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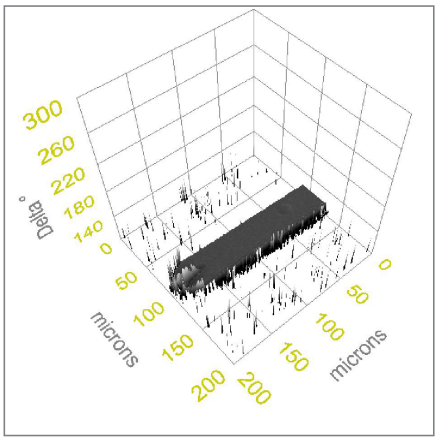
APPLICATION NOTE
Functional Coatings on Cantilevers

ABSTRACT

Bambu et. al (2004) developed a technique to cover microelectromechanical systems (MEMS), such as micromechanical cantilever (MC) sensors, with a covalently bound brush layer. The polymer layer was grown using a "grafting-from" synthesis of polymer brushes under mild conditions, by surface-initiated atom transfer radical polymerization. Imaging spectroscopic ellipsometry enable the characterization of coatings on the microscopic surfaces of micromechanical cantilever. From an angle of incident spectra the thickness and the optical properties of the gold layer were obtained. Four zone delta and psi-spectra in the UV/VIS range between 360 and 900 nm were recorded to determine the thickness of the cantilever and the thiol- or additional the polymere layer. The homogeneity of the polymere coating was controlled with thickness maps of a lateral resolution of 1 µm.



Delta map of a coated cantilever

Introduction

Micromechanical systems (MEMS), such as micromechanical cantilever (MC) sensors for use with atomic force microscopes can be functionalized by layers of polymer[1]. Alternatively the functional layer can be bound with thiol on a gold layer on the cantilever. MC sensors can be modified to detect surface stress changes, to measure trace gas, to detect mass loading and temperature changes. For MEMS thin and uniform coatings are desired to achieve reproducible results. The investigation of uniformity can be done with atomic force microscopy or with imaging ellipsometry. In the following the imaging ellipsometer is used, which gives thickness maps of the functional layer with higher lateral resolution, and with higher accuracy than ordinary ellipsometer.

Instrumentation

Spectroscopic Imaging Ellipsometer EP3SE, automatic sample handling stage, 10 x and 20 x objectives.

Task

Local measurement of the thickness of the thiol-layer and recording of a thickness map.

Measurements

In the ellipsometric contrast image (fig. 2) the cantilever appears inhomogeneous. This observation could not be done with an ordinary ellipsometer with microspot-option. The ellipsometric observables Delta/Psi are measured averaged over the region of interest (white box in fig. 2), which is set on the 60 µm wide cantilever. The angle of incidence spectrum (fig. 3) has been recorded on the gold-coated side without thiol. The thickness, refractive index and extinction of the gold film are then obtained from fitting the spectra of Delta/Psi. In another step Delta constant while the thickness of thiol is the only fit

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parameter in the simulation of the angle of incidence spectrum. It is assumed that the Si-substrate of the cantilever is nontransparent, which is true below 532 nm. Above this wavelength the extinction (absorption) of the Si is so small that the approximately 1 µm thin Si substrate is transparent. As a consequence the Si-substrate must be taken as a layer in the simulation.

The wavelength spectrum (fig. 4, recorded on a sample similar fig. 1) is fitted assuming a thin Si-layer. There are oscillations of Delta/Psi above 532 nm due to the transparence of the Si-substrate. The period of these oscillations is given by the thickness of /Psi are recorded on a thiol-coated piece of the cantilever. In order to obtain the thickness of the thiol layer, the optical parameters of the gold are held the Si-layer.

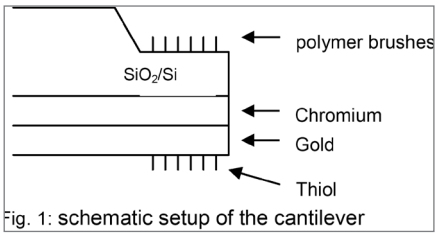

Fig 1: Schematic setup of the cantilever.

Sample
Si-wafer (substrate) with SiO_2-layer with variable thickness.

Steps of Evaluation
- Positioning of the cantilever in the contrast image (fig. 2)
- Define region of interest (ROI) where to measure (fig. 2)
- Record Delta/Psi spectrum as a function of the angle of incidence (fig. 3)
- Record Delta/Psi spectrum as a function of the wavelength (fig. 4)
- Fit the spectra in order to obtain the optical properties of the layer stack
- Record a Delta-map Calculate a map of the layer thickness while keeping constant all other optical properties of the layer stack (fig. 5 and 6)

Fig. 2: Ellipsometric contrast image of the cantilever at 532 nm wavelength, white box (region of interest, ROI) defines the area, over which an averaged ellipsometric Delta/Psi is measured, the surface of the cantilever appears inhomogeneous.

Therefore the thickness of the Si-cantilever is obtained from itting the spectrum above 532 nm. The thickness of the gold and of the thiol layers is also obtained from the fitting. The thickness of the polymer layer is similarly measured by eans of an angle of incidence spectrum or wavelength spectrum. Maps of Delta have been recorded at 400 nm and 532 nm wavelength. Their parameter Delta has been converted into the thickness of the polymer layer based on an optical model with a non-transparent Si-substrate. The resulting thickness map (fig. 5) at 532 nm wavelength contains stripes, which are absent at 400 nm wavelength. We conclude that the stripes are artifacts due to the wrong assumption of a non-transparent substrate at 532 nm. The stripes are caused by Interference in the Si-layer. The inhomogeneous areas of the cantilever are representing an inhomogeneous thickness of the Sicantilever. In fact we see in fig. 6 that the thickness of the polymer-layer is rather homogenous.

Results

The thickness of the following parameters has been measured: Si-substrate, gold-layer, thiol-layer, polymer-layer. The refractive index and the extinction of the gold-layer have also been measured. Thickness maps of the polymer-layer with one µm lateral resolution have been obtained which show that the polyer-layer is homogeneous, and that there is a variation in the thickness of the Si-substrate.

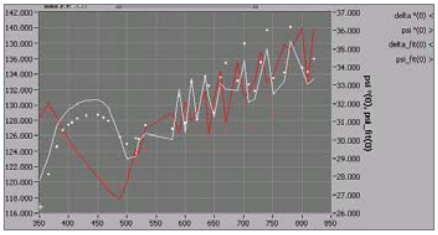

Fig. 4: Delta/Psi spectrum (four zone mean), sample similar fig. 1, resulting fit-parameters are the thicknesses d(Si) = 1475 ± 3 nm, d(au) = 14.7 ± 0.2 nm, d(thiol) = 2.0 ± 0.2 nm (where n(thiol) = 1.47 is assumed).

Conclusion

Due to its unique lateral resolution Nanofilm's imaging ellipsometer EP3 is perfectly suitable to inspect thickness of functional layers on cantilevers. To this end the EP3 measures

ellipsometric observables Delta/Psi as spectra, which are averaged over an arbitrary region of interest, or it maps the Delta and one corresponding layer thickness in the whole field of view.

Acknowledgement

We would like to thank Rüdiger Berger from the Max-Planck Institute for Polymer Research, Mainz, Germany, for supplying the sample.

References

[1] Synthesis and Characterization of Polymer Brushes on Micromechanical Cantilevers, G.G. Bumbu, G. Kircher, M. Wolkenhauer, R. Berger, J.S. Gutmann, Macromol. Chem. Phys., vol. 205, pp. 1713 (2004)

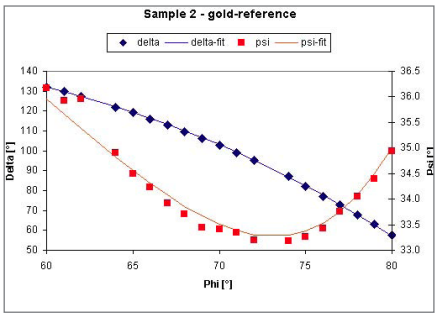

Fig. 3: Fit on the spectrum of Delta/Psi at 532 nm wavelength with the resulting fit-parameters of the gold-layer: thickness d = 23.5 nm, refractive index n = 0.548, extinction k = 2.37.

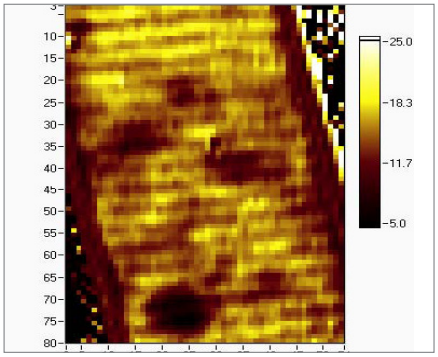

Fig. 5: Thickness map of the polymer-layer on the cantilever at 532 nm wavelength with 20x objective, calculated under the assumption of a nontransparent Si-substrate, grayscale in nm with arbitrary offset, lateral scale µm.

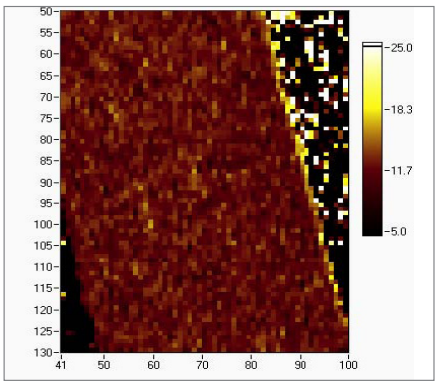

Fig. 6: Thickness map of the polymer-layer on the cantilever at 400 nm wavelength with 20x objective, calculated under the assumption of a non-transparent Si-substrate, lateral scale µm.