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Characterizing the material mechanical properties inside small holes

Min Xu, Uwe Brand, Ludger Koenders

Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100, D-38116 Braunschweig, Germany

Min.Xu@PTB.de

Abstract

To analyze the performance of high-aspect-ratio microstructures (HARMS), it is not only necessary to measure the inner form and surface topography, but also to characterize the inner material mechanical properties quantitatively and nondestructively. The Profilscanner with the slender piezoresistive microprobe enables performing traceable roughness measurements inside small holes with diameters down to 50 µm. Contact resonance technology makes it possible to quantitatively map the material mechanical properties on a nanoscale. In this paper, we apply the contact resonance technology to the Profilscanner and the slender piezoresistive cantilever microprobe to investigate the feasibility of quantitatively determining the material mechanical properties inside HARMS.

Keywords:
Contact resonance, high aspect ratio, material mechanical property

1. Introduction

In the past few decades, several novel technologies have been developed for the form and surface roughness measurement of high-aspect-ratio microstructures (HARMS) to overcome the difficulties caused by the extremely narrow probing spaces. Among the above technologies, the Profilscanner with the slender piezoresistive microprobe enables traceable roughness measurements inside small holes with diameters down to 50 µm [1, 2].

In some forms of application, the materials inside the HARMS are changed, such as in the case of the fouling due to fuel deposits inside injector nozzles. As a result, the form and surface roughness measurements alone are not enough for the HARMS performance analysis. It is also necessary to characterize the material mechanical properties quantitatively and nondestructively.

The mechanical properties measured from small length scale specimens can vary from those of bulk specimens. They are strongly influenced by the surrounding materials and by the boundary conditions in general. Contact resonance technology [3, 4] permits the possibility of quantitatively measuring the mechanical properties on a nanoscale through analyzing the resonant frequencies of the cantilever with analytical models. In this paper, we apply the contact resonance technology to the Profilscanner and the slender piezoresistive microprobe, and explore the feasibility of characterizing the mechanical properties inside small holes.

2. Contact resonance

In the contact resonance method, the flexural resonant frequencies of a cantilever beam, which is clamped at one end and free at the other end, are measured. If the tip at the free end of the cantilever is in contact with a sample surface, the cantilever end conditions are changed by the tip-sample interactions. Consequently the cantilever resonant frequencies are shifted. The shape and width of the resonant curves are also changed. Young's modulus of the sample surface can be determined through measuring these frequency shifts.

In the contact resonance technology, it is most important to model the flexural vibrations of the cantilever and the tip-sample interactions appropriately. The point mass model is often used to calculate the first free flexural resonant frequency ω_0 and contact resonant frequency ω of the cantilever:

$$\omega_0 = \sqrt{\frac{k_C}{m^*}} \tag{1}$$

$$\omega = \omega_0 \sqrt{1 + \frac{k^*}{k_C}} \tag{2}$$

where k_C and m^* are the static spring constant and effective mass of the cantilever, and k^* is the normal contact stiffness.

The tip-sample interaction is often modelled as that of a spherical tip with radius R contacting a flat sample surface with a normal force F_n according to the Hertzian model for elastic deformation. The normal contact stiffness k^* is given by:

$$k^* = \sqrt[3]{6E^{*2}RF_n} \tag{3}$$

where E^* is the reduced Young's modulus of contact which is given by:

$$\frac{1}{E^*} = \frac{1}{M_s} + \frac{1}{M_t} \tag{4}$$

Here, M_s *and* M_t are the indentation modulus of the sample and the tip, respectively.

Contact resonance technology has been adopted in atomic probe microscopy (AFM) to quantitatively map the material elastic modulus, and the traceability of the method can be ensured by calibrated reference standards. However, the AFM cantilever is only several hundred µm long and not sufficient for HARMS measurements. Therefore, we investigate the application of the contact resonance technique to the slender piezoresistive cantilever.

3. Experimental results

The experiment is performed with the Profilscanner and the experimental setup (Fig. 1) works in so-called ultrasonic atomic force microscope (UAFM) mode [5]. It means that the cantilever vibrates instead of the sample. The cantilever is fixed on a Z piezo stage (PI, model: P-622.ZCD) and the base of the cantilever is excited by a piezo actuator. A lock-in amplifier (SRS, model: SR830) excites the piezo actuator and analyses the spectrum of the cantilever vibrations.

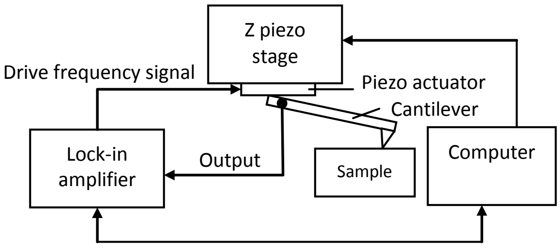
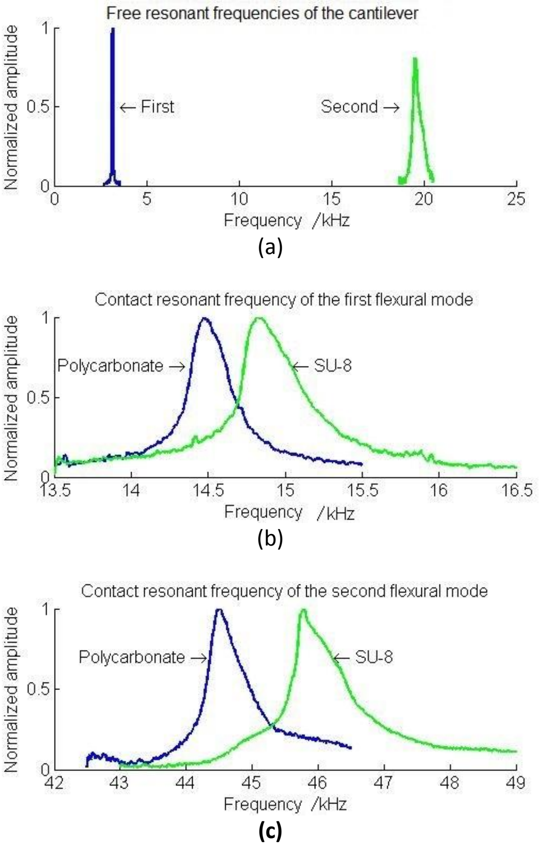


Fig. 1 The contact resonance experimental setup

The cantilever used in the experiments is a rectangular piezoresistive silicon cantilever with nominal dimensions of 5 mm length, 200 µm width and 50 µm thickness. The static spring constant of the cantilever is calibrated to be k_C = 12.8 N/m ± 5 % using a meander reference spring [6]. The measured object is a stripe-like specimen made of SU-8 on a silicon wafer. Because it was difficult to get precise knowledge of the tip radius demanded in eq. (3), reference measurements were used in the experiment and a polycarbonate standard sample worked as the reference sample. The Young's modulus of the polycarbonate standard sample is 2.7 GPa and the reduced modulus with a diamond probe is 3.10 GPa. The indentation modulus of the silicon cantilever tip is assumed to be 165 GPa.

The spectra of free vibration and contact resonance of the cantilever are shown in Fig. 2. The resonant frequencies of the first two flexural modes are 3.1 kHz and 19.6 kHz, respectively (Fig. 2(a)). The static load of the cantilever on the sample was 132 µN. On the polycarbonate and SU-8 samples, the contact resonant frequencies of the first two flexural modes shifted to 14.5 kHz, 14.8 kHz (Fig. 2(b)) and 44.5 kHz, 45.8 kHz (Fig. 2(c)), respectively. Applying the measured frequencies to the eqs. (1)–(4), the indentation modulus of the photoresist SU-8 obtained is 3.41 GPa. This result is obviously smaller (-25 %) than the measured value of 4.50 GPa using the surface deformation method and the literature value (i.e. $\frac{E}{1-v_{SU-8}^2} = 4.25 \pm 0.73$ GPa, where v_{SU-8} = 0.22) [7].

The experimental results indicate that the utilization of the contact resonance technology on the piezoresistive cantilever is possible. To increase the precision of the method, the modelling and the theoretical analysis have to be improved.



(a)

(b)

(c)

Fig. 2 The free vibration and contact resonant spectra measured with a piezoresistive cantilever

4. Conclusion

The application of the contact resonance technology on the slender piezoresistive cantilever is introduced and the experimental results prove the feasibility, but more work is required to improve the modelling for more precise measurements of Young's modulus.

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