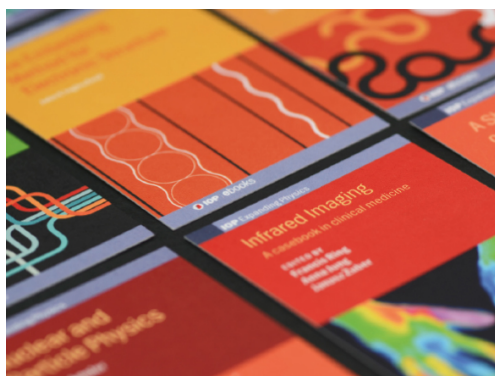# Force Modulation and Dynamic NanoIndentation Microscopy

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Force Modulation and Dynamic NanoIndentation Microscopy

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Abstract. We have made a Dynamic Nanoindentation Microscope (DNM) setup based on sample modulation, in order to allow a direct comparison between various dynamical mechanical measurement techniques such as Force Modulation Microscopy (FMM) and Dynamic Mechanical Analysis (DMA). The microscope is integrated to a standard Atomic Force Microscope (AFM) and uses a commercial nanoindentation system. Instead of the standard bimorph and force modulation configuration, we used a stacked ceramic sample actuator in displacement modulation. Both DMA measurements and DNM imaging were performed on each sample for the determination of the reduced, storage and loss modules, and a good agreement between the techniques have been found. Compared to FMM, we show that DNM has the advantage of always keeping the same contrast in the viscoelastic images as a function of the frequency.

1. Introduction

Among the various Scanning Probe Microscopy (SPM) modes, Force Modulation (FMM) has become a broadly used instrument for imaging the mechanicals properties of surface. Although many issues have been extensively reported in the literature, quantitative measurement remains laborious. Indeed, the choice of the cantilevers stiffness gives either good force sensitivity or good materials properties measurements and the overall cantilevers deflection includes uncontrolled and nonlinear tip/surface interaction. Measurement at high frequency removes some of those issues [1,2] but still do not offer quantitative measurements and often present ambiguous viscoelastic images [3].

On the other hand, dynamic or *ac*-nanoindentation [4] provides accurate materials properties measurements which can directly be compared to well-established Dynamic Mechanical Analysis (DMA) [5]. Scanning *ac*-nanoindentation in force modulation provides with quantitative images of materials mechanical properties [6]. Compared to FMM, dynamic nanoindentation microscopy gives a better control of the probe shape and contact area, less probe aging, while keeping lateral and vertical resolution in the nanometer range. So far, existing dynamic nanoindentation setups are based on force modulation while FMM is operated in both force and sample modulation.

2. Dynamical Mechanical Measurements

In all the above mentioned methods (DNM, DMA and FMM), a sinusoidal mechanical stress created either by displacement or force modulation is applied to the sample. The measurement of the phase shift and the amplitude of the resulting strain give the complete mechanical information of the sample,

as a viscoelastic material shows both a in phase (storage modulus E') and an out of phase (loss modulus E'') component of stiffness.

In standard DMA, E', E'' and the loss factor $\tan\delta = E'/E''$ are often measured as a function of the temperature or as a function of the frequency. In polymers science, this allows the determination of the dynamical glass transition, the characterization of the phase structure of mulitcomponent materials and the characterization of physical and chemical aging processes [7].

3. Instrumental Setup

The setup of our dynamical nanoindentation microscope is shown in figure 1. It consists of a displacement sample actuator (PICMA from Physik Instrumente GmbH), a nanoindentor head equipped with capacitive displacement sensing (Triboscope from Hysitron Inc.) and a scanning probe microscope base with piezoelectric scanner and related XYZ scanning electronics (AutoProbe CP from Park Scientific Instruments). A conical diamond indenter tip is screwed into the tip holder on the center plate, which is spring mounted to the frame and the outer plates are fixed; the indenter can move in the space between the two fixed plates.

The sample is mounted on top of the piezo actuator assembly, which itself sits atop of the AFM scanner. As the sample position is modulated (Ao sinωt), the displacement Zo sin(ωt+ϕ) of the indenter probe is monitored as the change of the capacitance between the plate on which the probe is mounted.
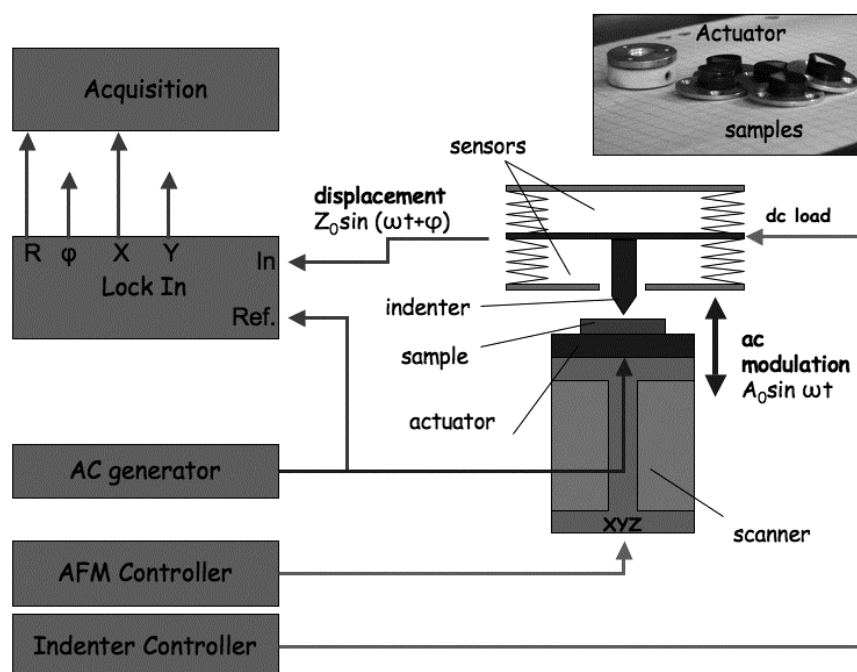

Figure 1. Dynamical nanoindentation microscope setup.

The sample actuator, made from a multilayer piezo element strongly attached between the aluminum sample holder and a macor base, offers a high resonant frequency (>300 kHz), a reliable piezoelectric effect at low operating voltage which allows modulation down to 1nm peak-to peak and very low temperature drift. The sample holder and actuator can be used for FMM measurements as well, allowing consistent experimental conditions between the two techniques, FMM and DNM. All

sample specimens have been encapsulated with phenol formaldehyde resin (Resole) and prepared at identical size and weight.

The probe displacement signal were measured with a dual phase lock-in amplifier (SR-830 from Stanford Research Systems), with the sample modulation as the reference signal. The storage modulus E' (X=R cosϕ) and the loss modulus E'' (Y=R sinϕ) were then imaged, with R and ϕ being the amplitude and the phase lag of the probe displacement relative to the sample displacement. For the excitation voltage we used an arbitrary waveform generator (33220A from Agilent Technologies) with frequencies ranging from 5Hz to 200Hz in DNM and sample modulation ranging from 5nm to 100nm.

For FMM measurement, we used a conventional AFM head and cantilever instead if the nanoindentor head and probe and higher frequencies (typically 10 kHz). To allow comparison, we have used the same sample actuator and the same acquisition system.

In both setup, after properly chosen the sample modulation amplitude and a frequency above the resonance frequency of the sample/probe system, we used the AFM control unit for the acquisition of the measured signals coming from the lock-in. The images, according to the signal displayed, show either E' or E'' for each single pixel measured. For FMM, it is more common to show ϕ signal (usually named "phase image") which is usually interpreted as the energy dissipation at the sample/probe junction.

4. Illustrative Examples

4.1. FMM Imaging

A polytetrafluoroethylene (PTFE) / Resole interface has been imaged using FMM (Figure 2). The experimental condition were the following: Si_3N_4 cantilever with ω_o =15 kHz and k = 0.27 N/m, sample modulation of 10nm peak-to peak at frequencies ranging from 10 to 30 kHz (as shown on the picture), a *dc* load of 15nN and a scanning rate of 0.75 Hz for a scanning area of 15μm x 50μm.

Figure 2 shows the topographic image (top) and the phase images (bottom) taken at various frequencies, bellow and above the resonance frequency (18 kHz). We see a contrast inversion on the phase images. Figure 3 is a cross section of the phase images for each frequency, for the same scanned line "A". Apart from the phase inversion, we see a much better signal contrast for the higher frequency.
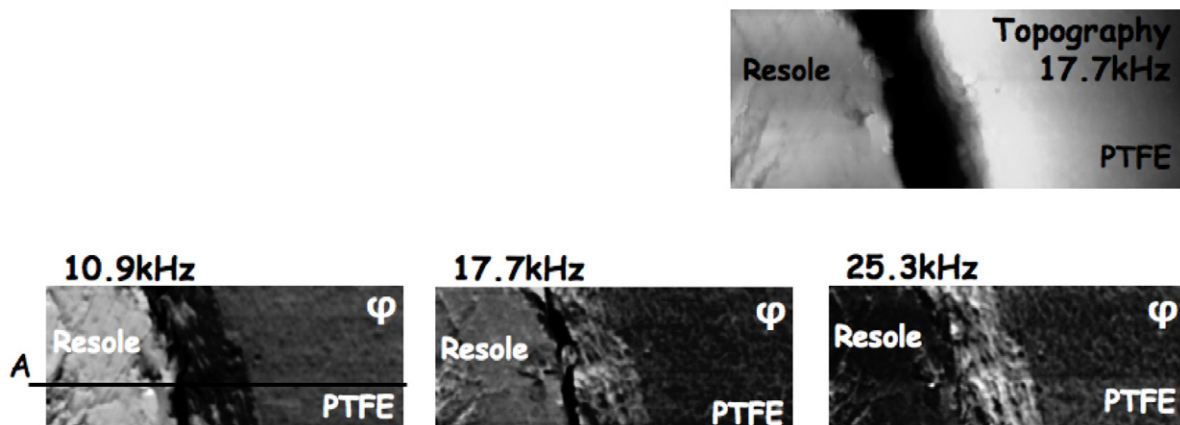

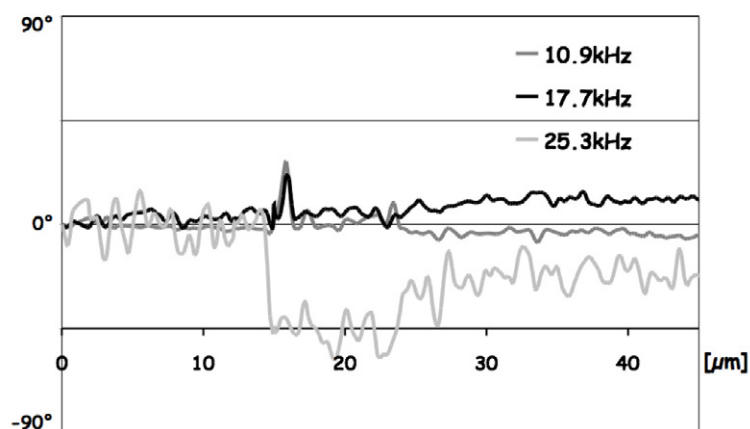**Figure 2.** Topographic and phase images of PTFE/Resole interface.

Figure 3. Phase image cross sections of lines "A".

4.2. DNM Imaging

In Figure 4, DNM technique has been used to image a polyvinylchloride (PVC) / Resole interface. The resonant frequency of the system in contact appeared to be 194Hz. Sample modulation was 50nm at frequencies ranging from 190 to 260 Hz, and a *dc* load of 10μN has been selected. Scanning rate of the acquisition was 0.2Hz for a scanning area of 5 μm x 5μm.

Figure 4 shows topographic image (left) and E'' images at various frequencies, bellow and above the resonance frequency. E'' images are very similar at all frequencies and present neither inversion nor significant contrast deviation. Figure 5 is a cross section of the E'' images for each frequency for the same scanned line "B".
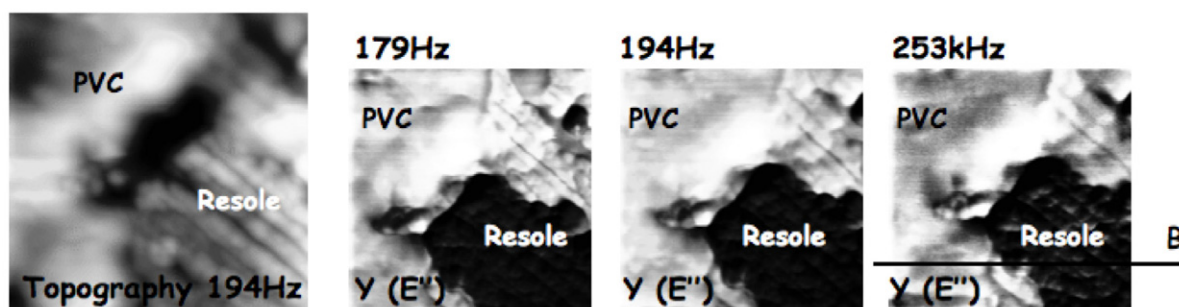

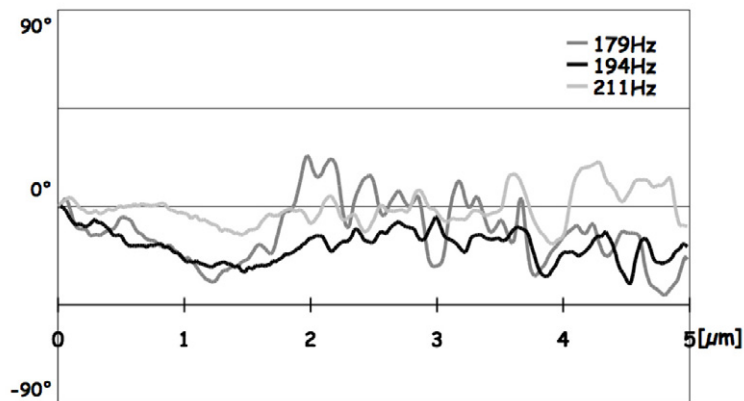**Figure 4.** Topographic and E'' images of PVC/Resole interface.

Figure 5. E'' image cross sections of lines "B".

5. Acknowledgments

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