研　究　速　報
特　集　9
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Trends in Nanoscale Science and Technology: Characterization of Thin Film Mechanical Properties

ナノスケール科学技術における傾向：薄膜の機械的特性の測定

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1. Introduction

Thin film materials are currently used in IC based process. The properties are almost scale dependent and are extremely sensitive to process conditions. Several techniques have been proposed and tested. We are investigating dynamic methods from which the exploitation of dynamic properties allows to determine mechanical properties such as Young's modulus and stiffness. The authors investigated also the characterization of thin film properties and different operation in Dynamic Atomic force microscopy from non contact to tapping mode.

2. Dynamic methods for Micro and nano Characterization of thin film materials

Different dynamic methods have been investigated:

2.1 Ultrasonic or acoustic wave velocity which is suitable for both thin and thick films. The measurement of the acoustic wave velocity allows to the calculation of mechanical properties. We investigated the contact less inspection by means of Electro-Magnetic Acoustic Transducer (EMAT).

2.2 Resonance method which is suitable for microstructures such as micro-cantilevers or micro-membranes. This methods refers to the calculation of Young's modulus through the value of resonant frequencies previously obtained experimentally[1]. Optical techniques for the measurement of resonant frequencies are currently employed and a global bench for a common use has installed for this purpose.

2.3 Local dynamic nano-indentation using scanning force microscope operating in tapping mode. this method is very suitable for local characterization in nanoscale range. Measurement of frequency shift of the vibrating cantilever while touching intermittently the sample, allows to determine the local stiffness. The knowledge of the interaction mechanisms between the tip and sample should be well controlled.

3. Tip-sample interactions in nanoscale

We made our own force microscope in order to study the interactions that occur when the vibrating beam is perturbed by a surface sample. For our experiments, we have been using different types of cantilever with different dimensions. To operate at high frequencies we have been using silicon microcantilever supporting a very sharp tip. On the figure 1, the vibration detection is insured by our 2 laser beams optical technique ; optical beam deflection and interferometry[2].

The interaction between the cantilever and the sample or between the tip and the sample depends drastically on their separation. On the figure 2 one can see five range of interaction

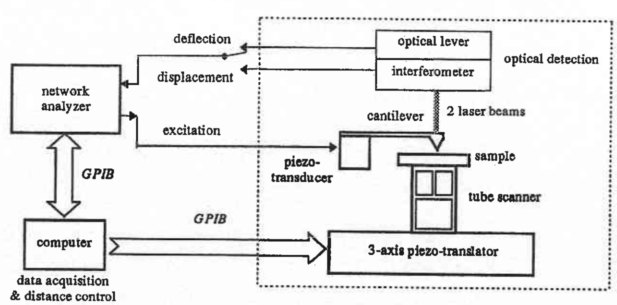

Fig. 1　Force microscope for tip-sample interaction measurement

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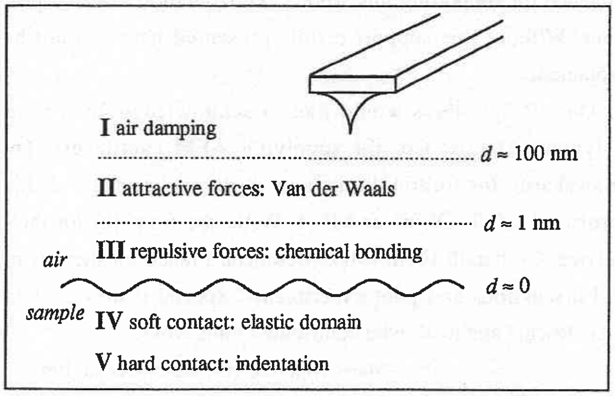

Fig. 2 Different probe-surface dynamic interaction as a function of tip-sample distance d

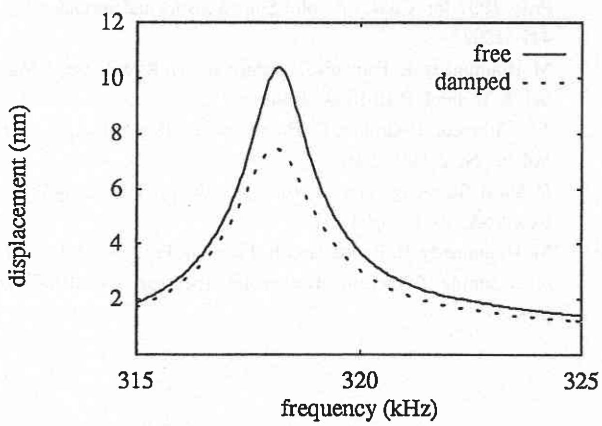

Fig. 3 variation of amplitude due to air damping

either from mechanical or electromagnetic origin. The amplitude of vibration, material of both tip and surface have to be taken into account to estimate the potential of the interaction.

The experimental setup is able to detect the resonance frequency shift or the decrease in the vibration amplitude when the lever is driven close to the resonance. The five domains are illustrated by the following figures:

Domain I: viscoelastic damping

The effect of the air damping on the vibrating beam occurs at large cantilever-sample separation. It can be used to detect the proximity to the surface of few micrometers. Figure 3 shows that damping leads to a strong amplitude decrease without major shift of the resonance frequency.

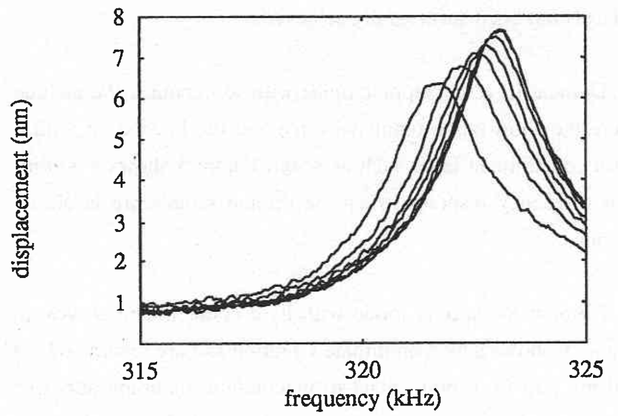

Fig. 4 resonance frequency shift due to Van der Waals attractive forces

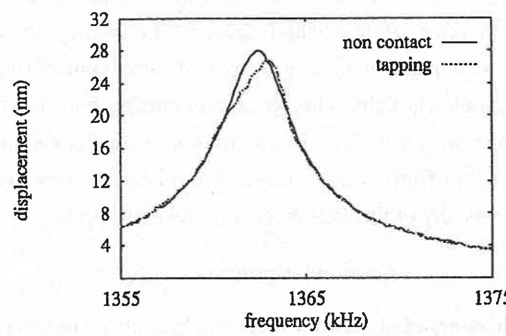

Fig. 5 frequency response for soft tapping mode (second mode)

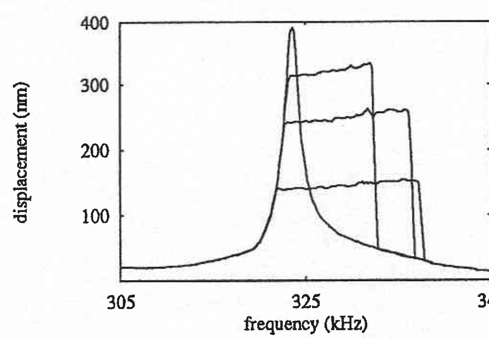

Fig. 6 frequency response obtained with a large amplitude of vibration

Domain II: Van der Waals attraction

We can detect force gradient acting on the tip by measuring the resonance frequency shift[3]. Van der Waals forces are attractive[4], then the additive compliance leads to a decrease of the cantilever spring constant and thus to negative shift of its resonant frequency. While the approach of the cantilever to the sample, frequency response of the first flexural mode have been measured for different Tip/Sample distances. Figure 4 shows different responses obtained with Tip/sample distance of each 10 nm gap, from 70 nm to 10 nm. Attractive force in the order of 1pN has been successfully achieved.

Domain III & IV: tapping mode with soft contact. We include here the short-range repulsive force and the interaction with a thin contaminant layer such as water. Figure 5 shows a nonlinear frequency response when the tip and sample are in elastic contact.

Domain V: tapping mode with hard contact. The curves on figure 6 shows a high amplitude vibration and are separated by a 50 nm gap from noncontact to indentation including adhesive forces.

These figures show clearly that operation in tapping mode allows to increase of resonant frequency. Frequency shifts depend of many parameters such as applied force, contact time and local sample elasticity. This result is promising since it will allow to measure local elasticity of any kind of materials and specially of thin films which for which mechanical properties depends drastically of the used process for their elaboration 5.

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