

Model-Based Identification of Nanomechanical Properties in Atomic Force Microscopy

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Abstract— The ability of the atomic force microscope (AFM) to resolve highly accurate interaction forces, has made it an increasingly popular tool for determining nanomechanical properties of soft samples. In this short paper, a model-based technique for resolving nanomechanical properties is presented. Both the sample and cantilever are represented by dynamic models. A recursive least squares method is then employed to identify the unknown parameters of the sample model, thus revealing its nanomechanical properties and simultaneously enabling identification of time- or space-varying parameters. The approach naturally handles nonlinear sample models without linearization, such as the Hertz contact model as demonstrated. Experimental results performed on a commercial AFM are presented.

I. INTRODUCTION

Atomic force microscopy (AFM) is a versatile tool capable of imaging rigid and soft samples at nano- to micrometer resolutions [1]. Due to its inherent cantilever-laser setup, the microscope is capable of measuring tip-sample interaction forces at the piconewton range, thus making it highly suitable for studying nanomechanical properties of materials [2].

Traditionally, by measuring the static force response as the cantilever tip indents a soft sample, elastic properties of the sample can be revealed from the resulting force-distance curves [3]. More recently, considerable efforts have been placed toward revealing additional nanomechanical properties through multifrequency approaches [4]–[6]. These efforts typically involve relating the observables in either single- or multifrequency dynamic modulation to mechanical properties of the sample. However, these relations are often quite complicated and are still under development [7]. Additionally, due to the nonlinear nature of the interaction forces, it can be argued that frequency domain techniques are not fully suitable, with the need to handle the resulting higher harmonics and the wide use of linearization operations. Thus, existing approaches are limited in terms of the sample properties that can be extracted, typically isolated to viscoelastic properties and their gradient along the depth axis.

In [8] – on which this short paper is based upon, by the same authors – a unique approach is presented, where both the sample and cantilever are represented by separate dynamic models. By employing identification techniques

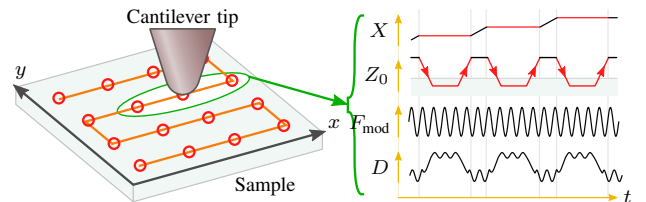


Fig. 1: The cantilever tip is regularly indented into the sample, pausing the lateral movement during each indentation. The cantilever is oscillated during the entire procedure, enabling identification of dynamical properties such as viscosity.

from the control literature, the parameters of the dynamic sample model can be identified from the observable signals. The observables are mapped to the sample parameters using a recursive least squares method. The procedure is operated entirely in the time-domain, thus, circumventing the need for linearization or demodulation. The cantilever and sample model separation makes it easy to substitute either one of them to fit any given application. The mode as implemented operates by regularly indenting into the sample as illustrated in Fig. 1.

II. SYSTEM MODELING

In this section, the dynamics governing the AFM cantilever interacting with the sample are established. The interaction between the various components of the system is shown in Fig. 2. The cantilever dynamics are subject to an external tip-sample interaction force F_{ts} , as well as a modulating input force F_{mod} . The resulting cantilever deflection, as well as the z -actuator position, determines the tip position Z . As the tip indents the sample at depth δ , restoration and viscous forces from the sample are acting on the tip.

The cantilever dynamics can be approximated by its first resonance mode [9], resulting in the spring-damper system

$$M\ddot{D} + KD + C\dot{D} = F_{mod} + F_{ts} \quad (1)$$

where M is the effective mass of the cantilever [10], and K, C are the cantilever spring constant and damping coefficient respectively.

Various contact models can be used to model the sample in AFM, the most widely used being the Hertz model describing the elasticity of soft samples [11], [12]. Here, a modified version of the Hertz model is used by including an

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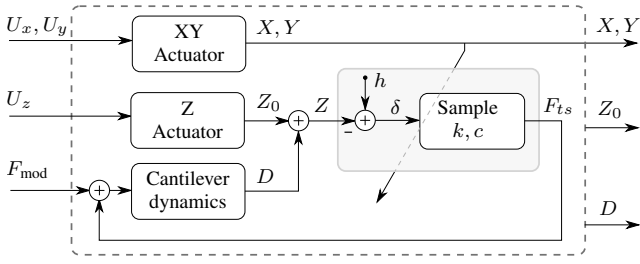


Fig. 2: Plant dynamics and corresponding inputs and outputs. The sample properties – here k, c, h – are to be identified.

additional viscous term. The resulting viscoelastic tip-sample interaction force is given by

$$F_{ts}^B = E' \delta^{\frac{3}{2}} + c \dot{\delta} \quad (2)$$

$$E = \frac{3}{4} R^{-\frac{1}{2}} (1 - \nu^2) E' \quad (3)$$

where E is the elastic modulus of the sample, E' is the variable identified by the parameter estimator and proportional to the elastic modulus, R is the cantilever tip radius, and ν is the Poisson ratio of the sample.

III. PARAMETER IDENTIFICATION

In this section, an online estimation scheme for identification of the unknown sample parameters, c, E , is established. The cantilever deflection D and indentation depth δ are considered known signals during estimation, while the cantilever dynamics from the previous section, represented by the parameters M, K, C , are considered fixed during experiments.

By applying the interaction force (3) to the cantilever model (1), the equations can be rearranged and rewritten in the complex s -domain as

$$Ms^2D + CsD + KD - F_{mod} = cs\delta + E'\delta^{1.5}. \quad (4)$$

Defining

$$w' \triangleq Ms^2D + CsD + KD - F_{mod} \quad (5)$$

the system (4) can be rewritten in parametric form as

$$w' = \begin{bmatrix} c \\ E' \end{bmatrix}^T \begin{bmatrix} s\delta \\ \delta^{1.5} \end{bmatrix} \quad (6)$$

$$= \theta^T \phi' \quad (7)$$

which gives the parametric formulation of the sample model.

Several estimation methods for the system (7) can be employed with similar stability and convergence properties. We have chosen the least squares method with forgetting factor from [13], due to its greater ability to suppress measurement noise over comparable techniques. In [8], it is shown that the signal vector ϕ is persistently exciting, thus exponential convergence of the estimated parameters is guaranteed [13]. Furthermore, an expression was found which determines how long the parameter estimator needs to run during a given indentation, in order to guarantee convergence to any specified error a priori.

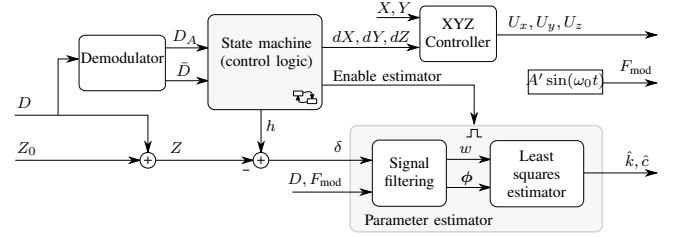


Fig. 3: Block diagram of the control logic and parameter estimator.

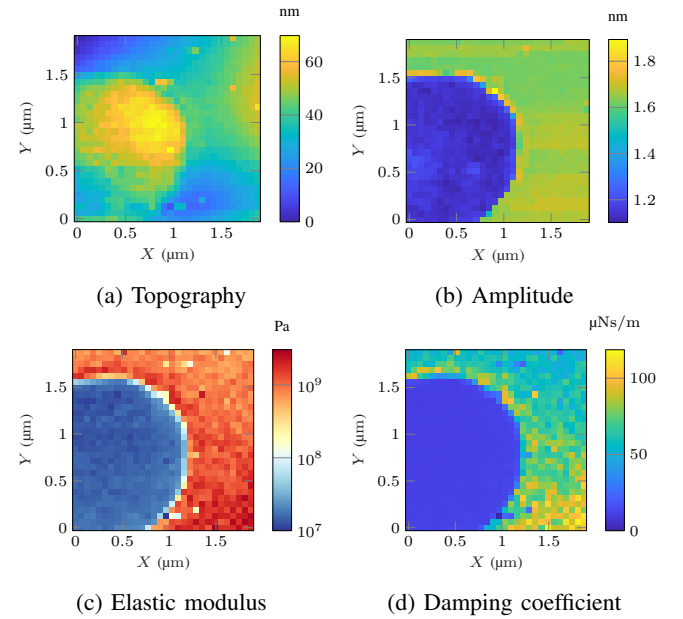


Fig. 4: AFM experiment for a two-component polymer sample.

IV. EXPERIMENTAL RESULTS

The control logic and parameter estimator is implemented according to the block diagram shown in Fig. 3. The state machine controls the logic of the operation, commanding the indentation of the cantilever into the sample as well as the positioning of the sample in the lateral directions. A demodulator is implemented exclusively for determining point of contact, and does not take part in the parameter estimation.

The method was implemented on a commercial AFM (Park Systems XE-70) using a spherical carbon tip cantilever with 40 nm tip radius (B40_CONTR). The system parameters M, K, C need to be known before experiments, and were determined using standard techniques [3], [8], [14]. The indentation time necessary to guarantee convergence of the parameters to 0.001% of the initial error was found to be $T = 1.13$ s.

The first experiment was performed to demonstrate the normal operating procedure of DIVE mode AFM, revealing spatially varying viscoelastic properties of the sample. A total of 30×30 indentations into a PS-LDPE-12M film sample were performed. This two-component polymer sample has

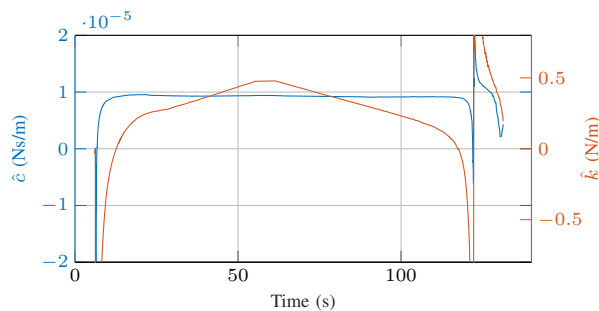


Fig. 5: Time-varying parameter estimates during indentation. The estimated spring constant changes with the indentation depth. Unmodeled adhesion effects give rise to unreliable results at the start and end of the experiment.

specified elastic moduli of around 0.1 GPa and 2 GPa for the two components. The results are shown in Fig. 4, where the contrast in elasticity between the two polymer components is clearly visible, and the resulting elastic moduli are close to their reference values. The amplitude values from the demodulator implemented is also shown for reference.

Since the approach presented in this article uses a recursive parameter estimation scheme, the time-varying nature of the parameters can be recorded. This is demonstrated by performing a single indentation into a soft sample. If the sample complies with the Hertz model, then the spring constant in a linear spring-damper model should increase with increasing indentation depth.

In this experiment the cantilever tip is lowered into the sample and raised again, thereby resulting in time-varying parameters. The experiment was performed on a soft gel sample (PDMS-SOFT-1-12M) using a linear spring-damper model with parameters k, c instead of the Hertz model in (3). The results of the experiment are given in Fig. 5. The parameter estimates demonstrate that the spring constant generally increases with increasing indentation depth as expected, and decreases as the tip is raised again. Additionally, several unmodeled effects attributable to adhesion and deformation can be seen, resulting in unreliable results near the start and end of the experiment. In order to mitigate the unmodeled effects seen in this experiment, the sample model can be modified to include adhesion, such as by employing the Johnson-Kendall-Roberts (JKR) or Derjaguin-Muller-Toporov (DMT) contact models [15].

V. CONCLUSIONS

A model-based identification technique is presented for determining spatially resolved nanomechanical properties in AFM. Both the cantilever and sample behavior is described by dynamic models. The cantilever dynamics are assumed known and identified before experiments, while the sample dynamics incorporate the unknown parameters to be identified. A recursive least squares estimator is used for identification of the sample parameters.

From the results, it is clear that the implementation of a recursive estimator has several advantages over offline techniques. (i) Allowing online identification and enabling

the operator to see real-time results, (ii) identifying time-varying parameter changes, and (iii) revealing erroneous conditions or unmodeled dynamics, as this can dramatically affect the estimated parameters.

The modeling- and identification scheme presented is favorable over comparable techniques in the sense that it clearly separates the model of the cantilever and the sample, allowing modifications to either one separately. As an example, in order to mitigate the unmodeled effects seen in the last experiment, the sample model can be modified to include adhesion. Furthermore, the presented approach naturally handles nonlinear sample models as seen by the implementation of the Hertz model. In addition, the measured signals are used directly for identification instead of measured through demodulators, thus avoiding the time delay, bandwidth limitations, and ultimately loss of information introduced by demodulators.

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