

Thin film characterization by atomic force microscopy at ultrasonic frequencies

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We present a technique in which atomic force microscopy at ultrasonic frequencies is used to determine the thickness of thin films. In this technique, the resonance frequency of a flexural mode of an atomic force microscope cantilever is used to determine the tip-sample contact stiffness. This allows the film thickness to be determined, provided that the tip and sample elastic moduli and radii of curvature are known. We report experimental results for thin metal and polymer films deposited on silicon substrates and compare them with the predictions of a theoretical model. © 2000 American Institute of Physics. [S0003-6951(00)00114-5]

The introduction of the atomic force microscope¹ (AFM) was soon followed by intense interest in its use as a probe for the hardness and elastic modulus of very small ($\sim \text{nm}^3$) volumes. Techniques in which the AFM is used to measure local elastic properties include force modulation microscopy,² the force curve method,³ nanoindentation,⁴ pulsed force mode,⁵ ultrasonic force microscopy,⁶ scanning microdeformation microscopy,⁷ and a technique in which contact resonances are measured⁸ (also referred to as atomic force acoustic microscopy⁹). In the latter method, the resonance frequencies of the cantilever flexural modes are used to determine the tip-sample contact stiffness.

In this letter, we report the application of atomic force acoustic microscopy to the measurement of the contact stiffness of thin metal (copper and tungsten) and polymer (photoresist) films on silicon substrates. This information, combined with the Hertzian contact model and surface impedance modeling of the layered structure, allows the film thickness to be determined.

The experimental apparatus used in these experiments is illustrated schematically in Fig. 1(a). The cantilevers are made of single-crystal silicon and have integrated piezoelectric (zinc oxide) actuators. Their fabrication has been described previously.¹⁰ The cantilevers are mounted in a commercial AFM¹¹ and the constant force mode is used to keep the tip in contact with the sample with a constant average cantilever deflection. A sinusoidal excitation signal is applied to the actuator by a network analyzer. The optical lever method is used to sense the cantilever deflection. The signal from the quadrant photodetector (vertical deflection) is amplified and input to the network analyzer, thereby allowing the cantilever frequency response to be measured from dc to 3 MHz. The upper frequency limit is due to the attenuation of higher frequencies by the quadrant photodetector and amplifier.

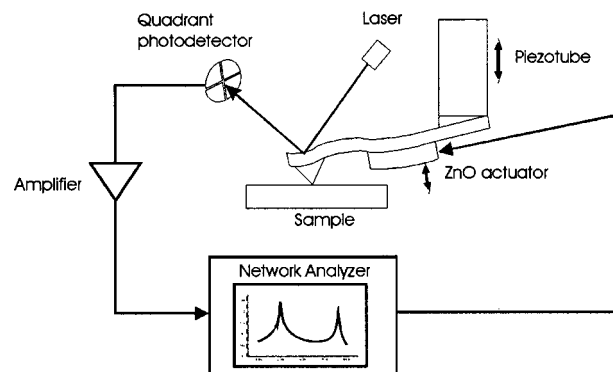
The tip-sample interaction is illustrated in Fig. 1(b). The forces acting on the tip include the cantilever deflection force (F_L), the capillary force (F_A) due to the water meniscus,¹² and the normal force (F_N) from the sample itself. The gen-

eralized Hertzian contact theory¹³ introduced by Fogden and White¹⁴ includes the effect of the water meniscus. In the experimental conditions under consideration¹⁵ however, the Hertzian load is simply the sum of the capillary force F_A and the cantilever force F_L :

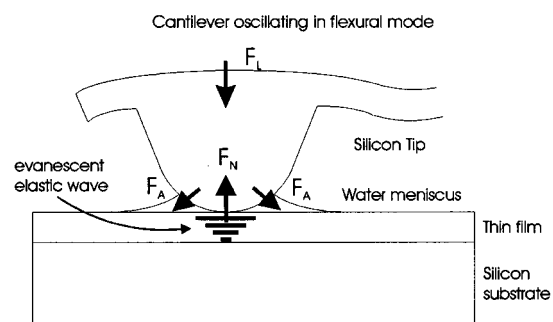
$$F_0 = F_A + F_L. \quad (1)$$

The contact stiffness is then given by

$$k = \sqrt[3]{6F_0RE^{*2}}, \quad (2)$$



(a)



(b)

FIG. 1. (a) Experimental setup used to measure in-contact flexural mode resonant frequencies of an AFM cantilever. (b) Forces acting upon AFM silicon tip when it is in contact with the sample in ambient conditions. F_L —force from cantilever. F_A —capillary force from water meniscus. F_N —normal force from sample. Oscillations in cantilever force F_L generate an evanescent elastic wave in the sample.

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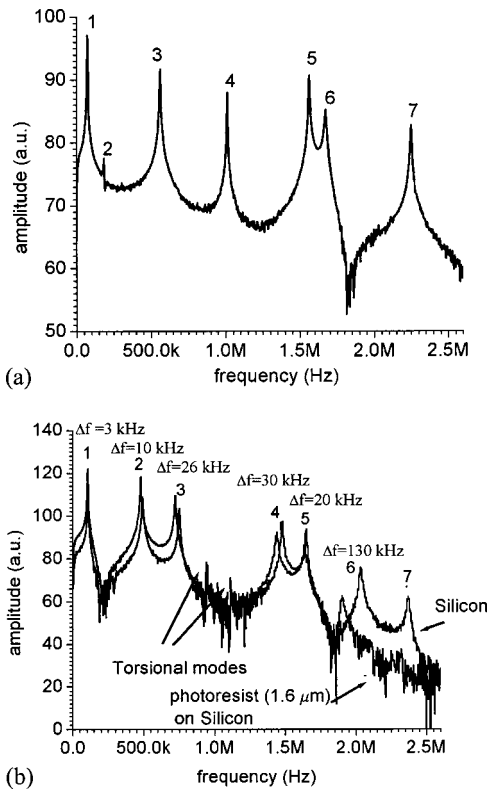


FIG. 2. (a) Free cantilever oscillation amplitude (arbitrary units) vs frequency. (b) Cantilever oscillation amplitude (arbitrary units) vs frequency. Cantilever resonances for tip in contact with silicon and in contact with photoresist (1.6 μm) on silicon. The two torsional modes, observed around 1 MHz, are largely insensitive to contact stiffness.

where R is the tip radius. E^* is the effective Young's modulus of the tip-sample interaction and is given by

$$\frac{1}{E^*} = \frac{1}{E_T^*} + \frac{1}{E_S^*}, \quad (3)$$

where

$$E_T^* = \frac{E_T}{1 - \rho_T^2} \quad (4a)$$

and

$$E_S^* = \frac{E_S}{1 - \rho_S^2}. \quad (4b)$$

E_T^* and E_S^* are the reduced Young's moduli of the tip and sample, respectively. E_S , E_T , ρ_S , and ρ_T are the Young's moduli and Poisson ratios of the sample and tip, respectively. The equation for E_S^* only applies, however, for bulk substrates. Layered materials require an approach based on surface impedance tensors.¹⁶

The tip-sample interaction stiffness may therefore be modeled as a lumped spring element, whose effect is to shift the resonance frequencies of the cantilever flexural modes (Ref. 9). Figure 2(a) shows the free cantilever resonances. The first seven flexural modes are shown. Figure 2(b) illustrates the difference between the cantilever resonances when the tip is in contact with silicon and in contact with a 1.6 μm photoresist film on silicon. The cantilever resonances are higher in frequency when the tip is in contact with the sample than for the free cantilever case. It is also clear from

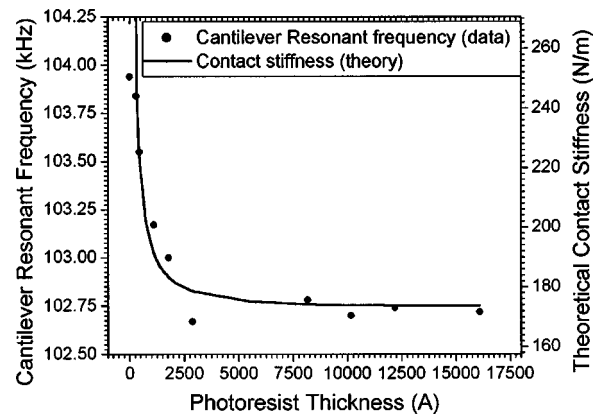


FIG. 3. Left-hand axis: resonant frequency (kHz) of first flexural mode of cantilever vs photoresist thickness (Å). Right-hand axis: theoretical contact stiffness (N/m).

this figure that the contact resonances are higher in frequency for silicon as compared to photoresist, which is due to the higher stiffness of silicon. Furthermore, it can be seen that the difference between the resonant frequencies of silicon and photoresist (denoted by Δf) is not the same for all cantilever flexural modes. Two torsional modes were observed around 1 MHz. However, torsional modes are expected to be sensitive to the lateral stiffness (see Ref. 8) of the tip-sample interaction rather than the contact stiffness and therefore have not been studied extensively in this investigation.

The high sensitivity of this technique to contact stiffness suggests that it may be used to measure the thickness of thin films. This has been carried out for photoresist, copper, and tungsten films on silicon. The substrates were p -type $\langle 100 \rangle$ 4-in. silicon wafers. The photoresist (Shipley 3612) was spun on to hexamethyldisilazane (HMDS) primed wafers at varying spin speeds to achieve the desired thicknesses. In order to achieve thicknesses below 3000 Å, it was necessary to thin the resist (Shipley type P thinner). The tungsten films were deposited by sputtering (Balzers 450). The copper films were deposited by evaporation. A chrome sticking layer (150 Å) was used for the copper films.

Figure 3 shows the measured resonance frequency (left-hand axis) of the first cantilever flexural mode as a function of photoresist thickness. The theoretical contact stiffness is also plotted (right-hand axis). This was calculated using the

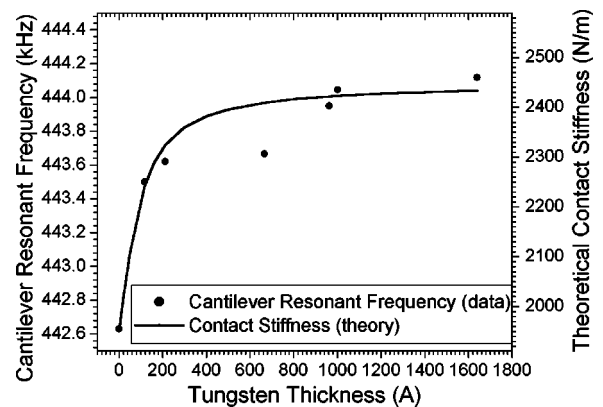


FIG. 4. Left-hand axis: resonant frequency (kHz) second flexural mode of cantilever vs tungsten thickness (Å). Right-hand axis: theoretical contact stiffness (N/m).

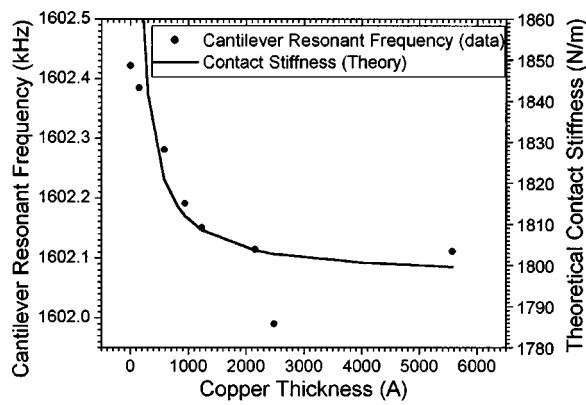


FIG. 5. Left-hand axis: resonant frequency (kHz) of fourth flexural mode of cantilever vs copper/chrome film thickness (Å). The first 150 Å of this film is a chrome sticking layer and the remainder is copper. Right-hand axis: theoretical contact stiffness (N/m).

method outlined in Ref. 16. In this calculation, the tip radius R was taken to be 220 nm and the Hertzian load F_0 to be 300 nN. The Young's modulus and Poisson ratio of the photoresist were taken to be 3.7151 GPa and 0.3712, respectively. For $\langle 100 \rangle$ silicon, the Young's modulus and Poisson ratio were taken to be 169 GPa and 0.278, respectively. From the figure, it is clear that the measured cantilever resonance frequency follows the same trend as the theoretical contact stiffness. As photoresist is softer than silicon, the contact stiffness will decrease as the photoresist thickness increases. The vibrating cantilever tip generates elastic waves in the photoresist film but, due to the small tip size, these waves are evanescent rather than propagating. Hence, the elastic waves have a limited penetration depth and for film thicknesses greater than this depth, the contact stiffness will be very close to that of a bulk photoresist sample. The experimental data suggest that, for photoresist, this technique is very sensitive for films below approximately 3000 Å.

The frequency of the second flexural mode of the cantilever has been plotted as a function of tungsten thickness in Fig. 4 (left-hand axis). The theoretical contact stiffness has also been plotted (right-hand axis). For this calculation, the tip radius R was taken to be 530 nm and the Hertzian load F_0 was taken to be 300 nN. The Young's modulus and Poisson ratio of tungsten were taken to be 361.83 GPa and 0.4378, respectively. Tungsten has a higher reduced Young's modulus than silicon so we expect the contact stiffness to increase with the tungsten thickness. The measured cantilever resonance frequency follows the same trend as the theoretical contact stiffness, apart from the data point at 670 Å. That this data point does not follow the general trend could be due to a lack of repeatability of the tip-sample contact (including the contact force) which would lead to a modification in the contact stiffness. In this experiment, it is observed that the technique is most sensitive for tungsten films thinner than approximately 500 Å.

The fourth flexural mode of the cantilever was used for the measurements on copper films. The frequency of this mode is plotted as a function of film thickness in Fig. 5 (left-hand axis). The theoretical contact stiffness has also been plotted (right-hand axis). From the figure, it is seen that the technique is very sensitive for samples thinner than approximately 2000 Å. The tip radius R and Hertzian load F_0 were taken to be identical to the values for tungsten as the same cantilever was used. The Young's modulus and Poisson ratio of copper were taken to be 126.2 GPa and 0.370, respectively. Copper is softer than silicon, so the contact stiffness decreases with the copper thickness. The measured cantilever resonance frequency follows the same trend as the theoretical contact stiffness, apart from the data point at 2480 Å (which may be due to the high surface roughness of these samples).

In summary, we have used atomic force acoustic microscopy to measure the contact stiffness of thin layers of metal and polymer films deposited onto silicon substrates. The behavior of the cantilever resonance frequency with film thickness is in agreement with theoretical calculations of contact stiffness.

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