Atomic Force Acoustic Microscopy for Surface Mechanical Characterization

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Abstract

Atomic Force Acoustic Microscopy is a measurement technique used for nanomechanical characterization of different materials. The present work discusses applicability of the technique for fast mapping and quantitative estimation of local surfaces elastic properties, with high lateral resolution even in presence of thin films. Investigations and comparison with nanoindentation technique are eventually reported on different samples produced by means of Plasma Enhanced Chemical Vapour Deposition technique (PECVD).

1 Introduction

Miniaturization in many fields of science and technology introduces new serious challenges in materials characterization, calling for the need for fast non-destructive mechanical testing in the submicrometre scale. New techniques are then needed for providing precise characterization of materials and surfaces properties at the micro and nanoscale. An interesting solution is given by scanning probe microscopy (SPM). The dynamic behaviour and response are in fact influenced not only by probe dimensions and stiffness, but also by the mechanical properties of the surface with which it interacts. This principle is used by Atomic force acoustic microscopy (AFAM), a relatively new dynamic AFM-based technique for fast nanomechanical non-destructive measurements, exploiting the spatial resolution typical of SPMs [1,2].

2 Modeling

In AFAM, the sample under investigation is vibrated at ultrasonic frequency, while the cantilever is contacting and scanning the sample surface through the tip. Any standard AFM with lateral actuation of the probe (the so called "scan by probe" set up) can be operated for AFAM measurements, simply implementing a longitudinal ultrasonic piezoelectric transducer under the sample. The sample is eventually fixed to the transducer: commonly liquid honey guarantees tight contact, with good acoustic conductivity. A schematic representation of AFAM interaction is given in Figure 1. During contact interaction between the tip and the sample surface, the cantilever free end turns to a coupled end. As a consequence, flexural resonance frequency shifts occur, proportional to the coupled end contact stiffness k^*. When the probe moves from a stiffer to a softer region and contact stiffness diminishes, a proportional variation in resonance frequency takes place. Also, being dampening larger in the case of a softer region, the relative peak at the resonance frequency has smaller amplitude. Simultaneously with acoustic imaging (which can be properly converted to quantitative mapping of mechanical properties) AFAM technique provides also reconstruction of surface topography. In this way different mechanical properties can be directly associated to different surface topography structures.

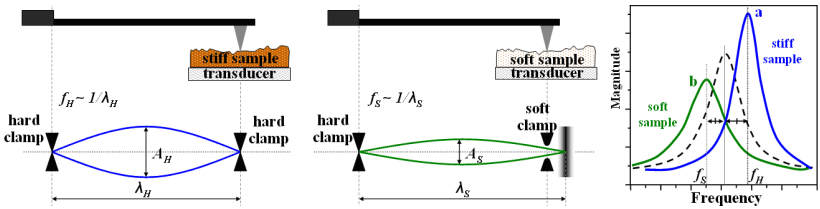

Figure 1: AFAM interaction when the probe is in approach respectively with a stiff (left) and a soft (center) surface, generating different flexural vibration wavelengths and frequencies (right).

3 Investigations

Probe dynamic behaviour, and thus atomic force acoustic microscopy, is determined not only by contact stiffness but also by cantilever geometry and mechanical properties (i.e. width, height, length, relative angle, density and modulus of elasticity) and tip to surface interaction (i.e. tip geometry, force and feedback). All these

parameters, if not properly estimated, may reduce the accuracy of quantitative nanomechanical measurements. Additionally, non constant phenomena as tip wear or interaction force, further increase uncertainty, asking for continuous monitoring of tip properties and interaction. In order to reduce the number of controlled parameters, and therefore the number of measurements, a task specific calibration can be implemented.

In the present study, applicative investigations were carried out on a set of multilayer (ML) silica coatings on a (100)-oriented silicon wafer substrate. Coatings had a thickness of 2 μm and were produced with different ML designs and different mechanical properties, by Plasma Enhanced Chemical Vapour Deposition (PECVD): main characteristics are reported in Table 1. AFAM measurements were repeated implementing three different probes with polycrystalline diamond coating. Concurrently, measurements were performed by means of nanoindentation instrument (Nanotest Platform, by Micro Materials). As reference for both instruments, the same (100)-oriented silicon used as a substrate was implemented. For each sample 20 nanoindentation measurements were performed: 10 with maximum load of 0.5 mN and 10 with maximum load within 1 and 5 mN. For these maximum loads, only the properties of the film are investigated avoiding substrates effects. Measurements of Young modulus from the nanoindenter and from the three different AFAM probes are reported in Figure 2. Values relative to uncoated silicon indicate AFAM data correction after calibration. Uncertainty for AFAM measurements was calculated including the standard deviation from measurements on 256×256 points (i.e. scan sampling), tip wear and calibration (i.e. relative standard deviation of nanoindentation measurements on reference surface). For nanoindentation, bars indicate standard deviation from measurements on 20 different points for nanoindentation.

Table 1: Main samples characteristics.

	M_5	M_{10}	M_{30}	M_{50}
Number of layers	5+5	10+10	30+30	50+50
Organic layers thickness [nm] (O)	340	170	56.6	34
Ceramic silica layers thickness [nm] (C)	60	30	10	6
Total O + Total C [μm]	1.7+0.3	1.7+0.3	1.7+0.3	1.7+0.3
Average roughness Sa [nm]	0.6	0.6	0.9	1.7

4 Discussion and conclusions

Agreement between nanoindentation and AFAM measurements was proved in particular for softer coatings (namely M5 and M10 in Table 1). A slightly worse agreement and larger deviations were experienced for stiffer samples (M30 and M50). This discrepancy was associated with two main factors: firstly the stiffer interaction between the tip and the surface probably caused instabilities and vibrations during measurement; secondly, samples M30 and M50 presented a higher average roughness (see Table 1) with a consequent larger variability on tip to surface interaction area and therefore larger deviations in measured data.

As a general statement, it is crucial to identify reference samples and standards which resemble actual surfaces not just in terms of contact stiffness, but also in terms of relative roughness. Indeed, while the calibration keeps linear even through one order of magnitude in contact stiffness, different results can be expected when roughness has sensible variations in terms of spacing and amplitude in particular when comparable with tip radius of curvature. Once proper reference surfaces are identified, AFAM demonstrates to be a valid characterization technique, for non destructive quantitative mapping of surface mechanical properties.

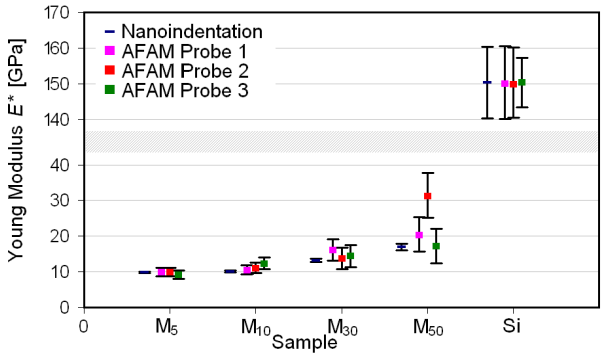

Figure 2: Results from nanoindentation and AFAM measurements.

References:

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