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Mapping of elastic modulus at sub-micrometer scale with acoustic contact resonance AFM

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ABSTRACT

Atomic Force Acoustic Microscopy (AFAM) is a powerful near field technique combining the high spatial resolution of Atomic Force Microscope (AFM) with ultrasonic stresses to access mechanical properties of material shallow surfaces (essentially local stiffness magnitudes like Young modulus). In this article, we discuss different experimental set-up and modelling approaches to determine quantitatively the Young modulus of thin films. Static experiments carried-out on dense and nanoporous silica have shown a good agreement with nanoindentation experiments. Stiffness mapping have also been performed on macroporous silica and copper interconnect structures, showing the ability of our set-up to sense different mechanical answers at sub-micrometer scale.

1. Introduction

With the wide development of nanostructured materials, new needs in local material characterization have risen. The knowledge of local material properties allows a better understanding of material behaviour under external stresses. For instance, multi-material structures (that can display sharp changes in stiffness and hardness constants) have to be assessed at their periodicity-scale. In order to fulfil this requirement of local analysis, characterization techniques have been developed around Atomic Force Microscopy (AFM) because of its great spatial resolution [1]. Standard AFM is well known for allowing accurate topography of micro-scale surfaces with a nano-scale resolution. In order to sense other magnitudes than surface topography, AFM-tip and AFM-cantilever are commonly functionalised to become sensitive to various physical properties (magnetic, electrical,...). For instance it can be set for the characterization of mechanical properties like elastic modulus [2].

For this latter concern, the basic approach lies on the ultrasonic resonant vibrations of the AFM-cantilever [3]. The fact that the AFM-tip is (or not) in-contact with a sample drastically disturbs the cantilever vibration spectrum [4]. The shift of cantilever resonance frequencies between free and in-contact spectra allows the extraction of physical data like sample's stiffness, tip-to-sample contact surface area. This technique is commonly reported as Atomic Force Acoustic Microscopy (AFAM) [5–7].

To extract quantitative information from these experiments, a modelling of the vibrating system is required [8]. All the developed models are based on Hertz contact theory [9] (assimilating the shape of the tip end to a rounded shape and disregarding adhesive forces).

In the present article we report results of AFAM experiments fitted with simple models of vibrating beams with different boundary conditions. Part of the experiments were carried-out under static conditions (no AFM scanning), while others were performed under dynamic conditions, allowing a stiffness mapping of material surfaces) [10].

2. Set-up, modelling and characterization procedure

2.1. Experimental set-up and actuation methods

Our experiments were performed on an AFM Nanoscope III Dimension 3100 from Veeco Instruments. Under AFAM setting, the cantilever was set into sine-wave oscillation at ultrasonic frequency by an actuation source. The sine-wave amplitude was fixed while the frequency was swept typically from 10 kHz to 2 MHz. The cantilever vibration amplitude was detected through a LASER beam reflected by the cantilever back. This signal was then analysed by a lock-in amplifier (Signal Recovery, model 7280).

Two different ultrasonic actuation set-ups have been assessed (Fig. 1). The first one uses the piezoelectric transducer built-in the AFM-cantilever holder (Fig. 1a). The second one uses the electrostatic interaction between a metallic electrode (located underneath the under-analysis sample) and the AFM-cantilever (both behaving like a capacitor) (Fig. 1b).

For the latter the electrostatic interaction has been assessed by following the cantilever vibration amplitude versus the electrode-to-cantilever distance (Fig. 2). One can see the linear evolution of

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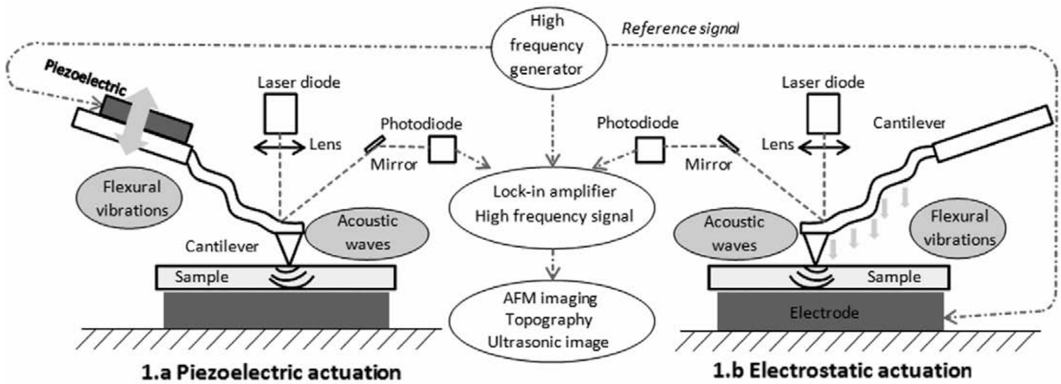

Fig. 1. Schematic of experimental set-up for AFAM.

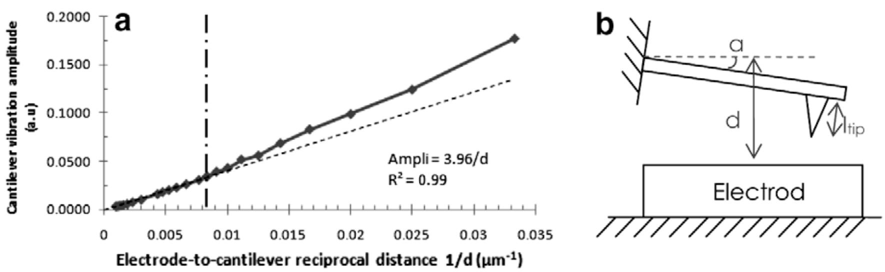

Fig. 2. Electrostatic force influence on the free cantilever vibration amplitude (a and b).

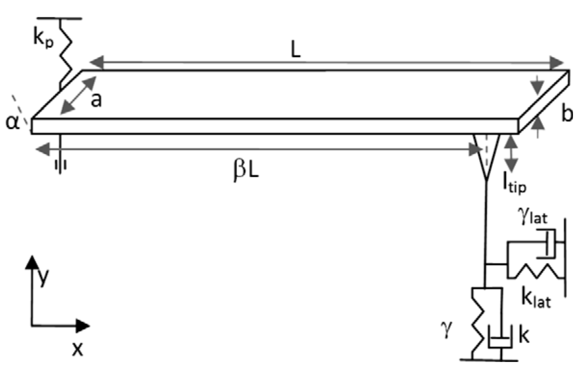

Fig. 3. Schematic of an in-contact cantilever.

the amplitude as the reciprocal of the distance ($1/d$) as long as the tip is not felt by the electrode ($d_{critical} \sim 10 \times l_{tip}$). Electrostatic actuation is one of the least explored ways of actuation [11], even though it is one of the most promising set-ups.

2.2. Modelling of the vibrating system

Extraction of the physical parameters impacting the vibration lies on the use of a sharp system modelling. An analytical modelling of the complete cantilever beam with different boundary conditions has been reported by Dupas [12]. The matching of the experimental vibration spectrum with the simulated one is obtained by the fitting of physical parameters like cantilever dimensions, cantilever physical properties, sample stiffness, tip-to-surface contact, etc.

This modelling first considers the AFM-cantilever as a large clamped beam and then applies an improved point-mass model (Fig. 3). The beam parameters are: length L, width a, thickness b, specific mass ρ and modulus E. The pyramidal tip is of length l_{tip} and mass m_{tip} and attached at point βL along the beam. It is suspended to his origin by a spring of stiffness k_p associated to a guiding sleeve describing the exciting transducer (piezoelectric actuation configuration (Fig. 1a)).

This system can be solved analytically via the resolution of two subsystems, one goes from the origin of the beam to the tip, and one from the free end of the beam to the tip, each one obeying to the equation of movement of a beam (Eq. (1)).

$$EI\frac{\partial^4 y}{\partial x^4} + \rho A\frac{\partial^2 y}{\partial t^2} = 0 \quad (1)$$

with y the cantilever local deflection, E the cantilever Young modulus, I the areal moment of inertia, ρ the specific mass and A the beam section [8].

2.3. Analysis procedure

The study of a given sample requires the following procedure:

1 First, a free cantilever spectrum is recorded. The only parameters impacting the vibration are the cantilever properties (L, a, b, ρ, E). Thus a set of these parameters is found to fit the experimental spectrum. Input values of these parameters are extracted from preliminary SEM observations on a LEO 440 microscope.

2 At this stage the probe is moved toward the sample surface (a constant monitored force F is applied) and an in-contact spectrum is recorded. The cantilever parameters being set from the first step, the remaining parameters to be fitted are now only related to the tip-to-surface contact: contact stiffness (k_n and k_{lat}) and tip RoC (R). Both magnitudes are related to the material Young modulus E_{sample} through Eqs. (2) and (3) [2].