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Atomic force acoustic microscopy: Influence of the lateral contact stiffness on the elastic measurements

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ABSTRACT

Atomic force acoustic microscopy is a dynamic technique where the resonances of a cantilever, that has its tip in contact with the sample, are used to quantify local elastic properties of surfaces. Since the contact resonance frequencies (CRFs) monotonically increase with the tip-sample contact stiffness, they are used to evaluate the local elastic properties of the surfaces through a suitable contact mechanical model. The CRFs depends on both, normal and lateral contact stiffness, k_N and k_S respectively, where the last one is taken either as constant ($k_S < 1$), or as zero, leading to uncertainty in the estimation of the elastic properties of composite materials. In this work, resonance spectra for free and contact vibration were used in a finite element analysis of cantilevers to show the influence of k_S in the resonance curves due to changes in the k_S/k_N ratio. These curves have regions for the different vibrational modes that are both, strongly and weakly dependent on k_S, and they can be used in a selective manner to obtain a precise mapping of elastic properties.

1. Introduction

Atomic force acoustic microscopy (AFAM) is a technique where the resonance of a cantilever with its tip contacting the sample surface is measured, and the excitation of the system is achieved through a piezoelectric device located under the sample [1–3]. This technique is particularly useful for studying the mechanical properties at nanometric level of materials such as biological samples [4], polymers [2,5], alloys [6] and others [7,8]. Depending on the mechanical properties of the material, the contact resonance frequencies (CRFs) obtained with the same probe or cantilever, take higher values as the contact stiffness in the tip-sample system increases. However, the sensibility and precision to detect changes in contact stiffness depend of the slope in the resonance curves (CRFs vs contact stiffness) which are specific of each vibration mode of the cantilever [9]. The resonance curves can be obtained by means of analytical equations [3,10,11] or by simulation [12,13] (or even with experimental data, which requires reference samples [12]), where the tip-sample contact situation is modelled by means of three orthogonal springs; their constants describe the contact stiffness, k_N for the spring normal to the sample surface and k_S for the springs in-plane with the sample surface [14,15]. In analytical expressions, with an idealized cantilever model, the tip-sample contact is generally approximated as a spring (where its spring constant is the contact stiffness k_N) in parallel with a dashpot (to take into account viscoelastic properties of the surface) [2,4,10,16,17]. On the other hand, contact models with idealized cantilever that consider the lateral contact stiffness in one direction (k_L, located along the sliding direction of the cantilever tip) [2,3,18,19] assume that the tip-end produces a Hertzian contact on the tested surface, so the ratio of the lateral stiffness to the normal stiffness varies from 2/3 to 18/19 with a typical value of 4/5 for isotropic homogeneous materials [20]. However, in practice, k_S/k_N ratios much smaller than 2/3 have been observed. There are reported values of 0.05 [14,21] and 0.2 [5,15], using a model of three orthogonal springs in the contact zone that were obtained by comparing the experimental and simulated CRFs. These low values of the k_S/k_N ratios when compared to those predicted by single-asperity Hertzian contact [3], could be related to the type of contact during the acquisition of AFAM spectra, including, additionally, the frictional behavior of the surface [22]. Studies on the contact mechanics of the cantilever tip on surfaces during AFAM test have indicated that the geometry of the tip-end is better represented by a paraboloid of revolution than by a hemispherical one, which becomes predominantly flat punch after a short time [23,24]. Studies of the frictional behavior of crystalline materials

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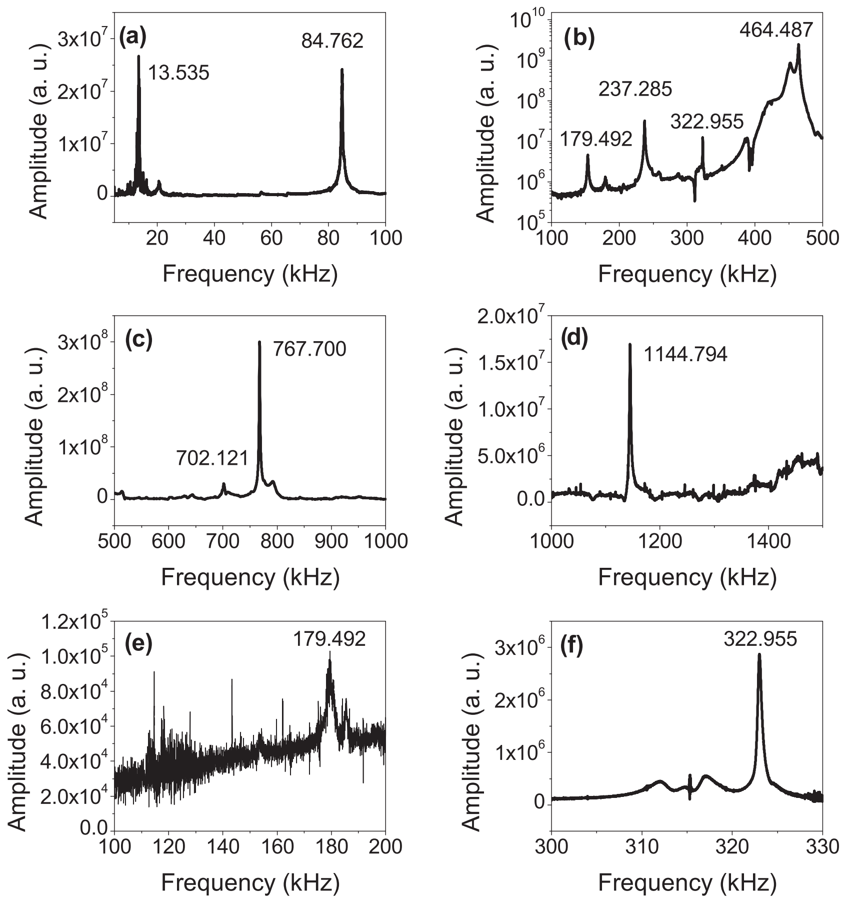

Fig. 1. Experimental spectra for free vibration of a silicon AFM cantilever coated with diamond like carbon (DLC), obtained from deflection (a–d) and torsional (e and f) signal of the AFM system.

Table 1
Experimental and simulated resonances and vibration mode of an AFM cantilever in free vibration.

Experimental frequency (kHz)	Simulated frequency (kHz)	Vibration mode
13.535	13.413	Flexural
84.762	83.999	Flexural
179.492	179.492	Torsional
237.285	234.880	Flexural
322.955	396.694	Torsional
464.487	464.484	Flexural
702.121	542.524	Torsional
767.700	767.700	Flexural
–	935.393	Torsional
1144.794	1144.794	Flexural
1595.719	–	Flexural

have shown strong differences in the friction force due to the structure [25] and to the crystallographic orientation [26], making the assignation of friction and k_S for a particular material very difficult. These considerations given above lead to uncertainty in the selection of the best k_S/k_N ratio, especially when the elastic properties of composite materials are evaluated, due to the inherent heterogeneities of the material. Thus, the fact of neglecting the existence of k_S, or assuming a k_S/k_N constant relationship ignoring its influence on the resonance curves [7] can lead to strong uncertainties in quantifying the elastic properties.

Experimentally, the contact resonance frequency (CRF) for a specific mode of vibration is obtained by monitoring the amplitude and phase signals, via lock-in amplifier, during a resonance sweep of the excitation signal around the CRF of interest. Under such conditions, spectra of amplitude and phase are acquired in the location of interest. In mapping configuration, this experiment is repeated in an array of N rows by M columns acquiring, in each intersection (N, M), the corresponding spectra. Observables such as amplitude (A), phase (φ), resonance frequency (ω_0) and quality factor (Q) are obtained by fitting a function of a simple harmonic oscillator (SHO) to the experimental spectra [19,27,28]. The ω_0 values obtained from the fitting process contain the resonance information of the tip-sample system, this data can be transformed to contact stiffness using the resonance curve of the vibrational mode of interest and then, through a suitable contact model (paraboloid of revolution or flat punch [23,29]), to the elastic modulus. The conversion to elastic modulus requires knowing the radius of the contact area (a_C), which experimentally is difficult to assess and generally this problem is addressed using a reference sample assuming that a_C of the reference should be equal to that of the interest sample [30].

In this work, we analyze the dependence of resonance curves of AFM cantilevers with the lateral contact stiffness (k_S) to show zones of convergence where the resonance curves are independent of k_S and regions where the influence of k_S is highly considerable, conducting to a high uncertainly in the quantification of the elastic properties. Also, we present an experimental procedure to determine a_C, which allows expressing the resonance curves as CRFs vs elastic modulus, in order to quickly determine the elastic properties of unknown samples using AFAM.