostructures. In this method a microtip containing a heat flux sensor measures locally the temperature of the sample.

The SThM system developed at the Wrocław University of Technology enables temperature changes of 20 mK to be measured with spatial resolution of 20 nm. In Fig. 5 the temperature distribution on the surface of integrated ciruit (IC) consisting of four resistors is presented. In our experiments the Wheatstone bridge formed by these resis-

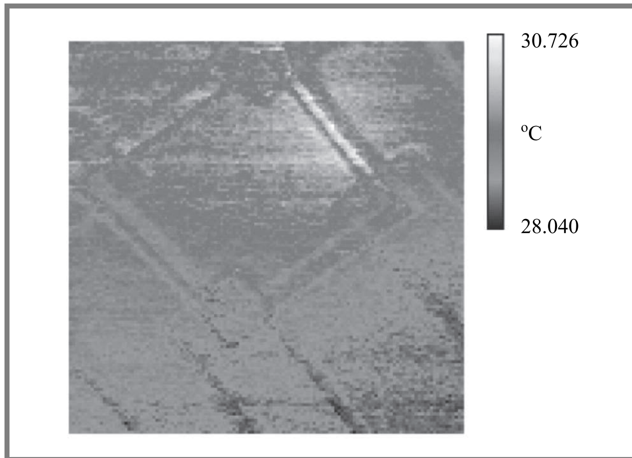

Fig. 5. Scanning thermal microscopy measurement of the temperature distribution of an integrated resistive bridge loaded asymetrically.

tors was supplied unsymetrically. The temperature in the region where most of the energy was dissipated is higher by 2 K than in the area of the other three resistors.

The experiments performed show the possibility of applying SThM methods in reliability investigations of micro- and nanostructures [5].

In the second region of the force-distance curves (Fig. 1), where the spacing between the tip and the surface is of 10–100 nm, very weak attractive interactions in the range of 0.1–0.05 nN are observed. Since the level of the interactions appearing in non-contact AFM (NC AFM) systems the force detection method is more elaborate. In this case the microscope cantilever vibrates at its mechanical resonance. The change of the beam resonance frequency or cantilever oscillation amplitude under the influence of the attractive force acting on the microtip is monitored and applied to control the probe-sample distance while sample scanning. The NC AFM techniques can be applied in the investigations of surface topography of soft materials, like, e.g., photoresists and biological samples. Because of high sensitivity of the force interaction measurements (enabling forces in the range of 0.01 nN to be detected), in NC AFM methods not only the topography investigations are performed but also electrostatic forces between the microtip and the surface can be monitored and applied for structure characterization. In this way the electrostatic force microscopy (EFM) has the potential to be a very promising analysis method for measurements of the properties of nanometer-size semiconductor devices and materials, such as dopant profiles, high-k insulator thickness. All applications indicated in literature show that EFM and SCM would be effective as a measurement tool for semiconductor devices that are continuously miniaturized. The measurement possibilities of the EFM based methods depend, however, strongly on the parameters of the applied probe. In our experiments we applied piezoresistive cantilevers [4] with conductive probes that enable voltage of 40 mV to be measured in the bandwidth of 30 Hz, and the silicon cantilevers with metallic tips for

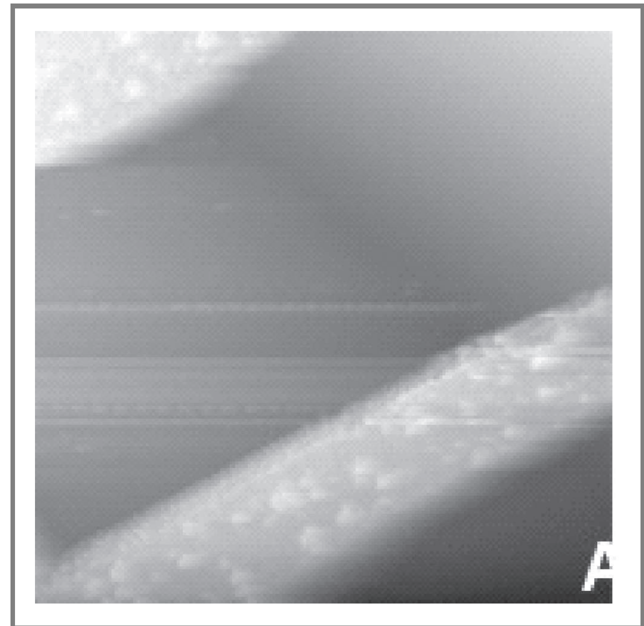

Fig. 6. Topography of an Al-SiO_2 sample observed with the EFM microscope (scanfield 45×45 μm, the structure height 300 nm).

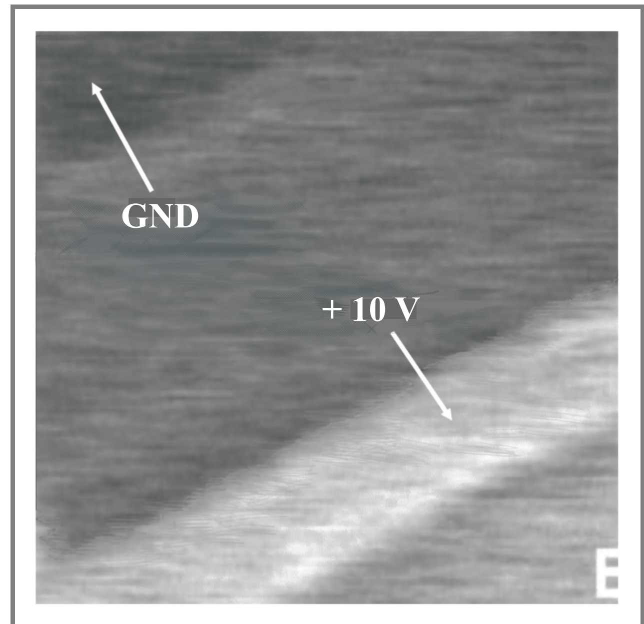

Fig. 7. Electrostatic force contrast observed on the Al-SiO_2 sample.

the measurements of voltages with the resolution of 30 mV in the bandwidth of 30 Hz [6]. In our investigations we applied a voltage $U = U_{dc} + U_{ac}\sin(\omega t)$ between the tip and the sample, which gives rise to an electrostatic force given by:

$$F = \frac{1}{2}\frac{dC}{dz}\left[(U_{dc}^2 + \frac{1}{2}U_{ac}^2) + 2U_{dc}U_{ac}\sin(\omega t) + \frac{1}{2}U_{ac}^2(1 - \sin(2\omega t)\right] = \frac{1}{2}\frac{dC}{dz}(F_{dc} + F_{\omega} + F_{2\omega}), \quad (1)$$

where C is tip-sample capacitance, ω is the frequency.

Using lock-in techniques we were able to distinguish forces corresponding to the potential at the structure surface and interactions corresponding to the variable capacitance between the microprobe and the sample. The topography of the sample formed by two Al lines deposited on the SiO_2 substrate measured with EFM microscope is presented in Fig. 6. The scan size is 45×45 μm, the structure height is of 300 nm. In our experiments we applied the voltage of 10 V to the metal strips. The image of electrostatic forces measured simultaneously with the topography is shown in Fig. 7. The dark region of the image corresponds to lower electrostatical force and grounded Al line, the brighter shade shows where the voltage of 10 V applied to the metal line was recorded.

3. Conclusions

In this paper we have presented the results of the experiments connected with the application of SPM methods in versatile diagnostics of micro- and nanostructure. The presented experiments shown that with the increasing innovations in SPM technology, the SPM instruments will be applied not only in university research techniques but also in industrial quality control and application measurements.

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References

[1] G. Binnig, H. Rohrer, and E. Weibel, "Surface studies by scanning tunneling microscopy", *Phys. Rev. Lett.*, vol. 49, p. 726, 1982.

[2] G. Binnig, C. F. Quate, and Ch. Gerber, "Atomic force microscopy", *Phys. Rev. Lett.*, vol. 56, p. 930, 1986.

[3] T. Gotszalk, P. Grabiec, and I. W. Rangelow, "Piezoresistive sensors for scanning probe microscopy", *Ultramicroscopy*, vol. 82, p. 39, 2000.

[4] T. Gotszalk, F. Shi, P. Grabiec, P. Dumania, P. Hudek, and I. W. Rangelow, "Fabrication of the multipurpose piezoresistive Wheatstone bridge cantilever with conductive microtip for electrostatic force and scanning capacitance microscopy", *J. Vac. Sci. Technol.*, vol. B16, no. 6, p. 3948, 1998.

[5] R. F. Szeloch, T. Gotszalk, and P. Janus, "Scanning thermal microscopy in microsystem reliability analysis", *Microelectron. Reliab.*, vol. 42, p. 1719, 2002.

[6] T. Gotszalk, P. Czarnecki, P. Grabiec, K. Domański, M. Zaborowski, and I. W. Rangelow, "Microfabricated cantilever with metallic tip for electrostatic and capacitance microscopy and its application to investigation of semiconductor devices", *J. Vac. Sci. Technol.*, vol. B1622, p. 506, 2004.

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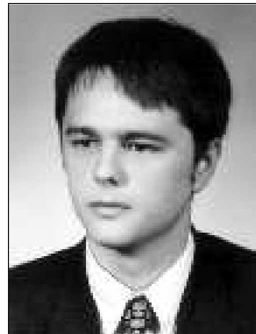

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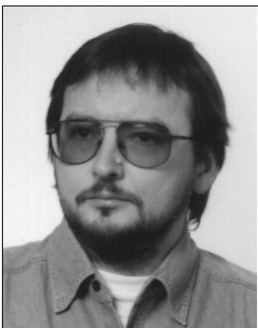

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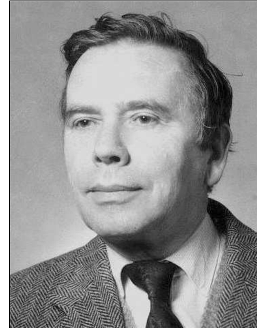

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