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# Scanning characterization of polymer coating layers using contact resonance with piezoresistive microprobes

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**Abstract**
Polymer coating layers are analyzed using microprobes operated in contact resonance (CR) mode. To generate a better understanding of CR, two approaches are described in this work: performing CR measurements on reference artefacts and comparing CR and force-distance curves (FDC). As reference artefacts, polymer films of different thicknesses on silicon are used. The relationship between film thickness and CR data is presented and evaluated. The samples are also analyzed using FDC to investigate the relation between CR and mechanical surface properties.

**Keywords:** contact resonance, force-distance curve, nano imprint resist layer, contact stiffness, Young's modulus

## 1. Introduction

The ongoing digitalization of industrial production generates a demand for high-speed methods to measure form, roughness and mechanical properties of workpieces and equipment in-line [1]. Tactile microprobes have been shown to be able to scan surfaces at velocities up to 15 mm/s and are, thus, promising for facing this challenge [2]. In a collaborative project within the frame of the European Metrology Programme for Innovation and Research (EMPIR) such devices shall be further developed [3]. One of the objectives of this project is to develop validated techniques for measuring the surface properties of workpieces on-the-machine using scanning probe methods based on contact resonance (CR) and force-distance curves (FDC). CR in an atomic force microscope (AFM) setting is an established method and its mechanisms were described in detail [4-6].

In this work, piezoresistive microprobes of much larger dimensions are considered, for which the theoretical aspects of CR are not yet fully understood, and an adapted physical model has to be developed. First, the measurement setup for CR with piezoresistive microprobes and its basic operation principle are described. Then, the method of acquiring and analyzing CR data is explained. Finally, results obtained on different thin-film nano imprint resist layers on silicon are presented and discussed in comparison to FDC measurements on the same samples.

## 2. Measurement setup and theory

The piezoresistive microprobes used in this work are silicon cantilevers, on which a full Wheatstone bridge is integrated for readout near the cantilever base as well as a silicon probing tip near the free end. The geometrical parameters of these probes are listed in Table 1. They are attached to a chip-size piezoactuator (PL055.30 by Physik Instrumente (PI) GmbH und Co. KG) on a carrier printed circuit board (PCB) using glue (Loctite 401 by Henkel AG & Co. KGaA). The carrier PCB shown in Figure 1, which is designed to provide a preamplifier, a piezoactuator and signal ports, is then mounted onto a homemade positioning and scanning table consisting of a stack of manual and piezo positioning stages. In Figure 2, the positioning and scanning table with a microprobe carrier PCB is shown close to a sample under test, which is positioned in close proximity to the microprobe using a manual $xyz$ stage.

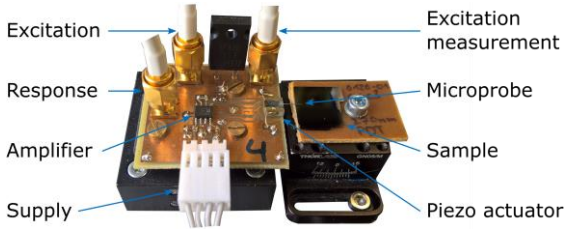


**Figure 1.** Overview of a microprobe carrier PCB next to a sample under test.

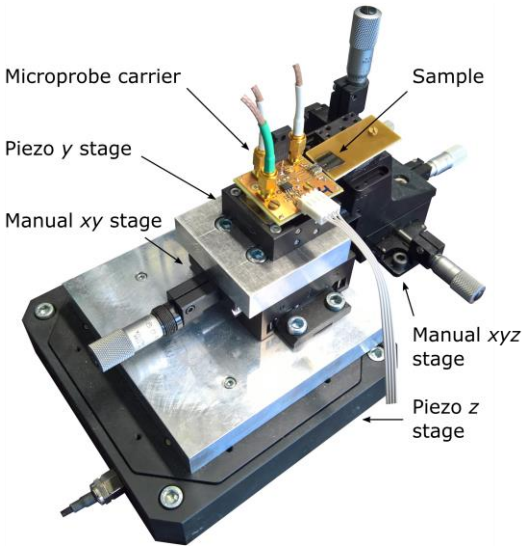


**Figure 2.** Photograph of homemade positioning and scanning table with microprobe carrier PCB. The cantilever tip is contact with a sample on a manual $xyz$ stage.

During measurement, first, the $z$ piezo stage is used to move the probing tip against the sample surface until contact is attained and the cantilever starts to bend. This movement is continued until the force applied by the cantilever to the surface according to the product of cantilever deflection and stiffness reaches a pre-defined value. Once this force is reached, the probe is excited into an out-of-plane bending-vibration mode by actuating the base of the cantilever via the chip piezoactuator underneath. As depicted in the schematic in Figure 3, the tip of the probe interacts with the surface of the sample, thereby shifting resonance frequency and amplitude of the cantilever to higher and lower values, respectively.

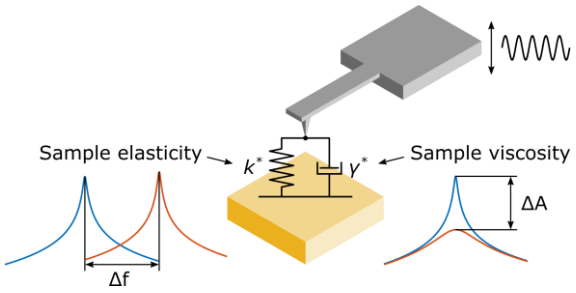


**Figure 3.** Schematic of the tip-surface interaction related to elasticity and viscosity of the sample during out-of-plane bending vibration of the cantilever leading to an upward shift of its resonance frequency as well as increased damping with respect to the free oscillation (inspired by [5]).

In [4, pp. 55-58], Rabe presented a physical model describing the dynamic behavior of AFM cantilever probes under the condition of tip-to-surface contact at defined coupling strength. This model is modified slightly for the present work. As the tilt $\varphi$ of our microprobe of ca. 1° is small compared to up to 20° [7] in the case of AFM cantilevers, its effect can be neglected here. Without this tilt, we can expect that the lateral spring-damper-element does not affect the vibration of the cantilever and can be neglected as well. Instead, the effect of air-damping is taken into account, which reduces the quality factor of resonance modes dramatically [8] and, thus, has to be considered to accurately determine values of contact damping from the measured data. Additionally, the boundary conditions corresponding to vibration excitation at the base of the probe are assumed according to the selected probe design with the piezoactuator assembled underneath the cantilever base. This configuration is as also known as ultrasonic atomic force microscopy (UAFM) operation mode in AFMs. The result is a physical model describing analytically the out-of-plane vibrations of a microprobe cantilever with typical dimensions given in Table 1 operated via its tip in contact with a viscoelastic material.

**Table 1** Measured geometry of the microprobe.

| Parameter | Symbol | Value |
|---|---|---|
| Length | $L$ | 4.73 mm |
| Length, anchor-to-tip | $L_1$ | 4.66 mm |
| Width | $w$ | 196 µm |
| Thickness | $b$ | 50.4 µm |

### *2.1. Methodology*

Before the actual measurements are conducted, reference measurements are carried out to determine refined values of air-damping, microprobe geometry, piezoelectric actuation coefficient and piezoresistive sensing coefficient for the modelling. The dimensional parameters can differ from the values measured by optical and scanning electron microscopy, as idealizations of the physical model often lead to certain discrepancies between measured and calculated values [6]. A still remaining slight deviation between modelled and measured resonance frequencies is removed using a rescaling of the frequency axis for the free-vibrating and in-contact conditions. The material and geometrical parameters of the microprobe used for the modelling, which will be described in the following, are listed in Table 2.

**Table 2** Microprobe parameters used for the analysis.

| Parameter | Symbol | Value |
|---|---|---|
| Density | $\rho$ | 2330 kg/m³ |
| Youngs modulus | $E$ | 169 GPa |
| Length | $L$ | 4.77 mm |
| Length anchor-to-tip | $L_1$ | 4.40 mm |
| Width | $w$ | 196 µm |
| Thickness | $b$ | 50.4 µm |
| Frequency-axis rescaling factor | $f_{\mathrm{rescaled}}/f$ | 1.06 |

For our experiments, we select nano imprint resist polymer films (four of mr-NIL210 and three of mr-UVCur06, respectively, by micro resist technology GmbH, Berlin) of different thicknesses deposited on silicon. Layer thickness is measured using a stylus instrument with steps generated by area-selective removal of the layer. On each film CR measurements in the first out-of-plane vibration mode are performed at ten different contact forces. These measurements are then analyzed by fitting our modified physical model of CR to the measured data. As a result, contact stiffness $k^*$ and contact damping constant $\gamma^*$ are determined for the layers. Figure 4 shows the Bode plot of the first out-of-plane bending-mode of the microprobe in contact with a thin film of mr-NIL210 nano imprint resist. Superimposed a fit using the modified physical model is displayed yielding best agreement with the measured values of resonance frequency and amplitude with the parameters in Table 2, a contact stiffness of $k^* = 749\ \mathrm{N/m}$, and a contact damping of $\gamma^* = 55.7\ \mu\mathrm{N}/(\mathrm{m/s})$.

In addition to the viscoelastic properties we determine the deformation (elastic and plastic) of the sample by the tip from the z-axis position and the DC component of the output voltage of the piezoresistive Wheatstone bridge, measured when the tip is in contact with sample. The $z$-axis position of the tip comprises both a cantilever bending deflection and the sample deformation. Thus, the cantilever bending deflection has to subtracted from the z-axis position of the tip to yield the deformation. The cantilever bending deflection can be calculated from the applied force and the cantilever stiffness, which is measured before on a silicon surface considered as stiff enough that no deformation occurs. Unfortunately, for thin layers of 160 nm and below our measurements yield so far unreasonable quantitative results, which we therefore do not show in the following.

Using our CR setup and the described methods we can determine the viscoelastic properties of coatings in dependence on elastic/plastic deformation. For comparison with CR, FDC measurements are performed using a Cypher AFM by Asylum Research combined with PPP-CONTSCAuD (stiffness of 0.2 N/m) and PPP-NCHAuD (stiffness of 42 N/m) probes by Nanosensors, which are used for measurements on the mr-NIL210 and mr-UVCur06 samples, respectively.

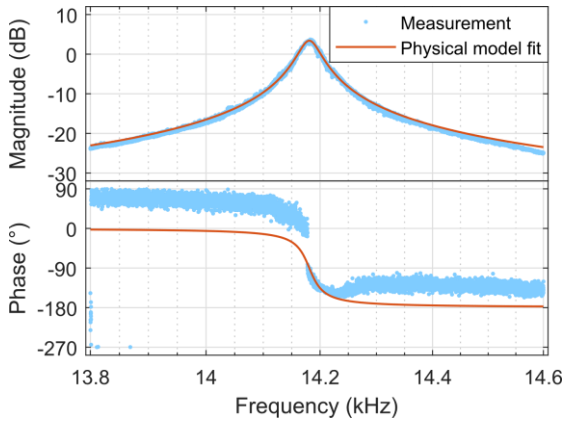


**Figure 4.** Bode plot of the first out-of-plane bending-mode contact resonance peak, measured on a thin film of mr-NIL210 nano imprint resist (blue) and a fit of the physical model generated from the measured values of resonance frequency and amplitude only (red).

### 3. Results

The first objective of this study is to analyze the effect of the thickness of polymer films on the contact resonance frequency. We can expect that the CR frequency decreases with increasing film thickness, as the effect or the substrate with its much larger Young's modulus on contact stiffness $k^*$ is decreasing. In Figure 5, these frequencies are shown in dependence on the elastic/plastic deformation of the sample. As stated above, due to unreasonable quantitative results, these values are not shown on the abscissa. We assume that inaccuracies of the measurement procedure, which are currently being investigated, are responsible for this shortcoming.

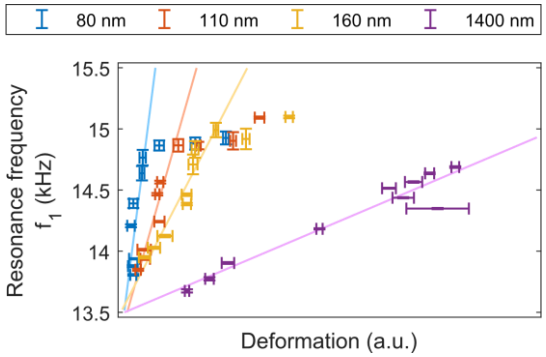


**Figure 5.** Contact resonance frequencies measured on polymer films of different thicknesses in dependence on deformation (error bars), and linear regressions to guide the eye (continuous lines, only points below 14.85 kHz are considered for the regressions).

Nevertheless, a clear discrimination between the CR frequency-deformation dependences can be observed for the different film thicknesses. At low deformations, the CR frequency seems to increase linearly with the deformation. The assumption of a linear relation in this region is supported by the correlation coefficients between frequency and deformation of 0.79, 0.92, 0.95 and 0.97 for the measurements on the 80 nm, 110 nm, 160 nm and 1400 nm thick film respectively. Above a value of about 14.85 kHz, however, the CR frequency saturates. This is likely caused by piercing of the probing tip through the polymer film and the resulting direct interaction with the substrate. Consequently, the observed further increase in deformation has to be interpreted as a measurement error. Therefore, all points measured beyond the saturation value of CR frequency are neglected in the subsequent discussions. If we take the linear regressions shown in Figure 5 as a calibration, we are able to deduce directly the film thickness from a measured pair of CR frequency and deformation, without needing calculations based on the described modified CR model.

The second objective of this study is to validate elastic parameters of the polymer layers measured by CR using the established FDC method. For this purpose, we derive the contact stiffness from CR measurements with mr-NIL210 layers on silicon shown using our modified physical model. The results of these investigations are depicted in Figure 6, where in addition parabolic fits are superimposed to the measured data points. A corresponding dependence of Young's modulus on the deformation was measured with the same samples using FDC as shown in Figure 7. Young's modulus $E$ is calculated from the measured FDCs according to:

$$E = \frac{k_c}{\sqrt{R}} \left( \frac{\partial D^{3/2}}{\partial \delta_c} \right)^{-1}$$

with the cantilever stiffness $k_c$, the tip radius $R$, the deformation $D$, and the cantilever bending $\delta_c$. The initial decrease of $E$ over the first ten nanometers of deformation may be related to a slightly increased stiffness of the layers at their surface.

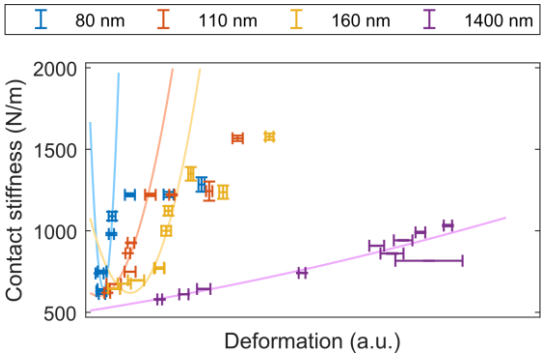


**Figure 6.** Contact stiffnesses calculated from the CR measurements corresponding to Figure 5 (error bars) in dependence on deformation and parabolic fitting curves to guide the eye (continuous lines, only points corresponding to CR frequencies below 14.85 kHz are considered for the regressions).

As quantitative numerical values of the deformation from our CR measurements are not yet available, a quantitative dependence between the contact stiffness from CR and Young's modulus by FDC cannot be determined so far. Nevertheless, the curves of contact stiffness and Young's modulus both show similar, parabolic shapes.

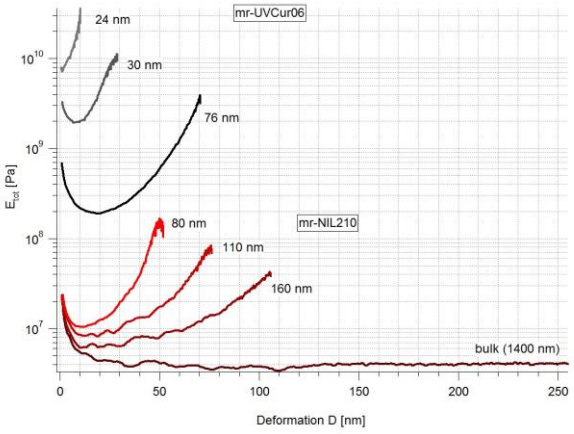


**Figure 7.** Young's moduli of polymer films (mr-UVCur06 and mr-NIL210 by micro resist technology GmbH) calculated from measured FDC data.

## 4. Conclusion

In this study, the effect of thickness of polymer films on silicon substrates on its viscoelastic properties measured by contact resonance (CR) spectroscopy was investigated. A linear dependence of CR frequency on sample deformation was found, showing that non-destructive thin-film-thickness measurement is feasible using CR without needing physical modelling of the tip-sample contact. Values of contact stiffness obtained by CR were validated using force-distance curves (FDC), indicated by observed quadratic dependences of contact stiffness and Young's modulus on deformation. However, a quantitative relation between the mechanical surface properties measured by CR and FDC has not been obtained yet.

Enhancements to the setup to combat measurement errors are being performed. Additionally, measurements on harder polymer films (mr-UVCur06 by micro resist technology GmbH) to investigate CR and FDC further are being carried out.

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