Stiffness Calibration of Atomic Force Microscopy Probes Under Heavy Fluid Loading

by

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Department of Mechanical Engineering and Materials Science
Duke University

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Piotr E. Marszalek

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Stefan Zauscher

Dissertation submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy in the Department of Mechanical Engineering and Materials Science in the Graduate School of Duke University

2010
ABSTRACT
(Engineering—Mechanical)

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Abstract

This research presents new calibration techniques for the characterization of atomic force microscopy cantilevers. Atomic force microscopy cantilevers are sensors that detect forces on the order of pico- to nanonewtons and displacements on the order of nano- to micrometers. Several calibration techniques exist with a variety of strengths and weaknesses. This research presents techniques that enable the noncontact calibration of the output sensor voltage-to-displacement sensitivity and the cantilever stiffness through the analysis of the unscaled thermal vibration of a cantilever in a liquid environment.

A noncontact stiffness calibration method is presented that identifies cantilever characteristics by fitting a dynamic model of the cantilever reaction to a thermal bath according to the fluctuation-dissipation theorem. The fitting algorithm incorporates an assumption of heavy fluid loading, which is present in liquid environments.

The use of the Lorentzian line function and a variable-slope noise model as an alternate approach to the thermal noise method was found to reduce the difference between calibrations preformed on the same cantilever in air and in water relative to existing techniques. This alternate approach was used in combination with the new stiffness calibration technique to determine the voltage-to-displacement sensitivity without requiring contact loading of the cantilever.

Additionally, computational techniques are presented in the investigation of alternate cantilever geometries, including V-shaped cantilevers and warped cantilevers. These techniques offer opportunities for future research to further reduce the uncertainty of atomic force microscopy calibration.
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## Nomenclature

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<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>$A_n$</td>
<td>scaling coefficient</td>
</tr>
<tr>
<td>$a$</td>
<td>radius</td>
</tr>
<tr>
<td>$b$</td>
<td>cantilever width</td>
</tr>
<tr>
<td>$C$</td>
<td>scaling coefficient</td>
</tr>
<tr>
<td>$c$</td>
<td>viscous damping coefficient</td>
</tr>
<tr>
<td>$d$</td>
<td>probe tip height</td>
</tr>
<tr>
<td>$E$</td>
<td>Young’s modulus</td>
</tr>
<tr>
<td>$f$</td>
<td>force</td>
</tr>
<tr>
<td>$f_{\text{hydro}}$</td>
<td>hydrodynamic force</td>
</tr>
<tr>
<td>$f_{\text{hydro-segment}}$</td>
<td>hydrodynamic force on a beam segment</td>
</tr>
<tr>
<td>$F_n$</td>
<td>modal forcing</td>
</tr>
<tr>
<td>$f_n$</td>
<td>natural frequency</td>
</tr>
<tr>
<td>$\tilde{f}_n$</td>
<td>fluctuating modal force</td>
</tr>
<tr>
<td>$f_r$</td>
<td>resonance frequency</td>
</tr>
<tr>
<td>$G$</td>
<td><em>in vacuo</em> cantilever transfer function</td>
</tr>
<tr>
<td>$H$</td>
<td>transfer function</td>
</tr>
<tr>
<td>$I$</td>
<td>second area moment of inertia</td>
</tr>
<tr>
<td>$i$</td>
<td>imaginary unit</td>
</tr>
<tr>
<td>$\text{Im}$</td>
<td>imaginary part</td>
</tr>
</tbody>
</table>
\( InvOLSC \): InvOLS determined through contact loading
\( InvOLS_N \): noncontact InvOLS estimation
\( J \): cost function
\( J_{FSI} \): FSI model cost function
\( J_R \): selection ratio cost function
\( J_{SHO} \): SHO model cost function
\( K \): hydrodynamic feedback controller
\( K_n \): modified Bessel function of the second kind, order \( n \)
\( k \): stiffness, normal cantilever stiffness
\( \hat{k} \): normalized stiffness
\( k_B \): Boltzmann’s constant
\( k_{eff} \): effective stiffness
\( \hat{k}_{eff} \): normalized effective stiffness
\( k_{eff,angle} \): effective stiffness with cantilever angle correction
\( k_{eff,t-len} \): effective stiffness with probe tip length correction
\( k_{eff,t-pos} \): effective stiffness with probe tip position correction
\( k_{eff,water} \): effective stiffness calculated in water
\( k_{est} \): stiffness estimate
\( k_H \): high frequency stiffness estimate
\( k_L \): low frequency stiffness estimate
\( k_n \): modal stiffness
\( k_{new} \): stiffness estimate, current iteration
\( k_{old} \): stiffness estimate, previous iteration
\( k_{Sader,air} \): stiffness estimate via Sader’s method in air
\( k_{unscaled} \): representative stiffness for identifying InvOLS

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<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>$l$</td>
<td>cantilever length</td>
</tr>
<tr>
<td>$L_0$</td>
<td>arbitrary length scale</td>
</tr>
<tr>
<td>$l_2$</td>
<td>cantilever length to probe tip</td>
</tr>
<tr>
<td>$m$</td>
<td>mass</td>
</tr>
<tr>
<td>$m'$</td>
<td>displaced fluid mass per unit length</td>
</tr>
<tr>
<td>$P$</td>
<td>closed-loop cantilever-fluid system</td>
</tr>
<tr>
<td>$P_{DC}$</td>
<td>DC power response</td>
</tr>
<tr>
<td>$P_n$</td>
<td>’fit-slope noise’ scaling coefficient</td>
</tr>
<tr>
<td>$P_p$</td>
<td>pink noise scaling coefficient</td>
</tr>
<tr>
<td>$P_r$</td>
<td>’square root noise’ scaling coefficient</td>
</tr>
<tr>
<td>$P_w$</td>
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</tr>
<tr>
<td>$PE$</td>
<td>percent error</td>
</tr>
<tr>
<td>$Q$</td>
<td>quality factor</td>
</tr>
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<td>$q$</td>
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<tr>
<td>$q_{ANSYS}$</td>
<td>cantilever displacement via ANSYS simulation</td>
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<tr>
<td>$q_{eff}$</td>
<td>effective cantilever displacement</td>
</tr>
<tr>
<td>$q_n$</td>
<td>modal displacement</td>
</tr>
<tr>
<td>$q_{ss}$</td>
<td>cantilever displacement via state space model</td>
</tr>
<tr>
<td>$\hat{q}$</td>
<td>normalized displacement</td>
</tr>
<tr>
<td>$\tilde{q}_n$</td>
<td>fluctuating modal displacement</td>
</tr>
<tr>
<td>$R$</td>
<td>selection ratio</td>
</tr>
<tr>
<td>$Re$</td>
<td>Reynolds number</td>
</tr>
<tr>
<td>$Re$</td>
<td>real part</td>
</tr>
<tr>
<td>$S_{cant}$</td>
<td>PSD of a cantilever dynamics model</td>
</tr>
<tr>
<td>$S_{data}$</td>
<td>PSD of a experimental data</td>
</tr>
</tbody>
</table>
$S_{\text{Lor}}$ : PSD of a Lorentzian

$S_{qq}$ : FSI model PSD

$S_{qq,n}$ : single-mode FSI model PSD

$S_{\text{SHO}}$ : PSD of an SHO

$S_{\text{SHO, white}}$ : PSD of an SHO with white noise

$S_{yy}$ : PSD of a cantilever-fluid system model

$s$ : complex frequency

$T$ : temperature

$t$ : cantilever thickness, time

$U_0$ : peak velocity

$V$ : voltage

$V_{\text{max}}$ : maximum photodiode voltage

$V_{\text{sum}}$ : sum of photodiode quadrant voltages

$W$ : mode shape

$w$ : displacement

$x$ : position along cantilever beam length

$\alpha_n$ : wavenumber

$\beta$ : noise slope coefficient

$\Gamma$ : the hydrodynamic function

$\Gamma_i$ : imaginary component of the hydrodynamic function

$\Gamma_{\text{ribbon}}$ : the hydrodynamic function for a thin ribbon

$\gamma$ : mass ratio

$\eta$ : dynamic viscosity

$\theta$ : cantilever angle to substrate

$\theta_b$ : cantilever bending angle
\( \mu \) : mass per unit length
\( \nu \) : kinematic viscosity
\( \rho_f \) : fluid density
\( \phi \) : cantilever tip angle to substrate
\( \chi \) : cantilever bending shape ratio
\( \Omega \) : hydrodynamic function correction
\( \Omega' \) : arbitrary geometry hydrodynamic function
\( \omega \) : angular frequency
\( \omega_H \) : high frequency estimate
\( \omega_L \) : low frequency estimate
\( \omega_r \) : angular resonance frequency
\( \omega_n \) : angular natural frequency

**Acronyms**

AFM : Atomic Force Microscope
CFD : Computational Fluid Dynamics
DC : Direct Current
DI : Deionized
EFB : Electrostatic Force Balance
FEM : Finite Element Method
FSI : Fluid-Structure Interaction
FWHM : Full-width at half-maximum
InvOLS : Inverse Optical Lever Sensitivity
NIST : National Institute of Standards and Technology
NPL : National Physical Laboratory
<table>
<thead>
<tr>
<th>Acronym</th>
<th>Definition</th>
</tr>
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<tbody>
<tr>
<td>PSD</td>
<td>Power Spectral Density</td>
</tr>
<tr>
<td>RC</td>
<td>Resistor-Capacitor</td>
</tr>
<tr>
<td>RMS</td>
<td>Root Mean Square</td>
</tr>
<tr>
<td>SHO</td>
<td>Simple Harmonic Oscillator</td>
</tr>
<tr>
<td>SI</td>
<td>International System of Units</td>
</tr>
<tr>
<td>SMFS</td>
<td>Single-Molecule Force Spectroscopy</td>
</tr>
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</table>
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I couldn’t agree more.
Chapter 1

Introduction

This research presents a new technique for performing force calibrations on atomic force microscopy cantilevers in liquid environments. It also contains a series of investigations into sources of error for such calibrations along with methods to mitigate their impact. In their seminal paper on the atomic force microscope (AFM), Binnig, Quate, and Gerber specifically identified the spring as a critical component of the device [4]. A measurement of the deflection of a spring can be used to determine the applied force, provided the stiffness of the spring is known with acceptable accuracy. The spring of an AFM is a microscopic cantilever beam, generally either rectangular or ‘V-shaped’. Cantilever beam stiffness is a simple function of the Young’s modulus of the beam material and the beam geometry; however, these properties are subjects of significant uncertainty stemming from the photolithographic processes used to manufacture microcantilevers. While these techniques enable the manufacturing of cantilever beams less than 1 µm in thickness, the associated thickness tolerances are such that manufacturers publish stiffness specification ranges that can vary by as much as a factor of five from the nominal value. Similarly, doping and coating processes alter the densities and Young’s moduli from the bulk properties of the cantilever materials.

Since AFM cantilevers cannot be manufactured with precisely known stiffnesses, they must be calibrated before they can be used to make force measurements. A great deal of effort has been spent pursuing calibration techniques [5, 6, 7, 8, 9, 10, 1, 11, 12, 13, 14]. Some existing calibration techniques are cumbersome, involving procedures that are comparable in complexity to the experiments that require the
calibrated cantilevers [5, 9, 15, 2]. Other techniques require only the measurement of the thermally driven, random vibration of a cantilever and perhaps knowledge of a few of the parameters characterizing the cantilever and its fluid environment [16, 17, 6]. Ideally, the process of calibrating a sensor is automatic, not requiring a user to have to develop a separate expertise in order to use the sensor. More importantly, the calibration of a sensor should be sufficiently precise and accurate to suit a user’s needs. The wide variety of calibration techniques currently available possess different strengths and weaknesses that users must be aware of to make scientifically valid observations. While commercial AFM systems have semi-automated calibration routines, there is still a significant amount of training and experience required to perform AFM force measurements. The goal of this research has been to create a new calibration technique that expands the set of circumstances where AFM calibration can be performed, to advance the understanding of the sources of error in existing techniques, and to identify new ways to reduce error when possible.

1.1 Research Motivation

AFM has been used to investigate the properties of biological molecules for over a decade [18]. In most cases, biological specimens require AFM operation in liquid environments. While it is acceptable to calibrate a cantilever in air before using it in liquid, it is more desirable to calibrate an AFM cantilever in situ. Air and liquid test setups can require different hardware and it is best to handle the fragile cantilevers as little as possible. More significantly, the refinement of calibration techniques for liquid environments could contribute to an increase in the accessibility of AFM measurements to a wider range of users.

One exciting potential application for AFM technology comes from the development of lab-on-a-chip devices. A single chip incorporating both AFM cantilevers
and the electronics required to perform automated calibrations and measurements could be populated with hundreds or even thousands of cantilevers. With sufficient development, such a device could be used by physicians or law enforcement agents to detect biological substances.

AFM-based lab-on-a-chip devices could be simplified if they had the ability to perform noncontact calibrations in liquid environments. Proper characterization of the thermal vibrations of a cantilever in air and in water requires different measurement parameters, specifically, different sample rates and displacement magnitude ranges to accommodate the two sets of system dynamics. Requiring a lab-on-a-chip to acquire data over multiple frequency and magnitude ranges adds to the complexity and cost of the design, therefore, the ideal situation would be for a lab-on-a-chip intended for liquid environments to be able to self-calibrate in liquid environments. Additionally, with the ability to periodically recalibrate itself in situ as a check for damage, such a sensor could be deployed to monitor liquids used in industrial settings or municipal water supplies.

The thermal noise method provides one path to liquid environment stiffness calibrations, however, with the availability of a new noncontact calibration technique, it could enable noncontact displacement calibration of a lab-on-a-chip device. The thermal noise method relates the stiffness to the magnitude of the free vibration of a cantilever; it requires calibrated displacement measurements to produce a stiffness calibration. Requiring a lab-on-a-chip to incorporate a calibrated actuator may present a significant barrier to its development, however, an alternate approach has already been demonstrated in air. In typical optical lever AFMs, the cantilever displacement calibration is referred to as the inverse optical lever sensitivity (InvOLS) [19]. It has been demonstrated that Sader’s method can be used to determine the stiffness of a cantilever, and the thermal noise method can then be used to determine
the InvOLS using the known stiffness [20]. The new heavy fluid loading technique presented in Chapter 5 can be used in a similar fashion to extend both noncontact force and displacement calibration to liquid environments. This could be advantageous both in the development of lab-on-a-chip devices and in research situations where soft cantilever or surface coatings inhibit the ability to perform static force InvOLS calibrations or where there is concern that the static deflection process might damage the cantilever tip [20].

Noncontact stiffness and displacement calibrations of AFM cantilevers (conducted in Chapter 5) result in a stacking of uncertainty. The stacking of uncertainty encountered by using two different AFM calibration techniques increases the need to understand the accuracy of both of them, what the potential sources of error are, and how to minimize them. The majority of the exploration into cantilever calibration techniques has been validated through the comparison of one technique to another. When the earliest calibration methods were developed, there was no available technique capable of measuring the small forces involved in a way that would be traceable to the International System of Units (SI). Without an SI-traceable measurement, the accuracy of force measurements performed through AFM is unknown. In recent years, the National Institute of Standards and Technology (NIST) of the United States Department of Commerce has built an electrostatic force balance (EFB) capable of performing SI-traceable calibrations [21].

While SI-traceable calibrations are currently unavailable to most researchers, NIST has published a set of SI-traceable calibrations along with calibrations performed using two more accessible methods [1]. The NIST study calibrated six cantilevers ranging in stiffness from 0.6 to over 50 N/m using four different calibration techniques and using the EFB. Currently, two of the most popular cantilever calibration methods are the thermal noise method [16] and Sader's method [17]. The first
method is based on equipartition theory, which expresses the time-averaged energy in the system in terms of temperature while the second incorporates a model of the fluid dynamics and is based on identifying features of the free vibration dynamics. Over the set of six cantilevers, Sader’s method agreed to within 12 % of the EFB measurement in all cases. The errors associated with the thermal noise method were higher, with a maximum error of 26 %, however, the cases where the error was the greatest corresponded to the cantilevers with the highest stiffness. These were noted to have poor signal-to-noise ratios, which likely contributed to the error. In most cases, researchers find that calibrations performed using Sader’s method and the thermal noise method agree to within about 20 % if both calibrations are performed in air [12, 1, 22].

In addition to the differences encountered using different calibration methods in air, there can be variation between calibrations performed on the same cantilever using the thermal noise method with data collected in both air and water [23, 6]. There should be no dramatic change in cantilever stiffness caused by changing the fluid environment from air to water. Changes in the resonant frequency correlating to a change in the surface stress on a cantilever have been observed [24], however, the changes are small compared to the frequency shift caused by the mass loading effect that a liquid such as water has on a beam, and the mechanism driving these changes is still debated [25]. Some of the reported differences are likely related from the signal-to-noise ratio issues mentioned by Langlois et al. [1] which are generally worse for thermal noise measurements conducted in liquid environments. This issue is addressed in Chapter 4 along with a strategy to reduce the difference between thermal noise calibrations conducted in air and in water.

There is also potential for AFM calibration errors to stem from irregularities in the AFM cantilever shapes. The photolithographic processes used to manufacture AFM
cantilevers can result in residual stresses that induce warping or bending with can change their mechanical behavior. The dynamics of warped cantilevers are explored in Chapter 8. Also, the theoretical foundations of some of the methods used in this research are applicable only to rectangular cantilevers. Since researchers frequently use V-shaped cantilevers, a specific effort is made to explore what improvements can be applied to the calibration of V-shaped cantilevers and what is needed to generalize the techniques used in this research to arbitrary cantilever shapes in Chapter 7.

1.2 Research Objectives

Objective 1: Identify a calibration technique for microcantilevers that works under heavy fluid loading.

The primary objective of this research effort has been to develop a technique to either modify Sader’s calibration method for use in heavy fluid loading situations or to develop a novel technique for such situations. In developing this technique, consideration was given to what data is available to a user. For example, while the physical dimensions of a cantilever can be measured, only the plan view dimensions can be known with reasonable accuracy by the tolerances of the manufacturing process. Slight changes to the thickness of a cantilever yields a dramatic change in cantilever stiffness. The variation in cantilever thicknesses drives the large specification range in commercially available cantilevers, therefore, the thickness of a cantilever was considered to be unavailable in the search for a new technique.

One desirable aspect of Sader’s method is that it is not impacted by the magnitude of measured cantilever data, only its dynamic characteristics such as the quality factor of the first bending mode and the resonance frequency. This is an important characteristic which enables the possibility of noncontact stiffness and displacement calibration when used in conjunction with the thermal noise method. Additionally,
a calibration technique that is not based on the magnitude of the thermal vibration data is not impacted by errors associated with the calibration of the cantilever displacement sensor.

**Objective 2: Characterize the performance of the new calibration technique.**

The ideal performance metric for a new AFM calibration technique would be a comparison to an SI-traceable calibration. This can be done at the NIST Small Force Metrology Laboratory; however, collaboration was not possible in the time frame of this research project. In the absence of an ideal performance metric, the methods developed in this research effort were characterized through comparisons between methods. The comparison of new calibration methods to existing methods has been the standard approach for characterization [6, 26]. Comparisons between methods that are not SI-traceable are limited in their ability to assess accuracy. However, when evaluating new techniques that expand on the conditions where calibration is possible, comparisons to existing techniques provide a valuable indicator of validity.

**Objective 3: Evaluate the potential for adapting the new technique to V-shaped cantilevers.**

A secondary objective of this research effort is to evaluate the potential to apply the advances achieved in rectangular cantilever calibration to V-shaped cantilevers. V-shaped cantilevers were originally developed as an alternative to rectangular cantilevers in an effort to maintain a low normal spring stiffness while increasing torsional stiffness. While it has since been shown that V-shaped cantilevers actually are more susceptible to torsion rather than less [27], V-shaped cantilevers have been found to be useful in AFM research.

The new calibration technique makes use of a theoretical model that describes
the mass loading and damping that a rectangular cantilever encounters in a fluid environment. Since many researchers use V-shaped cantilevers, the possibility of using finite element method (FEM) in combination with a computational fluid dynamics (CFD) model to generate a similar model for arbitrary geometries is explored. Additionally, techniques to improve the results of the thermal noise method through the use of alternate noise and system dynamics models are used to calibrate V-shaped cantilevers in both air and in water.

**Objective 4: Investigate the impact of strain induced warping and twisting of cantilevers on calibration techniques.**

The methods used to manufacture AFM cantilevers can result in residual strain causing cantilever warping. It is difficult for a researcher to characterize probes in a way that would identify any warping present. In the case of a cantilever used as part of a lab-on-a-chip, the necessary measurement may not be available. For this reason, the impact of cantilever warping on the static and dynamic properties that impact calibration are explored to identify under what circumstances warping might impact force measurements.

**1.3 Research Impact**

Enabling noncontact stiffness and displacement of AFM cantilevers in heavy fluid loading environments could have a far-reaching impact since it would expand the capabilities of an already versatile tool. AFM is an increasingly central technique to the field of nanobiotechnology capable of both probing and manipulating at the nanoscale. AFM can be used to measure several mechanical properties including geometry, static and dynamic compliance, friction, and interaction forces. Additionally, specialized AFM probes can perform chemical, electrical, and magnetic mea-
urements. AFM probes can even be modified through the attachment of specific biomolecules, enabling lines of research into individual biomolecular interactions and the mapping of the binding sites on biological samples.

The impact of an advance in AFM measurement capability is multiplied by the creativity and ingenuity of the biomolecular research community. The variety of AFM probes and techniques developed in the relatively short history of this sensor have enabled a wide range of novel observations. Single-molecule force spectroscopy (SMFS) can reveal the bond length, the magnitude of the energy barrier, and the off rate of a biomolecular bond [28]. The difference in the compliance of cancerous and normal cells can be detected through AFM, even when the cells are of similar shapes [29]. AFM topology scans can reveal clues to the functions of biomolecular mechanisms detailed enough to create molecular models [30]. The list of applications of the AFM to biological research is extensive and reviews are available [31, 32, 33]. Expanding the flexibility of the AFM by refining existing calibration methods and introducing new methods enables further growth in the variety of experiments that are possible with this tool.

1.4 Organization

The goal of performing noncontact AFM calibrations under heavy fluid loading involves both the development of new techniques and improvements on existing techniques. Chapters 2 through 5 are dedicated to the state of the art in AFM calibration and the advances of this research effort that are required for noncontact AFM calibration in liquid environments. The remaining chapters focus on issues that arise when dealing with cantilever geometries other than rectangular and flat.

Chapter 2 describes the state of the art in AFM stiffness calibration techniques. Special focus is placed on Sader’s method and the thermal noise method, which are
popular calibration methods, central to this research effort.

Chapter 3 discusses the models used to describe the hydrodynamic loading of AFM cantilevers along with the application of the fluctuation-dissipation theorem to describe the dynamics of the free cantilever thermal vibration.

Chapter 4 uses the models discussed in Chapter 3 along with various noise models to perform stiffness calibrations according to the thermal noise method. The models used in commercial systems are compared to alternate models that result in a reduction in the difference between calibrations performed in air and in water.

Chapter 5 describes a new AFM stiffness calibration method that incorporates a heavy fluid loading assumption. This method is then used in conjunction with the techniques developed in Chapter 4 to perform noncontact stiffness and displacement calibration under heavy fluid loading.

Chapter 6 describes FEM and CFD simulations that were conducted in order to explore the system dynamics of warped and V-shaped cantilevers.

Chapter 8 discusses the impact of warping on cantilever stiffness calibration. The methods of Chapter 6 are used to determine how warping changes the system dynamics.

Chapter 7 discusses the application of the methods of Chapters 4 and 6 to V-shaped cantilevers.

Chapter 9 discusses the conclusions reached through this research and potential avenues for further investigation.
Chapter 2

Existing Techniques

The stiffness calibration of AFM cantilevers has been the subject of an appreciable volume of academic research with a significant number of cantilever calibration techniques published [5, 6, 7, 8, 9, 10, 1, 11, 12, 13, 14]. Two methods have emerged as the most popular: the thermal noise method [16], which is based on the equipartition theorem, and Sader’s method [17], which is based on characteristics of the dynamics of thermal noise. Additionally, more recent work performed at NIST has focused on the pursuit of AFM calibration methods traceable to SI measurements [3, 34, 1]. Surveys of cantilever calibration methods that include descriptions of the accuracy generally claim $\pm 10\%$ accuracy, with some methods claiming $\pm 20, 30, \text{or even } 50\%$ [8, 2]. However, since most of these research efforts predate or did not have access to the SI-traceable methods available at NIST, these claims cannot be accepted authoritatively.

2.1 The Thermal Noise Method

One of the important, early developments with respect to the calibration of micro-cantilevers was Hutter and Bechhoefer’s [16] observation that the law of equipartition relates the cantilever stiffness to the thermally driven motion of the tip and the ambient temperature. The time-averaged potential energy stored in a cantilever beam at thermal equilibrium is described in the following equation:

$$\frac{1}{2}k_B T = \frac{1}{2}k\langle q^2 \rangle,$$

(2.1)
where $k_B$ is Boltzmann’s constant, $T$ is the absolute temperature, $k$ is the stiffness of the cantilever at the tip, and $\langle q^2 \rangle$ is the mean squared displacement of the end of the cantilever [16]. In addition to serving as the key to the thermal noise method, the law of equipartition is used to scale various models of the thermal response of cantilever-fluid systems as described in Chapter 3.

In the absence of other sources of noise, this relationship reduces calibration to a simple matter of measuring the temperature and the mean squared tip displacement. Because it does not rely on assumptions regarding the geometry of the cantilever or the properties of the fluid environment, the thermal noise method is an appealing technique. However, there are several factors that must be taken into account such as the distribution of energy between modes, noise sources present in the measurement system, and scaling factors that arise from the common practice of measuring cantilever deflection by the optical lever technique, which detects changes in cantilever angle rather than position. One result of the various factors that impact the thermal noise method is that calibrations performed in different fluid environments result in stiffness values that can vary significantly [6, 35]. A discussion of several known factors is included in Appendix A.

### 2.2 Sader’s Method

In 1998, Sader [36] formulated an expression for the mass loading and damping that a cantilever encounters when vibrating in a fluid and solved for the frequency response of a cantilever beam driven by stochastic excitation. The fluid loading is a function of the Reynolds numbers, which is a function of the density and the viscosity of the fluid, the width of the cantilever beam, and the oscillation frequency as discussed in Chapter 3. Sader also observed that through the appropriate frequency substitution ($\omega = \omega_r$), the system dynamics took the form of a simple harmonic oscillator (SHO).
While this substitution is only valid over a small frequency range, in cases where
damping is light (i.e., the quality factor, \( Q \gg 1 \)) it is valid over a sufficient portion
of the resonance peak.

Using the SHO approximation and the hydrodynamic function from [36], in 1999,
Sader published a calibration technique that estimates the stiffness as [17]:

\[
k = 0.1906 \rho_f b^2 l Q \Gamma_i(\omega_r) \omega_r^2.
\] (2.2)

Here, \( b \) is the cantilever width, \( l \) is the cantilever length, \( \rho_f \) is the fluid density,
\( \omega_r \) is the resonance frequency, and \( \Gamma_i \) is the imaginary part of the hydrodynamic loading
function on the beam evaluated at the resonance frequency. The light damping
assumption required to simplify the dynamics to those of an SHO limits its use in
most cases to cantilevers in gas environments.

It is important to note that Sader’s method is not impacted by the magnitude of
the data. Errors associated with the voltage-to-displacement calibration of an AFM
system do not affect stiffness calibrations performed via Sader’s method as they
would thermal noise method calibrations. However, the use of the hydrodynamic
function limits Sader’s method to rectangular cantilevers and the high quality factor
assumption generally restricts the use of this method to cantilevers in air.

### 2.3 Other Techniques

The earliest estimations of cantilever stiffness required knowledge of the geometry
and Young’s modulus, \( E \) [37]. The bending stiffness of a cantilever beam is:

\[
k = \frac{Et^3b}{4l^3},
\] (2.3)

where \( t \) is the thickness of a rectangular beam. While the simplicity of this approach
is attractive, the thickness tolerances of the photolithographic processes required to
manufacture AFM cantilevers result in a high degree of uncertainty in the stiffness. Equation 2.3 reveals that the stiffness varies with the thickness cubed. Additionally, photolithographic techniques often produce layers of materials with Young’s moduli that vary significantly from their bulk material properties.

Calibration has been performed by monitoring the change in resonance frequency while attaching beads of various masses to a cantilever [15]. Variations on this method include monitoring the change in resonance frequency before and after gold coating a cantilever [38] and while monitoring the evaporation of a drop of water placed on a beam [39].

Some nanoindentation devices contain capacitive sensors with adequate sensitivity to calibrate some of the less compliant AFM cantilevers. Cantilevers with stiffnesses in excess of 1 N/m can be calibrated by nanoindentation sensors with an accuracy of ±10 % [5]. Through the use of an integrated optical microscope, precision stages, and proper calibration of all the system components, it is possible to calibrate cantilevers with stiffnesses as low as 0.1 N/m with an accuracy of ±10 % [13].

It is also possible to calibrate a cantilever by pushing against a reference spring, often another cantilever which has been calibrated by other means [40, 41] such as the added mass method [42]. The added mass method has also been used to determine the bending stiffness of glass rods, which can then be used as reference cantilevers [43, 44]. In 2007, Aksu et al. used a nanoindenter to calibrate a piezoresistive cantilever which enabled a simpler measurement of the reference cantilever deflection [8]. In addition to reference cantilevers, microstructure devices specifically designed to serve as reference springs have been employed [2].

While the majority of the available cantilever calibration methods involve some combination of applying known forces, obtaining measurements of the cantilever geometry, or analyzing characteristics of the dynamics of the cantilever-fluid system,
some techniques go to greater lengths to obtain results. In 2007, Lubarsky and Hähner calibrated cantilevers by building a fluid cell that allowed for fluid flow along the length of a cantilever beam [11]. By changing the flow rate, the pressure gradient on one side of the cantilever changed which caused a shift in the resonance frequency from which, the stiffness could be determined. Maeda built 500:1 scale models of cantilevers and measured the deflections induced by fluid flows at various speeds to create functions relating calculated Stoke’s drag to the equivalent point force that would yield the same deflection for a specific cantilever geometry [45]. This curve was then used to calculate stiffness by observing the deflection of an AFM cantilever as it was moved through a liquid environment. While approaches such as these can be impractical in situations where a large number of cantilevers require calibration, it is valuable to identify different methods for acquiring dynamic response data to various inputs. Data of this nature can be particularly valuable in the creation of empirical models.
Before 2003, the smallest SI-traceable mass artifacts available at NIST were too large to be used in AFM calibration. Therefore, a five-year project was launched with the stated goal of developing an instrument capable of producing and measuring SI-traceable forces less than $5 \mu N$ to within a few parts in $10^4$ [21]. The EFB was built to achieve this goal [3]. A schematic of the NIST EFB is presented in Figure 2.2 and a detailed description of the applicable metrology hierarchy appears in [34]. The NIST EFB generates force through a set of nested cylinders that form a capacitor. Relative axial motion between the cylinders changes the condition of the capacitor such that the applied force is a function of the voltage between the cylinders and the capacitance. The outer cylinder position is fixed and the inner cylinder is suspended by a set of flexures. An interferometer is used to detect relative motion between the two cylinders and a control algorithm is used to modulate the voltage to maintain a null position. In this way a balance is established between the applied force and the applied voltage.
By 2004, the EFB had measured the gravitational force of proof masses at NIST smaller than 10^{-5} N with sufficient accuracy to demonstrate the calibration of cantilevers that could then be used as reference artifacts [3]. Difficulties in the positioning of the cantilever-on-reference cantilever contact point limits the estimated accuracy of calibration against a single cantilever to between ±10 % to ±30 % [41, 9] To improve on this, both the National Physical Laboratory (NPL) and NIST have developed microscale spring arrays to be used as artifacts [2, 46]. Additionally, both NIST and NPL produced reference cantilevers with piezoresistive elements to provide an alternate measurement of the deflection of the reference [1, 47].

Langlois et al. calibrated a set of seven cantilevers using the EFB, a NIST piezosensor, a nanoindenter, the thermal noise method, and Sader’s method [1]. The results, as percent differences from the stiffnesses measured by the EFB, are presented in Table 2.1. Sader’s method performed better for the less compliant cantilevers (±5.1 %) than for the more compliant cantilevers (±12.0 %). It was the reverse for the thermal noise method; in the cases of the least compliant cantilevers (six and seven), the poor signal-to-noise ratios were believed to contribute to the larger degree of overestimation. These measurements demonstrate that the state of the art in AFM cantilever calibration is subject to significant uncertainty under the best of circumstances, even for calibrations performed in air.
Table 2.1: Cantilever calibrations performed using the NIST EFB and the difference between calibrations performed by the EFB and four other methods expressed as a percentage of the EFB calibration value (NIST piezosensor, $k_{\text{piezo}}$, thermal noise method, $k_{\text{therm}}$, Sader’s method, $k_{\text{Sader}}$, and nanoindenter, $k_{\text{NI}}$.) Cantilever five was damaged before the EFB calibration could be performed and is therefore omitted. Original data presented in [1].

<table>
<thead>
<tr>
<th>Cantilever No.</th>
<th>$k_{\text{EFB}}$ (N/m)</th>
<th>$\Delta k_{\text{piezo}}$</th>
<th>$\Delta k_{\text{therm}}$</th>
<th>$\Delta k_{\text{Sader}}$</th>
<th>$\Delta k_{\text{NI}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0634±0.0007</td>
<td>5.2 %</td>
<td>2.8 %</td>
<td>9.5 %</td>
<td>N/A</td>
</tr>
<tr>
<td>2</td>
<td>3.68±0.01</td>
<td>22.3 %</td>
<td>15.2 %</td>
<td>12.0 %</td>
<td>N/A</td>
</tr>
<tr>
<td>3</td>
<td>4.38±0.02</td>
<td>11.0 %</td>
<td>9.8 %</td>
<td>-3.2 %</td>
<td>N/A</td>
</tr>
<tr>
<td>4</td>
<td>11.2±0.3</td>
<td>8.9 %</td>
<td>-17.9 %</td>
<td>2.7 %</td>
<td>4.8 %</td>
</tr>
<tr>
<td>6</td>
<td>49.2±0.5</td>
<td>15.7 %</td>
<td>25.8 %</td>
<td>-2.8 %</td>
<td>-17.9 %</td>
</tr>
<tr>
<td>7</td>
<td>53.4±1.0</td>
<td>12.0 %</td>
<td>21.2 %</td>
<td>-5.1 %</td>
<td>1.0 %</td>
</tr>
</tbody>
</table>
Chapter 3

Cantilever-Fluid System Modeling

As discussed in Chapter 2, while some AFM stiffness calibrations involve force and displacement measurements, others involve the measurement of other cantilever features that are used to solve for the cantilever stiffness through relationships established in models of dynamic cantilever behavior. Since measuring the power spectral density (PSD) of the random thermal vibration of a free cantilever is a relatively easy measurement to make, several calibration techniques involve models of the PSDs of a cantilever-fluid system. In this chapter, the most common models used to describe the dynamic behavior of a vibrating cantilever are presented.

3.1 The Simple Harmonic Oscillator

An idealized mass-spring system, if in motion and not subjected to external forces, will oscillate at a fixed frequency and is therefore known as a simple harmonic oscillator (SHO). Adding an idealized dashpot to the model will result in damped harmonic motion, however, the system is still commonly referred to as an SHO. In terms of mass, $m$, damping, $c$, stiffness, $k$, and displacement, $w$, the equation of motion is

$$m\ddot{w} + c\dot{w} + kw = 0,$$  \hspace{1cm} (3.1)

which is

$$\dot{w} + \frac{\omega_n}{Q}\dot{w} + \omega_n^2 w = 0,$$ \hspace{1cm} (3.2)
in terms of natural frequency, \( \omega_n \), and quality factor, \( Q \). The PSD of an SHO as a function of circular frequency, \( \omega \), is described by:

\[
S_{\text{SHO}}(\omega) = P_{\text{DC}} \frac{\omega_n^4}{(\omega_n^2 - \omega^2)^2 + \left( \frac{\omega \omega_n}{Q} \right)^2},
\]

where \( P_{\text{DC}} \) is equal to the DC power response of the SHO model (i.e., the response at 0 Hz, or in electrical terms, direct current.) An SHO model can provide a good estimation of the dynamic behavior of a cantilever provided the fluid loading is small (i.e., \( Q \gg 1 \)) [48]. However, for cantilever-fluid systems that exhibit a more highly-damped resonance, an SHO model does not capture the system dynamics as well.

### 3.2 Lorentzian Resonators

The Lorentzian line function arose from the study of the spectral distribution associated with a classical oscillator with a radiative damping mechanism [49]. In Lorentz’s case the oscillator was an electron and the radiation was optical; however, this is also a good description of the damping mechanism induced by a thermal bath. In [50], an SHO model is modified so that it takes the form of a Lorentzian function, expressed in terms of amplitude, natural frequency, and quality factor. (The modification involves invoking the approximation \( \omega = \omega_n \) in cases where the terms are multiplied and dividing the numerator and denominator by \( \omega_n^2 \).) The PSD of the Lorentzian expressed in this way is:

\[
S_{\text{Lor}}(\omega) = P_{\text{DC}} \frac{\omega_n^2}{4 (\omega_n^2 - \omega^2)^2 + \left( \frac{\omega_n}{Q} \right)^2},
\]

(3.4)
3.3 Euler-Bernoulli Beam Theory

While the SHO and Lorentzian models serve as useful approximations of cantilever-fluid system dynamics in certain circumstances, they are not based on any details specific to an AFM probe. A more detailed model can be achieved by incorporating some description of the fluid loading with a formulation of Euler-Bernoulli beam theory.

3.3.1 The Beam Equation

The mathematical description of transverse wave behavior in a beam (the beam equation) is well known as:

$$\mu \frac{\partial^2 w(x, t)}{\partial t^2} + EI \frac{\partial^4 w(x, t)}{\partial x^4} = f(x, t)$$  \hspace{1cm} (3.5)

where $w(x, t)$ is the transverse displacement of the beam at a position along its axis, $x$, at time, $t$. $E$ is the Young’s modulus of the beam material, $I$ is the second area moment of inertia of a cross section of the beam, and $\mu$ is the mass per unit length of the beam. The beam can be subject to the forcing function $f(x, t)$, however, to finding the mode shapes, requires solving the homogeneous partial differential equation:

$$\mu \frac{\partial^2 w(x, t)}{\partial t^2} + EI \frac{\partial^4 w(x, t)}{\partial x^4} = 0.$$  \hspace{1cm} (3.6)

Stating that there are mode shapes to be found implies that this equation is separable in space and time, that is:

$$w(x, t) = W(x)H(t).$$  \hspace{1cm} (3.7)

Substituting this into 3.6 and rearranging yields:

$$\frac{EI}{\mu W(x)} \frac{\partial^4 W(x)}{\partial x^4} = -\frac{1}{H(t)} \frac{\partial^2 H(t)}{\partial t^2}$$  \hspace{1cm} (3.8)
The left hand side of 3.8 is a function of position but not time while the right hand side of the equation is a function of time but not position. The variables have been separated, but the two sides are still equal for all $x$ and $t$. This can only be possible if each side is equal to a constant. It is appropriate, for reasons that will become clear, to label this constant $\omega^2$.

$$\frac{EI}{\mu W(x)} \frac{\partial^4 W(x)}{\partial x^4} = -\frac{1}{H(t)} \frac{\partial^2 H(t)}{\partial t^2} = \omega^2$$  \hspace{1cm} (3.9)

This results in two separate differential equations,

$$\frac{EI}{\mu W(x)} \frac{d^4 W(x)}{dx^4} = \omega^2$$  \hspace{1cm} (3.10)

and

$$-\frac{1}{H(t)} \frac{d^2 H(t)}{dt^2} = \omega^2.$$  \hspace{1cm} (3.11)

Rearranging the spatial equation yields a fourth-order homogeneous ordinary differential equation:

$$\frac{d^4 W(x)}{dx^4} - \frac{\mu \omega^2}{EI} W(x) = 0.$$  \hspace{1cm} (3.12)

The general solution to the equation is:

$$W(x) = A_1 \sin(\alpha x) + A_2 \cos(\alpha x) + A_3 \sinh(\alpha x) + A_4 \cosh(\alpha x).$$  \hspace{1cm} (3.13)

Recognizing that

$$\frac{d^4 W(x)}{dx^4} W(x) = \alpha^4,$$  \hspace{1cm} (3.14)

equation (3.10) can be rearranged to yield:

$$\omega = \alpha^2 \sqrt{\frac{EI}{\mu}}.$$  \hspace{1cm} (3.15)
The next step is to apply the boundary conditions. The base of a cantilever beam is fixed, so the position and slope are both equal to zero. This yields:

\[ W(0) = 0 \quad (3.16) \]

and

\[ \frac{dW(0)}{dx} = 0. \quad (3.17) \]

The free end of a cantilever, being a free surface, has no moment acting on it. There is therefore no bending and no change in bending at that point. Therefore,

\[ \frac{d^2W(l)}{dx^2} = 0 \quad (3.18) \]

and

\[ \frac{d^3W(l)}{dx^3} = 0. \quad (3.19) \]

Applying the boundary conditions to (3.13) and rearranging yields the characteristic equation:

\[ -1 = \cos(\alpha l) \cosh(\alpha l). \quad (3.20) \]

For a given length, l, there are an infinite number of values of \( \alpha \) that will solve this equation. The different values of \( \alpha_n \) (\( \alpha_1, \alpha_2, ... \)) will yield a different \( W_n(x) \) (\( W_1(x), W_2(x), ... \)). These \( W_n(x) \) equations are the mode shapes of the cantilever beam.

Additionally, applying the boundary conditions to (3.13) can yield:

\[ A_2 + A_4 = 0 \rightarrow A_4 = -A_2, \quad (3.21) \]

\[ \alpha(A_1 + A_3) = 0 \rightarrow A_3 = -A_1, \quad (3.22) \]

and

\[ A_1 = -A_2 \frac{\cos(\alpha_n l) + \cosh(\alpha_n l)}{\sin(\alpha_n l) + \sinh(\alpha_n l)}. \quad (3.23) \]
Finally, substituting (3.21), (3.22), and (3.23) into (3.13) eliminates $A_1$, $A_3$, and $A_4$, but not $A_2$. The values of $\alpha_n$ that satisfy the characteristic equation, (3.20), can be substituted into this equation to get the mode shapes and $A_2$ allows the mode shapes to be scaled. The beam shapes are described by:

$$W_n(x) = A_2 \left[ \cos(\alpha_n x) - \cosh(\alpha_n x) + \left[ \sinh(\alpha_n x) - \sin(\alpha_n x) \right] \right]$$

and (3.15) becomes:

$$\omega_n = \alpha_n^2 \sqrt{\frac{EI}{\mu}}.$$  

(3.24)

This describes the natural frequencies of the bending modes. The mode shapes are orthogonal, therefore:

$$\int_0^L W_n(x)W_m(x)dx = 0$$  

(3.26)

for all $n \neq m$. Normalizing the mode shapes by the mass, $A_2 = 1/\sqrt{t_\mu}$, yields

$$\ddot{H}(t) + \omega_n^2 H(t) = F_n(t).$$  

(3.27)

where $F_n(t)$ is the modal force. This equation describes the motion for an in vacuo cantilever beam and will appear in Section 3.5 as Equation 3.34.

### 3.3.2 Stokes’ Drag

An early effort to model the dynamics of a cantilever-fluid system involved adding mass and damping forces to the beam equation [51, 52]. The drag term included was that of Stokes’ law (i.e., $f = 6\pi a\eta \dot{x}$) for a sphere of arbitrary radius, $a$ in a fluid with viscosity $\eta$. The added fluid mass was equivalent to half of the mass of a sphere of radius, $a$. The value for $a$ was determined by fitting model parameters to measured cantilever vibrations in various fluids.
3.4 Sader’s Model

In 1998, Sader improved upon the model for the mass loading and damping induced by the fluid enveloping the cantilever [36]. To create this model, Sader turned to existing solutions to the Navier-Stokes equations for similar problems. The Navier-Stokes equations have not been solved for fluid flow around a rectangle [36], but they have been solved for a rigid cylinder oscillating in a direction perpendicular to the axis of the cylinder [53]. According to Rosenhead, [53], the force of a fluid with density, \( \rho_f \), on an infinitely long oscillating cylinder with radius, \( a \), with velocity \( \frac{d}{dt} w(x, t) = U_0 \cos(\omega t) = \text{Re} [U_0 \exp(i\omega t)] \) is expressed as a function of the Reynolds number, \( \text{Re} \);

\[
f_{\text{hydro}}(i\omega) = -m'U_0 i\omega \left[ 1 - \frac{4K_1(\sqrt{iRe})}{K_1(\sqrt{iRe}) + \sqrt{iRe}K_1'(\sqrt{iRe})} \right] \exp(i\omega t) \quad (3.28)
\]

where \( \text{Re} = \frac{\omega a^2}{\nu} \), \( \nu \) is the kinematic viscosity, and \( m' \) is the mass of the fluid displaced by a length, \( l \), of the cylinder given by

\[
m' = \pi \rho_f a^2 l \quad (3.29)
\]

and \( K_1 \) is a first order, modified Bessel function of the second kind. As noted by Sader [36] and Cole [54], the Reynolds number used in this loading expression is an alternate formulation to the commonly used nonlinear convective term in the Navier-Stokes equation.

This loading can be expressed for a segment of the beam, \( dx \), in separable terms:

\[
f_{\text{hydro-segment}}(i\omega) = \frac{m' \Gamma(i\omega)}{l} \omega^2 W(x) H(i\omega) dx, \quad (3.30)
\]

where \( W(x) \) is the function of position along the length of the beam that describes
the mode shape, \( H(i\omega) \) contains the mode dynamics and

\[
\Gamma(i\omega) = 1 - \frac{4K_1(\sqrt{i\Re})}{K_1(\sqrt{i\Re}) + \sqrt{i\Re}K'_1(\sqrt{i\Re})}.
\]

(3.31)

To improve on this approximation for the hydrodynamic loading on a beam, Sader employs a numerical solution for the hydrodynamic loading on a rigid, infinitely thin ribbon formulated by Tuck [36, 55]. Sader compares the solutions of Rosenhead and Tuck and gives a polynomial correction function, \( \Omega(\omega) \), that satisfies:

\[
\Gamma_{\text{ribbon}}(i\omega) = \Omega(\omega)\Gamma(i\omega).
\]

(3.32)

Substituting \( \Gamma_{\text{ribbon}} \) for \( \Gamma \) provides a more detailed model of the hydrodynamic forces on a rectangular beam provided the beam is sufficiently thin relative to its width. This improved model for hydrodynamic cantilever loading led to one of the key parameter-based calibration techniques discussed in Chapter 2.

### 3.5 Fluid-Structure Interaction and the Fluctuation-Dissipation Theorem

The modeled thermal response of the cantilever has been further refined by applying the fluctuation-dissipation theorem to the fluid loaded cantilever model [54]. Previous models implicitly treated thermal excitation as white noise [36, 20]. In 2004, Paul et al. modeled the thermal excitation of a microcantilever applying the results of the fluctuation-dissipation theorem to a fluid-loaded cantilever model [56]. The development of the fluctuation-dissipation theorem dates back to 1905, when Einstein theorized that Brownian motion and the drag on a particle forced to move through a fluid arose from the same mechanism [57]. This implies that thermal noise of a particle or structure is not necessarily white, rather it is related to the motility of that
particle or structure. This adjustment to the thermal excitation has been used in a few studies [58, 56, 54]. One interesting result of the application of the fluctuation-dissipation theorem to a microcantilever is that the displacement thermal response has zero magnitude at 0 Hz. While this result conflicts with other models of cantilever vibration which yield non-zero values at 0 Hz, this is in agreement with the expected thermal response of a microcantilever. A microcantilever that is not subject to a static load will vibrate about a point of zero deflection. The SHO and Sader models, as commonly used, contain non-zero values at 0 Hz stemming from white noise excitation assumptions. However, a non-zero thermal response at 0 Hz indicates a constant deflection; implying that the thermal excitation preferentially drives the cantilever either up or down some amount. Since this is clearly not the case, the thermal response at 0 Hz should be zero as the fluctuation-dissipation theorem predicts.

Cole [54] incorporates the fluctuation-dissipation theorem in a fluid-structure interaction (FSI) formulation of the system and treats the hydrodynamic forces as a feedback mechanism. In Cole’s model, the thermally-driven, random modal force, $\tilde{f}_n$, is normalized by the modal stiffness, $k_n$, to produce a fluctuating input displacement, $\tilde{q}_n$:

$$\tilde{q}_n = \frac{\tilde{f}_n}{k_n}. \quad (3.33)$$

The equation of motion for the in vacuo cantilever in terms of its mass, $m$, and its modal displacement $q_n$, is then:

$$m\ddot{q}_n + k_n q_n = \tilde{f}_n = k_n \tilde{q}_n. \quad (3.34)$$

The damping intrinsic to the beam is assumed to be trivial relative to the damping induced by the surrounding fluid. The resulting transfer function between input and
output displacements for the \textit{in vacuo} cantilever is:

\[
\frac{q_n}{\tilde{q}_n} = G(s) = \frac{\omega_n^2}{s^2 + \omega_n^2}.
\]  

(3.35)

The hydrodynamic loading on the cantilever is a response to cantilever motion; it is therefore feedback. Sader’s hydrodynamic function describes this feedback and is considered in the following controller, \( K \), which is the ratio between cantilever deflection, \( q_n \), and a fluid reaction displacement, which is the fluid reaction force normalized by the model stiffness, \( k_n \):

\[
K(s) = \gamma \frac{s^2}{\omega_n^2} \Gamma(s),
\]  

(3.36)

where \( \gamma \) is a mass ratio between the cantilever and the surrounding fluid:

\[
\gamma = \frac{m'}{m},
\]  

(3.37)

and the surrounding fluid mass for a cylinder enveloping a beam (radius \( a = b/2 \)) is:

\[
m' = \pi a^2 l \rho_f.
\]  

(3.38)

(In actuality, \( \Gamma \), as used here, is the complex conjugate of \( \Gamma \) as used in [36]; the difference stems from the different notations in [36] and [53].) The closed-loop system, \( P \), which includes both the \textit{in vacuo} response and the hydrodynamic feedback is:

\[
P(s) = \frac{q_n(s)}{\tilde{q}_n(s)} = \frac{\omega_n^2}{s^2 + \omega_n^2 + \gamma s^2 \Gamma(s)}.
\]  

(3.39)

Finally, applying the fluctuation-dissipation theorem yields the modal PSD of the thermal response:

\[
S_{qq,n}(\omega) = -\frac{2k_B T \text{Im}[P(i\omega)]}{k_n \omega}.
\]  

(3.40)
The sum of the modal PSDs scaled by the modal displacements yield the total dynamic response of the beam:

\[ S_{qq}(\omega) = \sum W_n^2(l) S_{qq,n}(\omega). \]  

(3.41)

This approach allows for the cantilever response to be modeled based on the cantilever length and width, the properties of the fluid environment, and two additional terms; the natural frequency, of the cantilever, \( \omega_n \), and the fluid-to-cantilever mass ratio, \( \gamma \). The cantilever stiffness can be determined if these terms are known.

### 3.6 Summary

A selection of models used to approximate AFM cantilever dynamics has been presented. In Chapter 4, the SHO, Lorentzian, and FSI models are used to perform thermal noise calibrations and the resulting stiffness estimations are compared. In Chapter 5, the FSI model is used to estimate cantilever-fluid system parameters in a new calibration technique. The new technique is designed for cantilevers under heavy fluid loading conditions such as those present in liquid environments.
Chapter 4

Modeling System Dynamics and Noise Sources

The thermal noise method of performing AFM cantilever stiffness calibrations is one of the most popular techniques used. A 2007 NIST study involved the calibration of six cantilevers ranging in stiffness from 0.6 to over 50 N/m using various methods including the thermal noise method and using the EFB, which is an SI-traceable method with little uncertainty [1]. Calibrations performed using the thermal noise method of Hutter and Bechhoefer varied from the EFB calibrations over a range of -17.9% to +25.8%. The cases where the error was the greatest corresponded to the cantilevers with the highest stiffness which were noted to have poor signal-to-noise ratios, which likely contributed to the error. Because of the popularity of the thermal noise calibration method, it is of significant importance that the AFM community be aware of the sources of error associated with its use and that continued emphasis be placed on the goal of refining this and other cantilever calibration techniques.

The thermal noise method is based on the equipartition theorem, which expresses the energy available to particles or structures excited by a thermal bath, at equilibrium, as a function of temperature [16]. Using this relation, a researcher can, in theory, determine the calibration constant of a cantilever with only a temperature measurement and a measurement of the time-averaged displacement. While the equipartition theory underlying the thermal noise method is unchallenged in its applicability to AFM cantilever calibration, various factors must be considered to apply it appropriately. Some of these factors are already well understood, such as the correction factor required to account for the difference in beam shape between...
a dynamically oscillating beam and a statically deflected beam [59]. This correction factor along with others are discussed in more detail in Appendix A.

One potential source of error in the time-averaged displacement measurement stems from ambient noise sources in the environment. Mechanical vibrations, acoustic disturbances, and electrical noise can impact cantilever displacement measurements and could be misinterpreted as thermal vibration. While the influence of these noise sources cannot necessarily be eliminated, their impact can be assessed by transforming the cantilever displacement data from the time domain to the frequency domain. After transforming, a frequency range can be selected where the first bending mode of the cantilever dominates the signal. Then, a model of the cantilever-fluid system dynamics along with other ambient noise can be curve fit to the selected data and an approximation of the vibration of the first bending mode in the absence of external sources of noise can be determined.

Commonly, an SHO model is used to approximate the thermal noise. Under light fluid loading conditions, AFM cantilever resonances exhibit high quality factors and SHO models provide excellent curve fits. However, under heavy fluid loading, errors can arise from the differences between the shape of an SHO model and the dynamics of the cantilever-fluid system. More recently, Pirzer and Hugel investigated the effect of using a Lorentzian rather than an SHO model to describe dynamic cantilever behavior in heavy fluid loading environments [50]. In that study, Pirzer and Hugel compare the fitting techniques used in commercial AFM systems (including SHO and Lorentzian models) to other fitting functions and integration methods. In heavy fluid loading environments calibrations relying on SHO model fits vary significantly from calibrations made using a Lorentzian model.

As discussed in Chapter 3, a more detailed model of the thermal response of a rectangular beam has been developed that includes the application of the hydrodynamic
forces that would act on an oscillation ribbon. These forces are applied to the beam equation to create a dynamic model and the excitation of the model as determined by the fluctuation-dissipation theorem [36, 54]. This fluid-structure interaction (FSI) model is used in this chapter for comparison to the SHO and Lorentzian models.

The differences between these models result in different approximations for the random vibration of a cantilever in the absence of noise. The more detailed FSI model is based on its own set of assumptions that make it an imperfect model of cantilever-fluid dynamics (e.g., the fluid motion is restricted to only two dimensions; motion along the length or around the end of the beam is not considered [54]). Therefore, the resulting differences between direct comparisons of the SHO or the Lorentzian and FSI models can only serve to provide an approximation of the error associated with using an SHO model in conjunction with the thermal noise calibration method. However, such comparisons can serve to reveal under what conditions this source of error is more significant and in what situations researchers should be concerned with this source of error.

In addition to errors stemming from the use of various models of the cantilever dynamics, error can also occur when the modeled system noise does not adequately match the dynamics of the actual system noise. System noise is commonly modeled as white noise; however, electronics often introduce noise that varies as a function of frequency. In this chapter, the SHO and Lorentzian models are fit to simulated data created using the FSI model to explore the differences between models under idealized conditions. Additionally, various combinations of noise and dynamics models are used to perform thermal noise calibrations on measured cantilever responses collected in air and in water to investigate the impact of the ambient noise present in a real system.
4.1 Theory

4.1.1 The Thermal Vibration Calibration Method

As presented by Hutter and Bechhoefer [16], the equipartition theorem dictates that the time-averaged potential energy stored in a cantilever beam at thermal equilibrium is

\[ \frac{1}{2} k \langle q^2 \rangle = \frac{1}{2} k_B T \]  

(4.1)

where \( k \) is the spring constant of the cantilever, \( q \) is the out-of-plane cantilever displacement at the end of the beam, \( T \) is the fluid temperature, and \( k_B \) is the Boltzmann constant [16].

4.1.2 Cantilever-Fluid Dynamics Models

The three models that are used to approximate the dynamics of a cantilever-fluid system, the SHO, the Lorentzian, and the FSI model are discussed in Chapter 3. The PSD of an SHO is described by:

\[ S_{\text{SHO}}(\omega) = P_{\text{DC}} \frac{\omega_n^4}{(\omega_n^2 - \omega^2)^2 + \left( \frac{\omega \omega_n}{Q} \right)^2} \]  

(4.2)

The parameters \( P_{\text{DC}}, \omega_n, \) and \( Q \) must be adjusted to fit the model to the measured data. The scaling term, \( P_{\text{DC}} \), is equal to the DC power response of the SHO model. An SHO model fit can provide a good estimation of the cantilever response provided the fluid loading is small (i.e., \( Q \gg 1 \)) [48]. However, for cantilever-fluid systems that exhibit a more damped resonance, an SHO model does not capture the system dynamics as well.

The Lorentzian line function is expressed as presented by Pirzer et al. [50] so that it can be expressed using the same fitting terms as are used in the SHO model. This
The function is expressed as:

\[ S_{\text{Lor}}(\omega) = P_{DC} \frac{\omega_n^2}{4(\omega_n - \omega)^2 + \left(\frac{\omega_n}{Q}\right)^2}. \]  

(4.3)

The PSD of the thermal response of the FSI models is:

\[ S_{qq}(\omega) = -\frac{k_B T \text{Im}[P(i\omega)]}{m\omega_n^2 \omega}. \]  

(4.4)

Where \( P(s) \) is the transfer function describing the closed-loop cantilever-fluid system as described in Chapter 3 and is given by:

\[ P(s) = \frac{q_n(s)}{\tilde{q}_n(s)} = \frac{\omega_n^2}{s^2 + \omega_n^2 + \gamma s^2 \Gamma(s)}. \]  

(4.5)

The fitting parameters of the FSI model are the natural frequency, \( \omega_n \) and the mass ratio, \( \gamma \). The magnitude of the FSI model is determined by the equipartition theorem, and with this restriction, the FSI model is extremely sensitive to the magnitude of the data. However, under heavy fluid loading, it is robust to perturbations in \( \omega_n \) and \( \gamma \). Therefore, an additional scaling parameter, \( C \), was added which made the FSI model:

\[ S_{qq}(\omega) = -CK_B T \frac{\text{Im}[P(i\omega)]}{m\omega_n^2}. \]  

(4.6)

The scaling parameter, \( C \), is required to accommodate any errors in the magnitude of measured data (which could arise calibrating a photodiode, for example) [48, 35]. By adding the scaling term, the curve fitting algorithm fits only the shape of a PSD, allowing the resulting magnitude to be used to find the cantilever stiffness according to the equipartition theorem.
4.1.3 Noise Models

In addition to cantilever dynamics in the absence of noise, a variety of noise conditions were considered. The PSD of a cantilever dynamics model, $S_{\text{cant}}(\omega)$, in the presence of white noise, $P_w$, is:

$$S_{yy}(\omega) = P_w + S_{\text{cant}}(\omega).$$

(4.7)

The term ‘$1/f$ noise’ is commonly used to describe noise sources that are of the form $1/\omega^\beta$. Pink noise is an example of $1/f$ noise and is a specific case where $\beta = 1$. A system with pink noise, $P_p$, is given by:

$$S_{yy}(\omega) = \frac{P_p}{\omega} + S_{\text{cant}}(\omega).$$

(4.8)

A lot of the noise observed in this study, appeared to fall somewhere between white noise and pink noise, and so the case where $\beta = 1/2$ was also considered. This noise will be referred to as ‘square root noise’ and is characterized by $P_r$ in the expression:

$$S_{yy}(\omega) = \frac{P_r}{\sqrt{\omega}} + S_{\text{cant}}(\omega).$$

(4.9)

Finally, noise was considered where both the magnitude, $P_n$, and the slope of the noise were allowed to vary. In this paper, this type of noise is referred to as ‘fit-slope noise’ and is described by the following equation:

$$S_{yy}(\omega) = \frac{P_n}{\omega^\beta} + S_{\text{cant}}(\omega).$$

(4.10)

Where fit-slope noise is used, both terms, $P_n$ and $\beta$ are determined by curve fitting.


4.2 Methods

4.2.1 Simulated Data

The FSI model was used in combination with the various noise models to generate idealized simulated data for cantilevers. The magnitude of the noise was set so that at resonance, the magnitude of the PSD of the noise was equal to 10% of the magnitude of the cantilever response. The simulated cantilevers were then calibrated using the thermal noise method after fitting various combinations of noise models and cantilever dynamics to the simulated data. For example, the response of a cantilever would be simulated using the FSI model with pink noise, and a Lorentzian with white noise would be fit to this model. The thermal noise method would then be applied to the idealized FSI model and the resulting Lorentzian for comparison. Simulated data were used to investigate the effect of cantilever dynamic model and noise model mismatches in the absence of unknown system noise. In addition to mismatched cases, the FSI model was fit to FSI generated PSDs as a verification of the fitting technique and to explore the effect of mismatched noise models.

Cantilever parameters were chosen directly from the Veeco product line. All cantilever coatings were ignored since they would not change the dynamics in ways significant to these simulations; the modeled idealized cantilever stiffnesses reflected properties of either silicon or silicon nitride.

4.2.2 AFM Thermal Vibration Measurements

In addition to the work with the idealized models, thermal noise calculations were performed on a set of three Veeco and three MikroMasch cantilevers using the various dynamic models. The cantilever response data were collected using an Asylum Research MFP-3D AFM system. The data were acquired according to the MFP-3D
cantilever stiffness calibration guidelines. Photodiode calibrations were determined by averaging the slopes of the voltage-position curves for both the approaches and retractions for five contact cycles between the cantilevers and a silicon substrate. The thermal responses of each cantilever was recorded five times in water and five times in air. The plan view dimensions of the cantilevers (required to generate the FSI model) were measured with an optical microscope.

4.2.3 Model Parameter Fitting

The curve fits were performed using algorithms based on the MATLAB `fminsearch` algorithm. The first step in the fitting process was to select a frequency range corresponding to the peak response of the cantilever. While this can be done by hand, for the idealized models, the process was automated. The frequency range was selected so that the maximum noise contribution, at the low frequency extreme of the selected data range, was equal to half of the cantilever response. An example plot demonstrating this range is presented as Figure 4.1.

In the case of experimental data, the signal-to-noise ratio is unknown and the frequency range was selected manually. The decision regarding what frequency range to use in the curve fitting was complicated by the presence of a high-pass filter employed in the data collection. The MFP-3D AFM system includes an optional high-pass filter which improves the signal-to-noise ratio for measurements of cantilever dynamics well above the filter cutoff frequency of 160 Hz. As shown in Figure 4.2, this filter impacts the system dynamics at low frequencies in a way that can cause curve fitting algorithms to underestimate the system noise. This underestimation of the noise could be minimized by simply not including low frequency data in the curve fitting process; however, as an alternate strategy, the filter can be included in the modeling so that the low frequency data can be used to help characterize the
Figure 4.1: An example plot of an SHO model with white noise fit to the FSI model with square root noise. The frequency range selected for fitting is symmetrical about the resonance peak and begins where the signal-to-noise ratio is equal to two. This range is highlighted in grey here.
Figure 4.2: Simulated power spectrums illustrating the impact of the high-pass filter. Curve fitting algorithms can underestimate the system noise if the filter is not accounted for and low frequency data is selected for fitting.

noise. This study includes comparisons of the impact of including or omitting this filter in the curve fitting models. In cases where the model did not include the filter, the selected frequency range was restricted to where the first cantilever resonance dominated the signal and could be identified clearly. Models that included a term simulating the high-pass filter were fit to ranges that included a significant amount of the low frequency data which was dominated by system noise. In this way, the frequency range selected for fitting was strategically matched to the features of the models used.

Once a frequency range had been selected for curve fitting, a search algorithm was used to adjust the appropriate model parameters. In the case of the SHO model with white noise, the model parameters were $P_{DC}$, $Q$, $\omega_n$, and $P_w$. The cost function used in the search algorithm is the 2-norm of the difference between the modeled and
where $S_{\text{SHO, white}}$ is the PSD of the SHO model with idealized noise. The 2-norm is the square root of the sum of the squares of a set of $n$ values and varies from the RMS of a set of values by a factor of $1/\sqrt{n}$. In this way, an SHO model was created that best fit the peak data of the FSI model. The models (with the noise removed) were then integrated to perform a calibration according to the thermal noise method [16].

Similarly, when fitting these models to data, the applicable parameters were adjusted to minimize the following cost function:

$$J_{\text{SHO}} = \|S_{\text{data}} - S_{yy}\|_2,$$  \hspace{1cm} (4.12)

where $S_{\text{data}}$ is the measured PSD.

In some cases, an iterative cost function strategy was used to fit the parameters describing the noise separately from the parameters describing the cantilever-fluid system dynamics. This strategy would alternate between fitting the system dynamics parameters by minimizing Equation B.2, and fitting the noise parameters using the following cost function:

$$J_{\text{SHO}} = \left\| \frac{S_{\text{data}} - S_{yy}}{S_{\text{data}}} \right\|_2.$$

(4.13)

Normalizing the cost function by the magnitude of the data is used to perform a fit in a way that is weighted towards smaller magnitudes, such as the data in frequencies where system noise is dominant and cantilever vibration is smaller.

### 4.2.4 Calibration Accuracy Metrics

In cases involving simulated cantilever data, stiffness is a known quantity, therefore, the accuracy of calibrations performed using the simulated data is also known. The experimental PSDs;

$$J_{\text{SHO}} = \|S_{\text{SHO, white}} - S_{yy}\|_2,$$  \hspace{1cm} (4.11)
results of this study are discussed in terms of percent error, $PE$:

$$PE = 100 \frac{(k_{est} - k)}{k},$$  \hspace{1cm} (4.14)

where $k$ is the known stiffness and $k_{est}$ is the stiffness determined using the thermal noise method and a given fitting method under investigation.

In cases involving experimental cantilever data, the cantilever stiffness is an unknown quantity that must be determined through calibration. Thermal noise calibrations were conducted in air using the various noise models and cantilever dynamics models considered in this study, and in all cases the results agreed to within 0.1%. This finding does not provide information regarding the accuracy of the calibrations conducted in air, but it does indicate that the specific noise model and cantilever dynamics model used to curve fit the data was not important for these high quality factor resonances.

Six cantilevers were calibrated in deionized (DI) water using the thermal noise method and every combination of three cantilever dynamics models and nine noise model and filter combinations. The percent error, $PE$, described in Equation 4.14 was found for each of these calibrations substituting the calibration performed in air for the known stiffness value. This resulted in both positive and negative values for $PE$, so the root mean square (RMS) was used rather than the average to quantify the $PE$ values of the six cantilevers generated under each set of fitting conditions. Additionally, the maximum $PE$ values for each testing condition were reported and are discussed in Section 4.3.2.
4.3 Results and Discussion

4.3.1 Fixed-Slope Noise Simulations

In the cases where data was simulated for hypothetical idealized cantilevers, the cantilever stiffness is a known property. A total of seventeen cantilevers were simulated with a minimum stiffness of 0.07 N/m and a maximum stiffness of 70 N/m. As discussed in [50], commercial AFM systems generally fit white noise to measured cantilever vibration data. In Figures 4.3 to 4.6, the FSI model was used along with the four fixed-slope noise types (none, white, square root, and pink) to create simulated PSDs. Each of the cantilever dynamics models (SHO, Lorentzian, and FSI) were then fit to the simulated PSDs along with white noise. Figures 4.3 and 4.4 look very similar. As was expected, the fitting algorithm (MATLAB’s FMINSEARCH routine) was able to fit the FSI model to the FSI generated data to within machine precision when the fitting noise was identical to the simulated noise or when there was no noise simulated. The SHO and Lorentzian models provided fits that resulted in stiffness errors less than 5% for quality factors above three. The calibrations performed for quality factors below two became less accurate, rapidly in the case of the SHO model. In Figures 4.5 and 4.6, modeling white noise to fit the presence of square root noise or pink noise can be seen to have a larger impact on the errors associated with Lorentzian fits to low quality resonance peaks. Error was even introduced to FSI model fits to FSI generated data when the wrong noise model was used in fits to resonance peaks with quality factors below two.

It is important to note that these simulations describe relative behavior for a specific set of circumstances and the results should not be interpreted to represent empirical curves that could serve as correction functions. For example, if the magnitudes of the noise models were adjusted relative to the cantilever dynamics models,
Figure 4.3: The errors associated with modeling white noise in combination with various cantilever dynamics models to perform thermal noise calibrations on simulated data. The simulated data was generated using the FSI model without any noise present. The shaded region highlights the quality factors encountered in experiments.

Figure 4.4: The errors associated with modeling white noise in combination with various cantilever dynamics models to perform thermal noise calibrations on simulated data. The simulated data was generated using the FSI model along with white noise. The shaded region highlights the quality factors encountered in experiments.
Figure 4.5: The errors associated with modeling white noise in combination with various cantilever dynamics models to perform thermal noise calibrations on simulated data. The simulated data was generated using the FSI model along with square root noise. The shaded region highlights the quality factors encountered in experiments.

Figure 4.6: The errors associated with modeling white noise in combination with various cantilever dynamics models to perform thermal noise calibrations on simulated data. The simulated data was generated using the FSI model along with pink noise. The shaded region highlights the quality factors encountered in experiments.
Figure 4.7: Filtered white, square root, and pink noise fit to vibration data collected in air with the data dominated by cantilever resonances removed. Additionally, each constituent noise element is plotted separately.

Changes to these curves would be expected. These simulations demonstrate a source of potential calibration error under idealized conditions. Experimental AFM stiffness calibrations could potentially be conducted in the presence of several types of noise such as those illustrated in Figure 4.7. This data shows the response of a cantilever in air where the resonances exhibit high quality factors. The resonance peaks were removed from the data, leaving a curve dominated by the noise present in the measurement. Curve fitting filtered white, square root, and pink noise to this curve provided an estimation of the noise present in the system. Under these fitting conditions, pink noise and square root noise dominated the signal at lower frequencies where the first resonances of the surveyed cantilevers occur, while white noise best fit the curve at higher frequencies.
4.3.2 Air and Water Calibration Comparisons

A set of six cantilevers were calibrated in both air and water. The tested cantilevers models were Veeco ORC8 (cantilevers B, C, and D) and Mikromasch CSC 38 (cantilevers A, B, and C). These cantilevers have the high length-to-width ratios required for use of the FSI model. The stiffness range of the set (as determined in air) was 0.0427 N/m to 0.516 N/m. The quality factors of the first resonance peaks of these cantilevers in water ranged from 1.4 to 2.4, which is a range that yields significant errors if the wrong noise model is used according to Figures 4.3–4.6. Since the true stiffness values of the cantilevers are unknown, calibrations performed in air were used for comparison, as discussed previously.

Thermal noise calibrations were performed in DI water using a wide variety of modeling configurations. The cantilever stiffnesses are presented in Table 4.1 as calculated in air and in DI water using two of the curve fitting models: the commonly used combination of an SHO model with white noise and a high-pass filtered Lorentzian model with fit-slope noise (described in Equation 4.10.) The percent difference between the water calibrations and the air calibrations were calculated for each modeling configuration as described in Equation 4.14. As shown in Table 4.1, the Lorentzian model in combination with the fit-slope noise provided calibrations in water closer to the calibrations performed in air than the combination of white noise and an SHO model for all but one of the cantilevers.

A significant number of combinations of cantilever dynamics models, noise models, filtering, and cost function weighting resulted in hundreds of calibrations. To consolidate the results, the RMSs of the PEs are presented along with the maximum PEs. In the case of the two situations presented in Table 4.1, the RMS of the PEs of the SHO calibrations was 13.7 % and the maximum difference was 20.2 % while for the Lorentzian case those values were 7.0 % and 9.5 % respectively.
Table 4.1: The stiffness values and percent differences between calibrations performed in air and in water using two different fitting models: the SHO model in combination with white noise and the high-pass filtered Lorentzian model in combination with a fit-slope noise source. The stiffness values are in N/m and the percent differences were determined according to Equation 4.14

<table>
<thead>
<tr>
<th>Cantilever</th>
<th>$k_{\text{air}}$</th>
<th>$k_{\text{SHO,white}}$</th>
<th>$PE$</th>
<th>$k_{\text{Lor,fit-slope}}$</th>
<th>$PE$</th>
</tr>
</thead>
<tbody>
<tr>
<td>ORC8 B</td>
<td>0.122</td>
<td>0.131</td>
<td>7.7</td>
<td>0.122</td>
<td>-0.5</td>
</tr>
<tr>
<td>ORC8 C</td>
<td>0.516</td>
<td>0.499</td>
<td>-3.4</td>
<td>0.470</td>
<td>-9.5</td>
</tr>
<tr>
<td>ORC8 D</td>
<td>0.0691</td>
<td>0.0788</td>
<td>14.1</td>
<td>0.0638</td>
<td>-8.0</td>
</tr>
<tr>
<td>CSC38 A</td>
<td>0.126</td>
<td>0.146</td>
<td>16.0</td>
<td>0.137</td>
<td>8.1</td>
</tr>
<tr>
<td>CSC38 B</td>
<td>0.0427</td>
<td>0.0486</td>
<td>13.8</td>
<td>0.0425</td>
<td>-1.2</td>
</tr>
<tr>
<td>CSC38 C</td>
<td>0.0708</td>
<td>0.0851</td>
<td>20.2</td>
<td>0.0762</td>
<td>8.5</td>
</tr>
</tbody>
</table>

The RMSs of the PEs for a selection of the tested cases are presented in Table 4.2 and the largest differences are presented in Table 4.3. The most simple set of calibrations performed involved the combination of single, fixed-slope noise models with each of the cantilever dynamics models. Thermal noise calibration using white noise and an SHO model (a commonly used technique) is among the configurations that result in the highest PEs. Relative to the SHO model, using a Lorentzian model with white noise reduced the differences between calibrations in air and water significantly. This observation agrees with the findings of Pirzer and Hugel [50].

Using pink, rather than white noise with an SHO also reduced the difference between air and water calibrations. In all cases, fittings involving a Lorentzian or an FSI model were better able to minimize the cost function than fittings involving an SHO model. In nearly all cases, FSI fits yielded smaller cost functions than Lorentzian fits. However, better cost function minimization did not correlate to smaller calibration differences between water and air. SHO models in conjunction with pink noise yielded some of the closest calibrations to those performed in air, even though they did not yield the best fits as quantified by the cost functions.

This result appears to be coincidental. As shown in Figure 4.8, the SHO model
overestimates the magnitude of the response for frequencies lower than the resonance, and underestimates the magnitude of the response for frequencies higher than the resonance. The over- and under-estimations both contribute to higher cost function values during curve fitting; however, they would partially cancel each other out during the integration involved in thermal noise calibration.

In an effort to explore another possible technique for better characterizing the system dynamics, a set of calibrations were performed with a high-pass filter model included in the curve fitting process. The Asylum Research MFP-3D used to collect the thermal data filtered the data with a high-pass resistor-capacitor (RC) circuit with a corner frequency of 160 Hz. To avoid the impact of this filter, two strategies were considered: fitting only data near the resonance peak where the impact of the filter is minimal, and including the filter in the dynamics model to allow the inclusion of the low frequency data in the curve fitting process. The data in Tables 4.2 and 4.3 reveal that neither strategy resulted in water calibrations consistently closer to the
Table 4.2: The RMS of the percent differences between calibrations performed in water with various model fits and calibrations performed in air. The rows are organized by the cantilever dynamics model used. The columns are organized by the noise-type (wh-white, s-square root, p-pink, some combination of the three fixed-slope noise types, or fit-slope noise). The subscript ‘f’ indicates that the model includes a high-pass filter, while a subscript ‘w’ indicates that the weighted noise algorithm (discussed previously) was used.

|        | wh | p  | p(|f|) | wh, s, p(|f|) | wh, s, p(|f|, w) | s, p(|f|, w) | fit-slope | fit-slope(|f|) | fit-slope(|f|, w) |
|--------|----|----|------|--------------|----------------|-------------|-----------|--------------|-----------------|
| SHO    | 13.7 | 6.0 | 6.5  | 6.6          | 12.9          | 9.1         | 8.4       | 6.0          | 6.3             | 11.8            |
| Lorenztian | 7.2  | 7.5 | 7.3  | 7.4          | 8.5           | 7.2         | 7.0       | 6.4          | 7.0             | 7.2             |
| FSI    | 8.1  | 7.5 | 7.8  | 7.2          | 10.6          | 7.8         | 7.4       | 10.0         | 7.5             | 7.8             |

Table 4.3: The largest single value of the percent differences between calibrations performed in water with various model fits and calibrations performed in air for each set of calibrations. The rows and columns are organized and labeled as described in Table 4.2.

|        | wh | p  | p(|f|) | wh, s, p(|f|) | wh, s, p(|f|, w) | s, p(|f|, w) | fit-slope | fit-slope(|f|) | fit-slope(|f|, w) |
|--------|----|----|------|--------------|----------------|-------------|-----------|--------------|-----------------|
| SHO    | 20.2 | -10.0 | 11.3 | 11.5         | 19.2          | 13.5        | 14.5      | 9.9          | 11.2            | 19.5            |
| Lorenztian | -11.4 | 13.7 | -11.5 | -12.2        | 16.0          | -11.6       | -11.6     | 10.4         | -9.5            | -12.1           |
| FSI    | 14.1 | 12.2 | 11.3 | 11.1         | 21.2          | 12.1        | 11.4      | 16.0         | 12.7            | 11.7            |

values determined in air. These results do not indicate that one strategy is better than the other, however, observing the model fit outside of the frequency range used for fitting suggested that using a larger frequency selection resulted in a better modeling of the noise. In many cases, the strategy of omitting the filter and using a smaller frequency range for fitting resulted in fits that modeled the noise poorly, as shown in Figure 4.9. No metric was used to evaluate the model outside of the frequency range used in the curve fitting algorithm, but fit-slope curve fits based on a larger, filtered frequency range did result in more consistent β values. Each calibration was repeated five times and averaged. Fit-slope β values over these five averages would vary by about 20% for the Lorenztian and FSI models when a larger, filtered frequency range was used, but would vary by a factor of two or more when just the data peak was selected.
Calibrations were also conducted with all three fixed-slope noise types simulated simultaneously. This strategy resulted in calibrations that varied from the values determined in air by as much as 19.2 %, 16.0 %, and 21.2 % for the SHO, Lorentzian, and FSI models respectively. One possible source of error stems from the inherent weighting of the cost function, which, as constructed in Equation B.2, results in better fits for the portions of the data of larger magnitude. For this reason, high flexibility in the noise model can yield results where the modeled noise does more to reshape the resonance than to fit the curve to the noise. To address this possibility, an iterative, weighted fitting process was implemented that fit the parameters associated with the cantilever dynamics using Equation B.2, and the parameters associated with the noise model using Equation 4.13. The curve fitting algorithm alternated between fitting each set of parameters with their respective cost functions in an iterative loop.
The iterative, weighted fitting technique was applied in combination with a variety of noise combinations. When all three noise types were modeled simultaneously, the iterative, weighted fitting technique did appear to correct the tendency of the curve fitting process to use the noise parameters to reshape the resonance curve. However, while this strategy yielded calibrations more in-line with previous strategies, it did not improve upon them. The results of the other strategies involving the iterative, weighted curve fitting process were also similar to (or even worse than) some of the simpler algorithms employed.

Figure 4.10: The average values of $\beta$ (from Equation 4.10) plotted against resonance quality factors. The range of quality factors encountered are highlighted in Figures 4.3 – 4.6.

The curve fitting strategies involving fit-slope noise models were impacted by filtering and the weighted, iterative cost function in ways similar to the strategies involving the various fixed-slope noise models. One interesting thing to look at regarding the fit-slope noise models is the resulting $\beta$ values. The most consistent $\beta$...
values resulted from the calibrations performed using high-pass filtered data (covering a wider frequency range) in conjunction with the iterative, weighted fitting algorithm. This calibration strategy yielded $\beta$ values within about 20% of each other across the five calibrations performed for each cantilever for averaging as discussed previously. However, the $\beta$ values varied more between cantilevers, with mean $\beta$ values ranging from 0.85 to 1.30 when fit to data in combination with the Lorentzian model and from 0.79 to 1.05 with the FSI model. This variation may reflect the nature of the noise present in the AFM system used in this work, and that result would not be surprising in light of the observed variation in the slope of the noise presented in Figure 4.7. However, plotting the $\beta$ values against cantilever stiffnesses or the resonance frequencies did not reveal as convincing a relationship as plotting against quality factors (Figure 4.10.) A relationship between the quality factor and the fit value of $\beta$ may suggest that the fitting algorithm is using the modeled noise to correct for some failing of the cantilever dynamics models (even though the cost function was weighted to minimize this effect as previously discussed.) With only six cantilevers tested, observed trends could be considered anecdotal, but it is worth noting that the relationship appears to be significantly more clear with the FSI model than the Lorentzian.

4.4 Summary and Conclusions

Preliminary testing involving thermal noise method calibrations performed on AFM cantilevers in air revealed that the cantilever dynamics model and the noise model used in the curve fitting process had very little impact in the resulting stiffness values. This reinforces the idea that, if possible, thermal noise method calibrations should be performed in gas environments or in vacuum to ensure that the measured resonance peak exhibits a high quality factor.
AFM cantilevers calibrated using the thermal noise method in environments resulting in low quality factor resonances are subject to error associated with the selection of the models used to account for both the cantilever dynamics and the noise present in the measurement system. For the calibrations performed here, either using a different cantilever dynamics model or different noise model yielded improvements over an SHO model/white noise fitting; however, changing both did not yield further improvement. More sophisticated fitting strategies were attempted, and while some strategies did a better job minimizing the cost functions associated with fitting models to data, and other strategies did a better job modeling the noise over larger frequency ranges, these improvements did not yield further reduction of the difference between calibrations performed in DI water and in air. The FSI model, while specifically designed to describe cantilever dynamics in heavy fluids and capable of fitting measured data with smaller resulting cost functions than a Lorentzian, did not yield closer stiffness calibrations in DI water. Similarly, in some configurations the SHO model yielded calibrations in DI water that were closer to calibrations performed in air than the Lorentzian or the FSI model, even though it resulted in the poorest curve fittings as determined by the cost function.

Based on these findings, thermal noise AFM cantilever calibrations performed in water should be conducted with a good understanding of the system noise present. The combination of the Lorentzian model with what is described in this chapter as ‘fit-slope noise’, fit over a wide frequency range and using a high-pass filter (if necessary) to reflect the measurement system, is a good compromise between performance determined by the comparison of air and water calibrations and by the minimization of cost functions and visual inspection of the noise fit beyond the frequency range considered in the fitting algorithm. This strategy resulted in a maximum difference between air and water calibrations of 9.5 % and an RMS of the differences between
calibrations of 7.0 %. The common strategy involving white noise in combination with an SHO model resulted in a maximum difference of 20.2 % and an RMS difference of 13.7 %.
Chapter 5

Noncontact Calibration in the Presence of Heavy Fluid Loading

Cantilever calibration techniques based on the analysis of the random, thermally-driven vibrations of a free cantilever are non-destructive and do not require any equipment in addition to what is normally required to make AFM measurements. This makes calibration techniques based on the thermal response highly desirable and a great deal of work has been aimed at improving such methods.

The ability to perform noncontact displacement and stiffness calibrations requires invoking the equipartition theorem along with a noncontact stiffness calibration method. Higgins et al., use Sader’s method to perform the stiffness calibration [20]. Since Sader’s method includes a light fluid loading assumption, this method is generally appropriate for experiments conducted in air or for experiments where the stiffness calibration can be conducted in air before the probe is used in a heavy fluid loading environment.

This chapter presents a noncontact cantilever stiffness calibration method that incorporates a heavy fluid loading assumption, which enables the stiffness calibration of cantilevers in liquid environments. The FSI model discussed in Chapter 3 is curve fit to the thermal noise of eleven cantilevers in water in a way that restricts the search space to parameters that reflect heavy fluid loading conditions. Sader’s method and the thermal noise method, discussed in Chapter 2 are conducted in air for comparison. Finally, Higgin’s approach is used in combination with this new calibration technique to perform noncontact photodiode and stiffness calibrations on the unscaled photodiode output voltage of cantilevers in water. Based on the results
presented in Chapter 4, a Lorentzian distribution in combination with variable-slope white noise is used to fit the unscaled voltage output of measured thermal noise to produce an InvOLS value. The combination of these two techniques enables both the stiffness and InvOLS calibrations to be performed in water, without the need to apply a known displacement to the tip.

5.1 Methods

5.1.1 Data Acquisition and Processing

The cantilever response data were collected using an Asylum Research MFP-3D AFM system. The data were acquired according to the in-program cantilever stiffness calibration guidelines. Photodiode calibrations were determined by averaging the slopes of the voltage-position curves for both the approaches and retractions for five contact cycles between the cantilevers and a silicon substrate. Each calibration was performed five times and averaged. The plan view dimensions, tip height, and the distance from the tip to the cantilever end were measured with an optical microscope.

The frequency response data were processed in MATLAB. To account for the difference between the slope-to-position ratios of the cantilever end under static loading (during photodiode calibration) and freely vibrating (during thermal data collection), the appropriate correction factor, $\chi$, was found according to [19]. In this study, $\chi$ values ranged from 1.027 to 1.082. (In the limit of an infinitely small laser spot, positioned exactly at the end of a rectangular cantilever, $\chi = 1.09$.) To compare both the intrinsic cantilever stiffness identified by Sader’s technique and the new technique presented here to the effective cantilever stiffness identified by the thermal noise method, corrections were made to account for the cantilever angle, the tip height, and the distance from the tip of the probe to the end of the cantilever [60, 61]. These corrections
are discussed in detail in Appendix A. To compare measured InvOLS values to InvOLS determined through the analysis of unscaled photodiode output, the thermal vibration data were recorded according to the in-program calibration guidelines and then modified in MATLAB (the PSDs of the thermal response were divided by the square of the measured InvOLS) to reflect what they would have been had the initial InvOLS calibration not been conducted.

5.1.2 Data Fitting Techniques

The FSI model of cantilever thermal response was fit to the measured data by minimizing a cost function that compared measured data to the theoretical response corresponding to a given set of input parameters. The hydrodynamic loading used within the model, \( \Gamma(i\omega) \), was determined using the measured specifications for the cantilever lengths and widths, and the room temperature properties of the fluids enveloping and exciting the cantilevers; it was assumed that these parameters are known with sufficient accuracy. The model fitting process did not assume knowledge of parameters such as the thickness and the Young’s modulus which cannot be known with sufficient accuracy due to the manufacturing methods. The remaining parameters needed to model the thermal response were the natural frequency, \( \omega_n \), and the fluid-to-cantilever mass ratio, \( \gamma \). Additionally, parameters were fit to account for measurement noise and a fitting parameter was added to allow the magnitude of the model to be re-scaled as discussed in Chapter 3. This ‘scale-less’ fitting was performed in response to the observation that the fitting process was extremely sensitive to the magnitude of the data, which is subject to the inaccuracy of the photodiode calibration process. For a specific set of parameters the resulting model of cantilever response was evaluated using the following cost function:

\[
J = \left\langle (S_{qq} - S_{data})^2 \right\rangle,
\]

(5.1)
where $S_{qq}$ is the PSD of the modeled thermal response and $S_{data}$ is the PSD of the measured thermal response of a microcantilever. The MATLAB function \texttt{FMINSEARCH} was used to minimize the cost function, $J$, by adjusting the fitting parameters. The frequency range of the data selected for fitting captured enough of the resonance peak so that the ratio of the maximum value in the selected data range to the minimum value in the selected data range was equal to five. While a value of five was selected arbitrarily, results calculated using data ranges with maximum-to-minimum ratios as small as two yielded similar results. An example curve fit is shown in Figure 5.1 along with SHO and Sader model fits for comparison.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure5_1}
\caption{The measured thermal vibration of MikroMasch cantilever model CSC38 B in water with SHO, Sader, and fluid-structure interaction model curve fits.}
\end{figure}
5.1.3 Mapping the Cost Function

The cost function, $J$, presented in Equation 5.1, serves as a means to compare the modeled cantilever response for a given set of parameters to measured data recorded for a specific cantilever. For a number of cantilevers, this cost function was evaluated at points over large ranges of $\omega_n$ and $\gamma$. For each pairing of $\omega_n$ and $\gamma$ values, the MATLAB function `FMINSEARCH` was used to scale the resulting model to minimize the cost function. In this way, plots were created that mapped the cost as a function of $\omega_n$ and $\gamma$. An example plot is presented as Figure 5.2.

5.1.4 Heavy Fluid Loading Stiffness Calibration Method

Based on the results of the initial data fitting effort and observations made by plotting the cost maps which will be discussed in the following section, a MATLAB program was written to automate the process of calibrating cantilevers by fitting the fluid-
structure interaction model to a measured cantilever response in a way that restricted
the fit to the heavy fluid loading region of the cost map. A flowchart of this method
is presented in Figure 5.3. The program first requires the user to provide initial
parameter guesses to create a rough curve fit to the data. The program then identifies
the frequency range of the data to be fit as was done in the unrestricted data fittings.
Parameter initialization is conducted through the use of a graphical user interface
shown in Figure 5.4. The program performs a series of curve fits to find points that
lie on the low cost function curve (discussed in Section 5.2.1). Points are identified
along the curve starting from a low frequency and then at increasing frequencies until
the resulting value for $k$ converges. To do this, the initial guess for $\omega_n$ is set to $\omega_r$.
The program then uses the FMINSEARCH function to find the value of $\gamma$ and the scaling
factor that best fits the model to the data for that specific value of $\omega_n$ (the scaling
factor is discussed as part of Equation 4.6, in Section 4.1.2). This process is then
repeated with a new $\omega_n$ guess that is 50% higher than the previous guess. (The
technique is robust to different size steps in frequency. Frequency step multipliers
from 1.1 to 1e5 produce similar results.) A value for $k$ is found for each set of
parameters using the following relation:

$$k = \frac{3m'\omega_n^2}{(\alpha_1l)^4\gamma}.$$  \hspace{1cm} (5.2)

Each sequential set of parameters identify a point along the low cost function curve
with a higher mass ratio, $\gamma$, than the previous point. In this way, the heavy fluid
loading calibration program walks along the low cost function curve until the resulting
value for $k$ converges and is therefore not sensitive to the specific values of $\gamma$ and $\omega_n$
identified as will be discussed in the following section. The program is presented
along with a detailed description in Appendix B.
5.1.5 Noncontact Displacement Calibration Method

To estimate the InvOLS value from unscaled data, two other elements of the testing conditions had to be accounted for: the laser position and the cantilever angle. In addition to the InvOLS factor, the MFP-3D automatically applies a $\chi$ factor of 1.09 described in Section 5.1.1 and Appendix A. Using the $\chi$ factors that properly account for the laser position, the relationship between the MFP-3D output and the cantilever displacement is:

$$V \times \text{InvOLS} \times \frac{\chi}{1.09} = q_{\text{eff}},$$  \hspace{1cm} (5.3)

where $q_{\text{eff}}$ is the effective displacement, meaning it is the displacement of the tip in the direction of interest. The direction of interest is the direction of any anticipated applied forces, which is not necessarily normal to the plane of the cantilever. In the MFP-3D, cantilevers were held at a $12.5^\circ$ angle to the substrate surface. Accounting for the angle between the pulling direction and the direction normal to the cantilever along with the position of the probe tip on the beam, the cantilever stiffness in the
Figure 5.4: A screenshot of the graphical user interface used to implement the heavy fluid loading stiffness calibration algorithm.
pulling direction is:

\[ k_{\text{eff}} = \frac{k}{\cos^2 \theta} \left( \frac{l}{l_2} \right)^3. \]  

(5.4)

The software written to perform thermal noise calibrations using various noise and system dynamics models for Chapter 4 is included in Appendix B.3. This software is intended to perform calculations according to the thermal noise method; however, applied to an unscaled voltage, it will yield an unscaled stiffness, \( k_{\text{unscaled}} \), equal to:

\[ k_{\text{unscaled}} = 0.971 \frac{k_B T}{\langle V^2 \rangle}. \]  

(5.5)

Invoking the equipartition theorem, plugging in Equations 5.3, 5.4, and 5.6, and solving for the InvOLS yields:

\[ \text{InvOLS} = \sqrt{\frac{k_{\text{unscaled}}}{k} \frac{1.09}{\chi} \cos \theta}. \]  

(5.6)

5.2 Results and Discussion

5.2.1 Noncontact Stiffness Calibration Results

The calibration algorithm presented here was based on the results of the analysis of the cost function maps described in the previous section. For this reason, the findings from the cost function map investigation are presented along with the results of the actual calibration technique.

Cost Function Maps

To better understand the stiffness estimates generated by an unrestricted fitting of the FSI model to measured data, MATLAB was used to map the cost function over
a range of \( \gamma \) and \( \omega_n \) values as discussed previously (Figure 5.2). Each point on the cost function map corresponds to a specific set of potential values of \( \gamma \) and \( \omega_n \). Dark regions of the plot identify a locus of parameters that yield FSI models that line up well with the measured cantilever frequency response. Each set of values for \( \gamma \) and \( \omega_n \) can be used to solve for a cantilever stiffness value. While accurate estimations of \( \gamma \) and \( \omega_n \) for a specific cantilever would presumably yield accurate cantilever stiffness estimations, the imperfections of the model can cause the cost function to be minimized for incorrect values of \( \gamma \) and \( \omega_n \).

The cost map looks different for cantilevers under light fluid loading. Figure 5.5 shows a cost function map for a cantilever in air. The low cost function locus is contained to a specific point. Under heavy fluid loading (see Figure 5.2) the low cost function locus forms a curve establishing a relationship between \( \gamma \) and \( \omega_n \). Any set of \( \gamma \) and \( \omega_n \) values falling within this locus will yield a reasonable model of the cantilever dynamics. This reflects the findings that under light fluid loading, parameter identification is straightforward while under heavy fluid loading, the FSI model is robust to parameter uncertainty which makes parameter identification based on curve fitting difficult [48].

Overlaying curves of the \( \gamma \) and \( \omega_n \) points that yield constant cantilever stiffness values onto a cost function map can illustrate the sensitivity of the resulting stiffnesses to changes in \( \gamma \) and \( \omega_n \) values. It is also illustrative to scale the abscissa against \( \omega_n^2 \) to linearize the curves of constant stiffness as is shown in Figure 5.6. The low cost function locus aligns with the lines of constant stiffness for high values of \( \gamma \) and \( \omega_n \). This indicates that for higher values of \( \gamma \) and \( \omega_n \) within the locus, the resulting stiffness is not sensitive to perturbations. If the mass loading describing the cantilever behavior is sufficiently high, the cantilever stiffness estimation is not sensitive to changes in the \( \omega_n \) and \( \gamma \) estimations as long as the FSI model provides
Figure 5.5: The cost function map for Veeco cantilever model ORC8 C in air rather than water.

a good fit to the data using those parameters. This implies that in these cases, cantilevers can be calibrated under heavy fluid loading by fitting the FSI model to data in a way that restricts the search to the high $\gamma$ region of the locus. While it is difficult to identify the individual $\omega_n$ and $\gamma$ parameters from the thermal response of a heavily loaded cantilever [48], this technique only requires the identification of the ratio of $\omega_n^2$ to $\gamma$. This can be seen in Equation 5.2, where identifying parameters $m'$, $\alpha_1$, and $l$ only requires knowledge of the fluid density and the plan-view dimensions of the cantilever beam. The effect of searching for the appropriate ratio of $\omega_n^2$ to $\gamma$, is to reduce two degrees of freedom in the fitting process to a single degree of freedom.

To illustrate the difference between an unrestricted curve fitting and the new technique presented here, the $\omega_n$ and $\gamma$ values corresponding to an unrestricted curve fitting have been identified on a cost map in Figure 5.7. The line running through this point, indicating the values of $\omega_n$ and $\gamma$ that would yield the same stiffness, can be seen to diverge from the low cost function locus. This indicates that the resulting
stiffness estimate from the unrestricted curve fitting is sensitive to perturbations to the fit parameters. Sensitivity to perturbations is undesirable since there is significant uncertainty involved with the curve fitting of cantilevers under heavy fluid loading [48]. The second line identifies parameter combinations that result in the stiffness estimate generated by the algorithm presented here. It runs along the low cost function locus for high fluid loading values identifying a range of parameters that yield good fits between the FSI model and the measured data.

**Heavy Fluid Loading Curve Fit Results**

Calibrations were performed in air using Sader’s method and the thermal noise method and in deionized water using the thermal noise method, the unrestricted fitting of the FSI model, and using the new, restricted FSI model fitting algorithm presented here. The results are presented in Table 5.1. To illustrate the difference
Figure 5.7: The cost function map for Veeco cantilever model ORC8 C in water. The circle indicates the unrestricted minimum point of the cost function. The dashed line corresponds to the stiffness estimated by the unrestricted data fitting while the solid line indicates the points that correspond to the stiffness estimated using the new heavy fluid loading algorithm. The solid line remains within the low cost locus for higher values of $\gamma$. 
between each calibration method and Sader’s method, the percent differences were plotted in Figures 5.8 and 5.9.

The fluid loading model assumes that the cantilever length greatly exceeds the cantilever width. Figure 5.8 reveals that the percent differences are largest when the $l/b$ ratio is small. The results in Figure 5.9 are ordered according to the $l/b$ ratios such that from left to right, $l/b$ values decrease, and the $l/b$ assumption becomes less valid. In the initial presentation of Sader’s method, accuracy is claimed for cantilevers with $l/b$ ratios that exceed three [17]. These results demonstrate this new method performed in water to be within 10 % of Sader’s method performed in air for $l/b$ values that exceed 3.5 (which is indicated by the vertical gray line in Figure 5.9.) This may be an indication that the $l/b$ requirement plays a larger role under heavy fluid loading.

The restricted FSI fitting calibration technique can be seen to provide a closer stiffness value to Sader’s method than the unrestricted FSI fitting for all cases tested. For the eight cantilevers with $l/b > 3.5$, the absolute improvement ranged from 0.9 % for cantilever two to 6.4 % for cantilever ten. The average improvement was 3.4 %. In three cases (cantilevers five, seven, and ten), the new method improved the calibration from a difference larger than 10 % to a difference smaller than 10 %.

The calibrations produced using the thermal noise method are included in Table 5.1 and Figure 5.9 for comparison. The thermal noise calibrations performed in air and in water varied from the Sader’s method calibrations and from each other over a range up to the typical 20 % (with the exception of cantilever eleven, which exhibited a poor signal-to-noise ratio during testing.) The selection of Sader’s method to serve as the standard to plot against should not be interpreted as an indication of the superior accuracy of one method over another. The eleven cantilevers used in this study were pointed or ‘dog-eared’ at the free end rather than rectangular. This is
Figure 5.8: The percentage difference between calibration values determined using the new method and the calibration determined via Sader’s method plotted against the $l/b$ ratio.

A common feature of commercially available cantilevers that is not accounted for in the fluid loading model and could contribute to inaccuracy. Additionally, the shape of the free end of the cantilever could impact the $\chi$ value, which would affect the comparison of intrinsic stiffnesses to effective stiffnesses. The thermal noise data collected for the higher stiffness cantilevers (cantilevers nine and eleven) exhibited weaker signal-to-noise ratios which may impact the thermal noise calibration results as has been noted elsewhere [1]. For these reasons, further study is warranted into the accuracy of various calibration techniques under various conditions. The results presented here suggest that the restricted FSI fitting technique can serve as a tool to pursue this line of study.
Table 5.1: Comparison of stiffness estimations for eleven different cantilever models. The ORC8 and RFESP cantilevers were manufactured by Veeco, while the CSC38 and NSC36 cantilevers were manufactured by Mikromasch. The methods used include Sader’s method, an unrestricted fitting of the fluid-structure interaction model to data, the new method presented in this document, and the thermal noise method. The values for stiffness, $k$, are in N/m. The length-to-width ratio, $l/b$, is assumed to be much greater than one in the fluid loading model which impacts all methods here except for the thermal noise method.

<table>
<thead>
<tr>
<th>No.</th>
<th>Model</th>
<th>$k_{\text{Sader, air}}$</th>
<th>$k_{\text{FSI fit, water}}$</th>
<th>$k_{\text{new, water}}$</th>
<th>$k_{\text{therm, air}}$</th>
<th>$k_{\text{therm, water}}$</th>
<th>$l/b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>ORC8 A</td>
<td>0.848</td>
<td>0.970</td>
<td>0.961</td>
<td>0.885</td>
<td>0.827</td>
<td>2.6</td>
</tr>
<tr>
<td>2</td>
<td>ORC8 B</td>
<td>0.127</td>
<td>0.137</td>
<td>0.136</td>
<td>0.125</td>
<td>0.131</td>
<td>5.1</td>
</tr>
<tr>
<td>3</td>
<td>ORC8 C</td>
<td>0.467</td>
<td>0.486</td>
<td>0.455</td>
<td>0.564</td>
<td>0.544</td>
<td>5.2</td>
</tr>
<tr>
<td>4</td>
<td>ORC8 D</td>
<td>0.0674</td>
<td>0.0708</td>
<td>0.0682</td>
<td>0.0709</td>
<td>0.0752</td>
<td>10.2</td>
</tr>
<tr>
<td>5</td>
<td>CSC38 A</td>
<td>0.158</td>
<td>0.175</td>
<td>0.172</td>
<td>0.128</td>
<td>0.145</td>
<td>6.9</td>
</tr>
<tr>
<td>6</td>
<td>CSC38 B</td>
<td>0.0494</td>
<td>0.0514</td>
<td>0.0491</td>
<td>0.0429</td>
<td>0.0467</td>
<td>10.6</td>
</tr>
<tr>
<td>7</td>
<td>CSC38 C</td>
<td>0.0802</td>
<td>0.0883</td>
<td>0.0868</td>
<td>0.0713</td>
<td>0.0808</td>
<td>8.4</td>
</tr>
<tr>
<td>8</td>
<td>NSC36 A</td>
<td>2.23</td>
<td>2.69</td>
<td>2.52</td>
<td>2.11</td>
<td>2.01</td>
<td>3.0</td>
</tr>
<tr>
<td>9</td>
<td>NSC36 B</td>
<td>4.69</td>
<td>5.68</td>
<td>5.53</td>
<td>4.85</td>
<td>4.26</td>
<td>2.4</td>
</tr>
<tr>
<td>10</td>
<td>NSC36 C</td>
<td>1.48</td>
<td>1.71</td>
<td>1.62</td>
<td>1.38</td>
<td>1.30</td>
<td>3.6</td>
</tr>
<tr>
<td>11</td>
<td>RFESP</td>
<td>5.76</td>
<td>6.29</td>
<td>5.94</td>
<td>4.35</td>
<td>4.03</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Figure 5.9: The percentage difference between each calibration value and the calibration determined via Sader’s method for each cantilever. The cantilevers are ordered according to their \( l/b \) ratio and are numbered to match the labels in Table 5.1. Cantilevers to the right of the vertical gray line have \( l/b \) ratios less than 3.5.
5.2.2 Noncontact Displacement Calibration Results

By combining the heavy fluid loading fitting technique with the equipartition theorem using a Lorentzian distribution and fit-slope noise to model the thermal response, both stiffness and InvOLS calibrations were performed on ten cantilevers using unscaled thermal vibration data. (Cantilever number eleven was omitted due to the high signal-to-noise ratio.) A comparison of Tables 5.1 and 5.2 reveal that changing the scale of the data does not impact the resulting stiffness estimation. Table 5.2 also shows that for this set of cantilevers, the maximum difference between noncontact InvOLS\textsubscript{N} and contact InvOLS\textsubscript{C} is 11.3%.

Table 5.2: Stiffness and InvOLS estimations generated from the analysis of unscaled thermal vibration data for rectangular cantilevers in water. Stiffness values are in N/m and InvOLS are in nm/V. The final two columns present the percent change in the calculated InvOLS and the percent change in the resulting force measurement.

<table>
<thead>
<tr>
<th>No.</th>
<th>Model</th>
<th>( k_{\text{eff, water}} )</th>
<th>InvOLS\textsubscript{C}</th>
<th>InvOLS\textsubscript{N}</th>
<th>( \Delta \text{InvOLS} ) (%)</th>
<th>( \Delta f ) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>ORC8 A</td>
<td>0.961</td>
<td>30.52</td>
<td>28.29</td>
<td>-7.3</td>
<td>5.0</td>
</tr>
<tr>
<td>2</td>
<td>ORC8 B</td>
<td>0.136</td>
<td>52.13</td>
<td>50.06</td>
<td>-4.0</td>
<td>2.8</td>
</tr>
<tr>
<td>3</td>
<td>ORC8 C</td>
<td>0.455</td>
<td>39.51</td>
<td>42.53</td>
<td>7.6</td>
<td>4.9</td>
</tr>
<tr>
<td>4</td>
<td>ORC8 D</td>
<td>0.0682</td>
<td>78.68</td>
<td>77.73</td>
<td>-1.2</td>
<td>-0.0</td>
</tr>
<tr>
<td>5</td>
<td>CSC38 A</td>
<td>0.172</td>
<td>121.94</td>
<td>111.12</td>
<td>-8.9</td>
<td>-0.1</td>
</tr>
<tr>
<td>6</td>
<td>CSC38 B</td>
<td>0.0491</td>
<td>163.83</td>
<td>160.96</td>
<td>-1.8</td>
<td>-2.3</td>
</tr>
<tr>
<td>7</td>
<td>CSC38 C</td>
<td>0.0868</td>
<td>89.15</td>
<td>82.66</td>
<td>-7.3</td>
<td>0.4</td>
</tr>
<tr>
<td>8</td>
<td>NSC36 A</td>
<td>2.52</td>
<td>37.37</td>
<td>33.48</td>
<td>-10.4</td>
<td>1.2</td>
</tr>
<tr>
<td>9</td>
<td>NSC36 B</td>
<td>5.53</td>
<td>26.30</td>
<td>23.32</td>
<td>-11.3</td>
<td>4.6</td>
</tr>
<tr>
<td>10</td>
<td>NSC36 C</td>
<td>1.62</td>
<td>42.80</td>
<td>38.73</td>
<td>-9.5</td>
<td>-0.9</td>
</tr>
</tbody>
</table>

To illustrate the impact of using these techniques rather than using Sader’s calibration method in air and then performing a static force displacement InvOLS calibration in water, the final column of Table 5.2 presents the following ratio as a percentage:

\[
f_{\% \Delta} = \left( \frac{k_{\text{eff, water}} \text{InvOLS}\textsubscript{N}}{k_{\text{Sader, air}} \text{InvOLS}\textsubscript{C}} - 1 \right) \times 100%. \tag{5.7}\]
Force measurements making use of the noncontact stiffness and InvOLS calibrations in water presented here would have been within 5 % of the same measurements made using Sader’s calibration method in air followed by an InvOLS measurement using contact displacement in water.

5.3 Summary and Conclusions

A new cantilever calibration technique has been presented that is specifically designed for calibrating rectangular cantilevers by analyzing the thermal response under heavy fluid loading. This technique is similar to Sader’s calibration method in two ways; it does not require absolute magnitude scaling of the thermal vibration measurement (i.e., it does not require photodiode calibration for optical lever type AFM measurements), and it requires only knowledge of the length and width of a cantilever along with the density and viscosity of the surrounding fluid. This technique was used to calibrate eleven cantilevers (six MikroMasch and five Veeco cantilevers) that covered a stiffnesses range from 0.05 N/m to 5.8 N/m. For comparison, the cantilevers were also calibrated in air using Sader’s method. For cantilevers with $l/b$ ratios greater than 3.5, the maximum difference between Sader’s method performed in air and this new method performed in water was 9.4 %.

Using this heavy fluid loading calibration technique in combination with the equipartition theorem enables noncontact InvOLS calibration. Using the fit-slope noise model in combination with a Lorentzian distribution to fit the thermal vibration as described in Chapter 4, InvOLS values were calculated that varied from measured InvOLS (which required contact) by a maximum of 11.3 %. Comparing the stiffness and InvOLS values revealed that force measurements made using these noncontact methods in water would have varied from measurements made using Sader’s calibration conducted in air in combination with a forced, static displacement InvOLS.
measurement by a maximum of 5.0 %. While a sample set of ten cantilevers can only be considered anecdotal evidence, it is possible that the combination of factors that vary by 10 % yielding force measurements that vary by 5 % suggests that there are systematic biases to some or all of the calibrations involved. It is possible that biases cancel each other out resulting in force measurements that are more accurate than the stiffness and InvOLS calibration methods. Alternately, it is possible that there are systematic biases common to both sets of approaches resulting in force measurements that share the same bias. In either case, this result suggests that there is a need for further research to explore the possible bias sources.
Chapter 6

Finite Element Modeling of Fluid-Structure Interaction

Finite element method (FEM) was used to investigate the system dynamics in cases that involved geometries or fluid conditions that are difficult to address analytically or experimentally. Examples include V-shaped cantilevers, which are discussed in Chapter 7, and warped cantilevers, which are discussed in Chapter 8. The ANSYS Academic Research, Release 12.1 software package was used to run a variety of analysis types including static loading reactions, transient responses, and modal analyses of cantilever structures in the absence of fluid loading. Additionally, multi-field analyses coupled the structural FEM model of a cantilever to a computational fluid dynamics (CFD) model of the liquid environment. The following sections describe these simulations and how the results were used to determine cantilever-fluid system properties.

6.1 Static Displacements

Static structural analyses enable the simulation of the reaction of a structure to an applied static force. Static loads were applied at the tip of the cantilever probe in two different directions. Loads were applied normal to the plane of the cantilever to evaluate meshing requirements and compare the resulting deflection to the theoretical expectation. In Chapter 8, loads were applied at 12° to the direction normal to the plane of the cantilever as shown in Figure 6.1 to simulate mounting conditions of a cantilever in a typical AFM system.
Figure 6.1: The end of a cantilever simulated in ANSYS. The red arrow illustrates that the static load was applied to the probe tip at a $12^\circ$ angle.

The mesh was generated automatically within the ANSYS Workbench environment. Except where specified otherwise, the default meshing options were used, including the selection of Mechanical for the Physics Preference. With these settings, the meshing algorithm uses a Patch Conforming technique, which scales element features to the geometry of the structure without taking loading or boundary conditions into consideration. The mesh fineness was controlled by adjusting the maximum element size of the mesh and the Relevance setting, which adjusts mesh parameters in a way that offers a tradeoff between mesh fineness and simulation run time. An example mesh is shown in Figure 6.2.

The simulated cantilever beam in the convergence study was modeled on the Veeco ORC8-D cantilever. It is 200 $\mu$m long by 20 $\mu$m wide. The thickness, 0.54288 $\mu$m, was selected (using Equation 2.3) so that using the material properties of silicon nitride, the cantilever would have a bending stiffness of $k = 0.02$ N/m, which is the nominal value for cantilever ORC8-D. A load of 0.02 $\mu$N was applied to the probe tip in the $+Y$ direction (using the orientation system shown in Figure 6.1 where
Figure 6.2: An example mesh; only the elements on the surface of the cantilever are visible.

+Y is normal to the top surface of the cantilever). The simulated displacement of the tip in the +Y direction was then used to determine the cantilever stiffness. A convergence metric was not established prior to simulation, rather, the entire range of the Relevance setting was explored (-100 to 100) under the default settings; then the mesh was further refined by reducing the maximum element size. In the static convergence study, simulations were run with as many as 80,000 nodes, resulting in a cantilever stiffness of 0.02014 N/m, which is within 1 % of the expected value. Potential differences between simulated performance and theoretical expectations based on analytical expressions could stem from the presence of the probe tip at the end of the cantilever and the measurement technique, which observed displacement in the +Y direction, but ignored motion in the -X direction.

Figure 6.3 illustrates the convergence of the cantilever stiffness as the mesh was
refined. The stiffness values are normalized against the stiffness of the simulation with the highest node count, i.e.:

\[ \hat{k} = \frac{k}{0.02014}. \]  

(6.1)

Doubling the number of nodes from about 8,500 to about 17,000 results in a change in stiffness of about 0.1 %. Therefore, meshes with more than 8,500 nodes were deemed sufficient for the simulation of static displacements.

### 6.2 Modal Analyses

The cantilever geometry and meshing techniques used in Section 6.1 were also applied to the modal analysis. The intent of examining the modal analysis in Chapter 8 involves details of the mode shapes as well as the frequencies of the first bending mode and the first twisting mode. These correspond to the first and fourth modes
Figure 6.4: The normalized natural frequencies of the first bending and twisting modes of the simulated cantilever as a function of mesh nodes. In each case, the natural frequencies were normalized against the natural frequencies of the simulation with the finest mesh.

of the example cantilever. To determine convergence, the natural frequencies of the first and fourth modes were determined and normalized similar to the stiffnesses in Section 6.1. The normalized natural frequencies are presented in Figure 6.4. As can be observed in the figure, the first bending mode converges more quickly than the first twisting mode. The least refined mesh contained 5,802 nodes and had a first twisting mode natural frequency within 1% of the final value for all meshes run. At 10,826 nodes, the natural frequency was within 0.5% of the final value.

The expected natural frequencies for the first bending and torsional modes are 16,815 Hz and 330,490 Hz, respectively. The simulated natural frequencies for a mesh with 64,983 nodes were 16,279 Hz and 333,610 Hz, which vary from the analytical values by -3.1% and 0.9%. The mass of the probe tip is likely to have a bigger impact on the first bending mode than the first torsional mode since it is located at a position where the modal displacement of the first bending mode is large, but the
modal displacement of the first torsional mode is small as shown in Figures 6.5 and 6.6.

6.3 Transient Responses of Cantilevers

The setup of a transient response simulation requires consideration of both spatial and temporal parameters to ensure sufficient accuracy. Transient response simulations
were conducted for cantilever beams without a fluid environment to more-quickly
determine some of the temporal resolution requirements that would also apply to
cantilever-fluid simulations. The cantilever modeled in these simulations was identical
to the model used for the static loading and modal analyses described in Sections 6.1
and 6.2.

6.3.1 Simulation Time Steps

To determine an appropriate time step for the simulation, a step input was applied to
the structure and the simulation was run for 6e-4 s, which was enough time to capture
the maximum displacement. A mesh containing 17,897 nodes was used and the time
step was varied from 1e-5 to 1e-7 s. At 1e-7 s, the maximum displacement of the
probe tip was 1.9548 µm. Figure 6.7 shows the maximum displacement normalized
by the final displacement value (as was done in Sections 6.1 and 6.2) plotted against
the inverse of the time step for each simulation. Time steps smaller than 1.25e-6 s
yielded displacements within 1.0 % of the final value, and time steps smaller than
7.5e-7 s reduced this metric to within 0.5 %.

6.3.2 Simulation End Time

Transient responses were simulated in order to determine the dynamic properties of
cantilever-fluid systems. In cases where the quality factor is much greater than one,
an SHO can be used to approximate a cantilever resonance and identify appropriate
values for $k$, $Q$, and $f_n$. While the stiffness and natural frequency of the cantilever
can be identified through simpler simulations, the transient response can be used to
identify the quality factor of a cantilever-fluid system resonance. The technique for
identifying dynamic system properties from a transient response involved fitting a
model containing four SHOs to the simulated response, which is discussed in Section
Figure 6.7: The normalized maximum displacement of the cantilever in response to a step function as a function of the inverse of the time step. The displacements were normalized against the displacements of the simulation with the finest mesh.

6.4. To identify how long a simulation should run in terms of end time, damping was added to the cantilever model simulated in Sections 6.1 and 6.2 and the $k$, $Q$, and $f_n$ values that resulted from fitting responses of different time lengths were evaluated.

The transient response simulations were similar in nature to an impact hammer test. A simulated load was applied to the probe tip in the Y+ direction (using the coordinate system illustrated in Figure 6.1) for a set period of time, followed by a period of free cantilever oscillation without an applied load. Like an impact hammer test, the relative excitation of various cantilever modes was impacted by the length of time of the applied force. The time step convergence tests (Subsection 6.3.1) revealed that the time constant of the system was approximately 1.2e-5 s. An applied force duration of roughly double the time constant, in this case 2.5e-5 s, resulted in forcing the cantilever for roughly 90% of the time associated with the initial displacement. Simulations were run with the default maximum number of steps, 1,000, at a step
time of $7.5 \times 10^{-7}$ s, which resulted in twelve oscillation cycles. The results are presented in Figure 6.8.

Using six oscillation cycles, the first natural frequency and the beam stiffness were within 0.2 % and 0.7 % of their respective values at twelve oscillation cycles. However, the quality factor changed 1.6 % as a result of fitting twelve rather than ten oscillation cycles, which suggests that more than twelve oscillation cycles are required to identify the quality factor under these conditions. Simulations involving more than twelve oscillation cycles were not explored because, as will be discussed in Section 6.5, it was not possible to run to transient responses of coupled cantilever-fluid systems in excess of 1,000 time steps with the available resources. In Chapters 7 and 8, research that would be enabled by these simulations will be proposed as future work.
6.4 Identifying Parameters from the Transient Solution

The simulated transient cantilever oscillations were used to identify dynamic properties of cantilever-fluid systems using a program based on the MATLAB `fminsearch` function, which is the function used in the software discussed in Chapters 4 and 5 to fit models to cantilever frequency response functions. The dynamic model consists of four SHOs formulated as a state space model. The transient response of the state space model to the forcing function (supplied by the user) is generated using the `lsim` command, and compared to response generated by the ANSYS simulation. The input parameters are adjusted to minimize the following cost function:

\[ J = \langle (q_{\text{ANSYS}} - q_{\text{SS}})^2 \rangle, \tag{6.2} \]

where \( q_{\text{ANSYS}} \) is the response generated in ANSYS and \( q_{\text{SS}} \) is the response of the state space model. Several initialization and fitting strategies were tested in an effort to reduce the tendency for two or more SHOs to be assigned similar natural frequencies, effectively splitting the motion of the first bending mode between multiple SHOs. In the strategy used in this research, the stiffness, natural frequency, and quality factor of the first resonance peak were initialized by the user, and two fitting steps adjusted the parameters to match the state space model output to the ANSYS results. In the first step, the second, third, and fourth SHO were assigned the same mass as the first SHO, and the stiffness values were scaled such that the ratios between natural frequencies were fixed according to beam theory. The quality factor of each SHO was allowed to vary independently. In the second fitting step, the mass, natural frequency, and quality factor of each SHO model was free to vary independently of the other SHOs. The fitting algorithm is presented in Section B.2.
6.5 Transient Responses of Coupled Fluid-Structure Models

Cantilever-fluid interaction simulations include two separate element meshes; one of the cantilever geometry, and one of the fluid geometry with boundary surfaces that define where the fluid and structure models interact. At each time step, the cantilever FEM is solved to generate a set of node displacements. The displacements at the interaction boundary are then used to generate displacement inputs for the fluid mesh. Following the displacement input, a CFD calculation is performed, yielding a set of reaction loads at the interaction boundary that are then used as discrete force inputs to include in the subsequent iteration of the structural FEM calculation. When the displacements and forces at the FEM/CFD interface boundary are sufficiently converged, the simulation moves on to the next time step. All of the resolution issues discussed in Sections 6.1, 6.2, and 6.3 apply. Additionally, the fluid mesh must be sufficiently detailed and more fine than the structural mesh at the fluid-structure boundary.

The mesh requirements at the fluid-structure boundary stem from forces that were passed from the fluid model to the cantilever model, while displacements were passed from the cantilever model to the fluid model. The fluid and cantilever meshes were generated by different algorithms and as a result, the nodes of the two meshes did not align at the interface. Two different types of interpolation were used to transfer information between the unaligned elements. To transfer displacements, a profile preserving interpolation was used. This transfers displacements to fluid mesh (receiving side) nodes from an interpolation of the cantilever (sending side) mesh faces. For displacement transfer, it is better for the receiving side to have smaller elements to preserve the detail of the sending mesh. To transfer loads, a globally
conservative interpolation was used. This sums the loads at each node on the sending side that is in contact with a specific face on the receiving side and assigns the total load to that face. For load transfer, it is better for the sending side to have smaller elements to avoid a situation where loading is unevenly distributed on the receiving structure. If the receiving side elements were smaller than the sending side elements, it would be possible for some receiving side element faces to not come in contact with any sending side nodes, resulting in no loading on the receiving side element.

The transient cantilever-fluid simulations were conducted on a Dell Precision T5500 Workstation. This computer had two, 4-core, 2.0 GHz processors and 6 Gbytes of memory. Preliminary testing revealed that the size of the fluid mesh that the computer could process would likely limit the degree of convergence that would be achieved, therefore, properties of the simulation were designed with the goal of reducing the computational demands wherever possible. The cantilever simulated in the transient fluid-structure simulations was identical to the cantilever simulated in Sections 6.1, 6.2, and 6.3, except that it did not have a probe tip. Omitting the probe tip also maintained conditions similar to Sader’s hydrodynamic loading model [36]. The cantilever mesh was reduced to 773 nodes. The fluid environment was cylindrical and all walls were at least 100 µm from the cantilever. The model was cut in half and symmetry rules were applied to reduce the required computational load as shown by the example fluid mesh presented in Figure 6.9.

The fluid mesh elements were scaled in a way that increased the level of detail near the cantilever beam. Element faces that were in contact with the top or bottom surfaces of the cantilever were assigned sizing limits, and the growth rate in element size between adjacent elements was also assigned a numerical limit. A detail view of the fluid mesh where it comes into contact with the fluid-cantilever interface is presented in Figure 6.10. For reasons discussed in Chapter 7, it is beneficial to
Material properties were selected such that the expected parameters (determined by curve fitting an SHO to an analytical FSI model [54] of the cantilever dynamics) were $k = 0.15 \text{ N/m}$, $Q = 5.9$, and $f_n = 41.55 \text{ kHz}$. Several rounds of simulations were run with increasing fluid mesh detail and decreasing numbers of time steps.
Table 6.1: Comparison of stiffness, quality factor, and natural frequencies estimations for the first modes of transient responses involving three different fluid meshes.

<table>
<thead>
<tr>
<th>Mesh No.</th>
<th>nodes (millions)</th>
<th>time steps</th>
<th>$k$ (N/m)</th>
<th>$Q$</th>
<th>$f_n$ (kHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5.1</td>
<td>200</td>
<td>0.154</td>
<td>7.07</td>
<td>42.28</td>
</tr>
<tr>
<td>2</td>
<td>6.4</td>
<td>200</td>
<td>0.154</td>
<td>7.27</td>
<td>42.31</td>
</tr>
<tr>
<td>1</td>
<td>5.1</td>
<td>79</td>
<td>0.154</td>
<td>7.33</td>
<td>42.28</td>
</tr>
<tr>
<td>2</td>
<td>6.4</td>
<td>79</td>
<td>0.154</td>
<td>7.66</td>
<td>42.28</td>
</tr>
<tr>
<td>3</td>
<td>11.9</td>
<td>79</td>
<td>0.154</td>
<td>6.56</td>
<td>42.14</td>
</tr>
</tbody>
</table>

The results from the three fluid meshes with the highest number of nodes are presented in Table 6.1. Mesh one contained 5.1 million nodes and took 33.5 h. Mesh two had the same element size restrictions on the cantilever-fluid boundary, but the growth rate was reduced from 1.04 to 1.035, which resulted in 6.4 million nodes and a 41.5 h simulation. For mesh three, the face sizing on the cantilever-fluid boundary was reduced from 0.75 $\mu$m to 0.50 $\mu$m. This resulted in 11.9 million nodes and a simulation that crashed after nineteen days. While the simulations involving meshes one and two completed their 200 time steps, mesh three only completed 79 time steps. In terms of oscillations, these simulations only completed roughly 1.25 and 0.5 oscillations, which is not enough to yield converged solutions based on the results presented in Figure 6.8, but is useful in comparing the three meshes. A 20% increase in the number of nodes results in a 2.8% increase in the quality factor, estimated by 200 time step simulations, and a 3.9% increase when estimated by 79 time steps. Roughly doubling the number of nodes resulted in a drop in the quality factor by one-sixth.

Chapter 7 explores the possibility of using FEM-CFD simulations to create an estimation of the hydrodynamic function for specific geometries. This function could be used to perform cantilever calibrations under heavy fluid load, however, simulating the fluid loading with sufficient detail to create a useful estimation of the hydrody-
namic function would take more computational resources than are available in this research project. For that reason, discussions on what could be possible using these techniques are presented as proposals of future work.

6.6 Summary and Conclusions

In this chapter, FEM and CFD techniques are presented that are used in Chapters 7 and 8. Simulations involving only the cantilever structure, including static loading and modal analyses, display stiffnesses and natural frequencies that converge as the FEM mesh is refined.

The analysis of simulated transient responses could prove to be a useful approach in the study of cantilever-fluid systems. A system identification technique was demonstrated that estimated the stiffness, quality factor, and natural frequency for cantilever-fluid systems based on a transient response. The simulation of a damped cantilever (without a coupled CFD model) that produced oscillations exhibiting a quality factor around 75, revealed a change in the estimated first mode quality factor of 1.6 % going from ten to twelve oscillations. Coupled FEM-CFD simulations of transients that completed less than one oscillation, resulted in quality factor estimates that dropped from 7.66 to 6.56 when the fluid mesh was increased from 6.4 million nodes to 11.9 million nodes. The number of nodes of the fluid mesh and the number of time steps that could be simulated were limited by the computational resources available. With greater computational resources, future research could use these techniques to produce parameter estimates that characterize fluid loading with sufficient convergence to be useful in the study of cantilever calibration in fluid environments.
Chapter 7

V-Shaped Cantilevers

In addition to rectangular AFM cantilever beams, many researchers use V-shaped cantilevers. V-shaped cantilevers have two beams, or legs, that extend from the chip at an angle that meet to form a single structure. If a plan view schematic of a V-shaped cantilever is oriented so that the base of the cantilever is at the top of the schematic, the shape of the cantilever resembles the letter ‘V’, as illustrated in Figure 7.1.

The V-shaped cantilever was initially designed in an effort to reduce lateral twisting of the cantilever induced during scanning, while maintaining normal compliance [62, 37]. Upon investigating the issue, Sader discovered that V-shaped cantilevers were more compliant torsionally than rectangular cantilevers [27]. Analytical formulations of the ratios of the lateral stiffnesses of V-shaped and rectangular cantilevers with identical normal stiffnesses reveal this to be the case for lateral loading in both directions along and across the cantilever axis, over a range of possible V-shape geometries. Additionally, higher torsional susceptibility of V-shaped cantilevers has been demonstrated experimentally, using macroscale models [63].

It is a fundamental principle of measurement that maximizing sensitivity to the property being measured while minimizing sensitivity to everything else is desirable. For this reason, V-shaped cantilevers may be better suited for experiments that involve detecting lateral forces rather than normal forces. Therefore, motivation to advance normal stiffness calibration methods is less for V-shaped cantilevers than for rectangular cantilevers. It is also important to promote awareness among AFM users regarding proper sensor selection for their intended experiments.
While normal stiffness calibration techniques for rectangular cantilevers warrant more emphasis than techniques for V-shaped cantilevers, there is still value to pursuing techniques for V-shaped cantilevers. The ability to monitor both lateral and normal forces simultaneously creates additional research possibilities. In this chapter, two strategies for the normal stiffness calibration of V-shaped cantilevers are discussed. The first involves applying the modeling strategies employed in Chapter 4 to the thermal noise method. The second strategy involves the use of the FEM and CFD methods from Chapter 6 to create the necessary hydrodynamic function required to use the calibration method for Chapter 5 and is suggested as future work.

7.1 System Dynamics and Noise Models

In Chapter 4, thermal noise calibrations were performed on a set of rectangular cantilevers in both air and in DI water using a variety of models to approximate both the cantilever-fluid system dynamics and the ambient system noise. For a set of six
Table 7.1: The stiffness values and percent differences between calibrations performed in air and in water using two different fitting models: the SHO model in combination with white noise and the high-pass filtered Lorentzian model in combination with a fit-slope noise source. The stiffness values are in N/m and the percent differences were determined according to Equation 4.14

<table>
<thead>
<tr>
<th>Cantilever</th>
<th>$k_{\text{air}}$</th>
<th>$k_{\text{SHO,white}}$</th>
<th>$PE$</th>
<th>$k_{\text{Lor,fit-slope}}$</th>
<th>$PE$</th>
</tr>
</thead>
<tbody>
<tr>
<td>MLCT D</td>
<td>0.0477</td>
<td>0.0524</td>
<td>9.8</td>
<td>0.0433</td>
<td>-9.2</td>
</tr>
<tr>
<td>MLCT E</td>
<td>0.178</td>
<td>0.188</td>
<td>5.6</td>
<td>0.174</td>
<td>-2.2</td>
</tr>
<tr>
<td>MLCT F</td>
<td>0.936</td>
<td>0.799</td>
<td>-14.6</td>
<td>0.795</td>
<td>-15.1</td>
</tr>
</tbody>
</table>

rectangular cantilevers, the difference in calibrations performed in the two fluids was reduced significantly by changing the model used for curve fitting. The commonly used combination of an SHO model with white noise resulted in a maximum difference of 20.2 %. Using a Lorentzian model with noise that could be curve fit by changing the slope of the frequency roll-off reduced the maximum difference between air and water calibrations to 9.5 %. The same techniques were applied to four V-shaped cantilevers, Veeco MLCT C through F. Cantilever C was damaged during handling. As was the case with the rectangular cantilevers, calibrations performed on V-shaped cantilevers in air using different dynamics models were nearly identical. The results are presented in Table 7.1.

Changing the theoretical model used to implement the thermal noise method did not produce a significant reduction in the difference between calibrations performed in air and in water. The maximum difference between calibrations performed in air and in DI water actually increased from 14.6 % to 15.1 %. The success in the case of rectangular cantilevers but not V-shaped cantilevers could suggest that the hydrodynamic loading function is significantly different for the two geometries. In Section 7.2, a strategy is proposed to calculate the hydrodynamic loading function for cantilevers of arbitrary shape.
7.2 Noncontact Calibration for Arbitrary Geometries under Heavy Fluid Loading

The cantilever stiffness calibration technique presented in Chapter 5 requires knowledge of the mass loading and damping that the fluid environment contributes to the cantilever-fluid system. Sader identified a hydrodynamic function that approximates the fluid loading for rectangular cantilevers [36] and enables the use of the heavy fluid loading calibration technique. If the hydrodynamic function could be identified for V-shaped cantilever geometries, the technique presented in Chapter 5 could be applied to V-shaped cantilevers. Sader et al. have also presented an experimental method for measuring the hydrodynamic functions for cantilevers of arbitrary geometry provided the cantilever stiffness is a known entity. Experimental limitations prevent this technique from characterizing the hydrodynamic function over the range of Reynolds numbers required for heavy fluid loading calibrations, however, coupled FEM and CFD analysis offer a potential alternative. This technique is proposed as future research. Provided sufficient computational resources, this technique could enable the noncontact stiffness calibration of V-shaped cantilevers in heavy fluid loading.

7.2.1 Measuring the Hydrodynamic Function of Arbitrary Cantilever Geometries

Using dimensional analysis, Sader et al. identified a dimensionless function relating the energy dissipated per cycle of an oscillating structure to its Reynolds number through an unknown hydrodynamic loading function. This function can be expressed as:

\[ \Omega'(Re) = \frac{k}{\rho_f L_0^3 \omega_r^2 Q}, \]  

(7.1)
where $L_0$ is an arbitrary length scale describing the oscillator. Sader et al. compared this expression to their rectangular hydrodynamic function which yields the following calibration expression [17]:

$$k = 0.1906 \rho f b^2 \Gamma_i(\omega_r) \omega_r^2 Q,$$

where $\Gamma_i(\omega_r)$ is the imaginary part of the hydrodynamic function discussed in Section 3.4. Selecting $L_0 = b/2$ and substituting 7.3 into 7.1 yields:

$$\Omega'(Re) = 1.525 \frac{l}{b} \Gamma_i(\omega_r).$$

This result reveals that the hydrodynamic function for arbitrary geometry is related to the hydrodynamic function for rectangular cantilevers by a constant function of geometry. Since the heavy fluid loading technique presented in Chapter 5 does not require that data be scaled accurately (i.e., it is based on fitting the shape of the PSD of cantilever thermal vibration) $\Omega'(Re)$ could be used as a substitute function for $\Gamma_i$.

To identify the hydrodynamic function experimentally, Sader et al. measured the thermal noise spectrum for calibrated cantilevers, and determined the resonance frequency and the quality factor by fitting an SHO model to the Fourier transform of the thermal noise. This procedure identifies the hydrodynamic function at one Reynolds number. To evaluate the hydrodynamic function at other Reynolds numbers, adjustments were made to the density of the fluid. The AFM was placed in a bell jar and the fluid density was reduced by lowering the pressure. The hydrodynamic function was also determined at one higher Reynolds number by filling the bell jar with ambient pressure carbon dioxide, which has a lower viscosity than air.

Creating a hydrodynamic function appropriate for use under heavy fluid loading conditions requires exploring cantilever behavior at higher Reynolds numbers. Theoretically, this could be achieved by observing the thermal fluctuations of cantilevers
in various fluids, however, under heavy fluid loading, cantilever motion tends to be heavily damped. In the work presented in Chapter 4, SHO models were observed to result in poor fits to heavily damped cantilever resonances as determined by the cost functions used to perform the fittings. The Chapter 4 analyses involved models and measured thermal vibration presented as PSDs. Therefore, the thermal vibration curve fittings involved functions of frequency while the transient responses were curve fit as functions of time. Figure 7.2 presents a simulated transient response of the cantilever discussed in Chapter 4 enveloped in air. The displacement is dominated by the first mode, which yields a close fit to the total response. The second mode oscillates at a much higher frequency which can be seen in Figure 7.3. The theoretical ratio between the first and second bending mode frequencies under no damping is approximately 6.25. The third and fourth modes, at even higher frequencies and higher modal stiffnesses, contribute very little to the total cantilever motion. A simulated transient from the same cantilever enveloped in water is presented in Figure 7.4. The transient response of a cantilever-fluid system with heavily damped first mode cannot be approximated well by an SHO model. In this case, the frequencies of the higher modes were lowered by the curve fitting algorithm until they were near the frequency of the first mode. Changes to the modal stiffnesses from the air simulation to the water simulation suggest that the higher frequency modes were being used to reshape the response of the first bending mode rather than to describe higher modes. To avoid this situation, cantilever-fluid system transients can be simulated that exhibit both high quality factors and high Reynolds numbers.

To produce the required conditions, ANSYS was used to simulate the transient response of cantilever-fluid systems using fictional cantilever and fluid material properties. These simulations were part of the effort described in Chapter 6 aimed at identifying the simulation parameters required for the results to converge. The FSI
Figure 7.2: Multiple SHO models fit to a simulated cantilever-air transient response. Modes 3 and 4 are included to show that their magnitudes are small in relation to the first mode.

Figure 7.3: Modes 2, 3, and 4 from the results presented in Figure 7.2. Modes 3 and 4 are included to show that their magnitudes are small in relation to the second mode.

model was used to predict the dynamic characteristics of rectangular cantilevers in fictional fluids and generate expected results for the analysis of the transient responses. For example, the rectangular cantilever model described in Chapter 4 was modified such that its Young’s modulus was 1,500 GPa and it was enveloped in a fictional fluid with a density of 10 kg/m³ and a viscosity of $10^{-4}$ kg/ms. The simulation ran for
Figure 7.4: Multiple SHO models fit to a simulated cantilever-water transient response.

roughly 1.3 oscillation cycles, which is insufficient for convergence, with a fluid mesh containing 3.1 million nodes, which is also insufficient for convergence. The computational resources required to run simulations to a sufficient degree of convergence were not available as explained in Chapter 4. The parameters estimated by fitting the FSI model and the ANSYS transient are compared in Table 7.2. The value of $\Omega'$ generated by analyzing the ANSYS simulation was 21.7% lower than the expected value.

With sufficient computational resources, these experiments could be conducted at higher resolutions and for higher oscillation counts. With satisfactory convergence, $\Omega'$ functions could be generated for both rectangular cantilevers, V-shaped cantilevers, or for cantilevers of other shapes. For rectangular cantilevers, the resulting $\Omega'$ function may offer an improvement over the hydrodynamic function currently used to describe the fluid loading since the current model does not allow for fluid motion along the length of the beam or around the cantilever end. While the validation of these methods presents the same challenges as other new calibration techniques, this technique could be used to enable the use of the noncontact calibrations meth-
ods presented in Chapter 5 for arbitrary cantilever geometries, including V-shaped cantilevers.

**Table 7.2**: Comparison of cantilever-fluid system parameter estimations based on the FSI model and on the analysis of an ANSYS transient response simulation.

<table>
<thead>
<tr>
<th>parameter</th>
<th>FSI model</th>
<th>ANSYS simulation</th>
</tr>
</thead>
<tbody>
<tr>
<td>$k$ (N/m)</td>
<td>0.150</td>
<td>0.154</td>
</tr>
<tr>
<td>$Q$</td>
<td>5.81</td>
<td>7.27</td>
</tr>
<tr>
<td>$f_n$ (Hz)</td>
<td>41,200</td>
<td>42,300</td>
</tr>
<tr>
<td>Re</td>
<td>2.61</td>
<td>2.66</td>
</tr>
<tr>
<td>$\Omega'$</td>
<td>37.3</td>
<td>29.2</td>
</tr>
</tbody>
</table>

7.3 **Summary and Conclusions**

In this chapter, the modeling techniques presented in Chapter 4 were applied to V-shaped cantilevers. For rectangular cantilevers, performing thermal noise calibrations in both air and DI water on a set of six cantilevers using white noise and an SHO model to curve fit the thermal vibrations resulted in stiffness estimates that varied by as much as 20.2 %. Using a Lorentzian in combination with a noise model that allowed the slope of the noise to vary improved the maximum difference between air and water calibrations to 9.5 %. Using this same technique on V-shaped cantilevers yielded no improvement. The largest difference in air and water calibrations increased from 14.6 % to 15.1 % by changing to the new modeling technique.

A potential explanation for why this strategy would be successful for rectangular geometry but not for V-shaped cantilevers could be that the hydrodynamic function for V-shaped cantilevers is dramatically different than the hydrodynamic function for rectangular cantilevers. A means of investigating this scenario has been proposed in this chapter as future work. A technique was demonstrated that could enable the estimation of an empirical hydrodynamic function for cantilevers of arbitrary
geometry based on FEM/CFD simulations. An estimation of the hydrodynamic function for V-shaped cantilevers could be used to perform thermal noise calibrations or it could enable the use of the heavy fluid loading calibration technique presented in Chapter 5.
Chapter 8

Warped Cantilevers

AFM cantilevers are manufactured through photolithographic techniques and are commonly coated on one or both sides to increase reflectivity or some other desired property. The cantilever coating processes can subject cantilevers to stresses that induce bending or twisting. AFM manufacturers have worked to minimize such warping and are able to offer cantilevers with specified maximum warping as low as 2°. In this section, FEM is used to explore how such bending and twisting impacts the stiffness and the calibration of AFM cantilevers.

Cantilever irregularities can result in changes to both the normal cantilever stiffness and the effective cantilever stiffness. When the impact on the normal and the effective cantilever stiffnesses are different, the differences can lead to error in force measurements. The presence of a probe tip complicates matters by altering the loading conditions. For this reason, ANSYS simulations were run both with and without a probe tip to evaluate the effects of bending and twisting on cantilever stiffnesses.

8.1 Impact on Stiffness and Natural Frequency

Using the techniques described in Chapter 6, a series of static displacement simulations were run to determine how bending or twisting a cantilever impacts the stiffness. The modeled cantilever was 200 μm by 20 μm by 0.543 μm. Simulations were conducted both with and without a 5 μm probe tip positioned at the end of the cantilever as illustrated in Figure 8.1. Loads were applied both normal to the top surface of the cantilever and at a 12° angle to the normal to identify both the
Figure 8.1: Detail of the cantilever tip modeled for static loading simulations of twisted cantilever; a) side view, b) bottom view.

Figure 8.2: AFM systems generally hold cantilevers at an angle to a substrate, resulting in an effective stiffness, $k_{\text{eff}}$, in the direction of the motion of the AFM stage, which varies from the normal stiffness of the cantilever, $k$.

Normal stiffness and the effective stiffness that would be observed in a typical AFM system. The FEM meshes had over 20,000 elements to ensure convergence, based on the results of Chapter 6.

Nominal values were established by simulating cantilevers without any bending or twisting. The normal stiffness of a flat cantilever, with or without a tip, was $k = 0.02014 \text{ N/m}$. It is also important to consider the effective stiffness of a cantilever, which is illustrated in Figure 8.2. Loading a cantilever and measuring the
resulting displacement at a $12^\circ$ angle resulted in an effective cantilever stiffness of $k_{eff} = 0.02104 \text{ N/m}$, which is within rounding error of the expected $k_{eff}$ presented in Equation 8.1 [61]. Adding the 5 $\mu$m probe tip to the ANSYS model increased the effective stiffness to $k_{eff} = 0.02137 \text{ N/m}$, which is 2.1 % higher than the value predicted by theory. If a user were to calibrate a cantilever using a technique that identifies the normal stiffness before conducting quasistatic force measurements, this difference would result in a 2.1 % underestimation of measured forces.

$$k_{eff} = \frac{k}{\cos^2 \theta} \left( \frac{1 - \frac{2d}{l} \tan \theta}{1 - \frac{3d}{2l} \tan \theta} \right)^2. \tag{8.1}$$

A series of cantilevers were simulated with various degrees of bending or twisting, both with and without probe tips. The changes to the frequencies of the first bending and torsional modes for selected cantilevers are presented in Table 8.1. The stiffnesses and effective stiffnesses were normalized by the nominal normal stiffness according to Equation 6.1. The normalized stiffnesses are presented in Table 8.2. Twisting the cantilever had very little effect on the stiffness or the dynamics of the cantilever. A $20^\circ$ twist caused a 1.1 % increase in the normal stiffness and a 3.4 % increase in the natural frequency of the first torsional mode. Bending had a bigger effect on the cantilever parameters, in particular, the frequency of the first torsional mode. A $20^\circ$ bend reduced in frequency of the first torsional mode by nearly half and changed the mode frequency order. The first torsional mode of a flat cantilever is higher in frequency than the third bending mode. For cantilevers with $10^\circ$ or $20^\circ$ of bending, the first torsional mode frequency falls in between the second and third bending mode frequencies.

Potential calibration errors stemming from the changes in normal and effective stiffnesses of bent and twisted cantilevers can be estimated by applying the correction
Table 8.1: The percent changes in the first bending and first twisting mode frequencies resulting from the bending and twisting of cantilever beams.

<table>
<thead>
<tr>
<th>cantilever condition</th>
<th>$\Delta \omega_{\text{bend}}$ (%)</th>
<th>$\Delta \omega_{\text{twist}}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10° bend</td>
<td>0.1</td>
<td>-24.7</td>
</tr>
<tr>
<td>20° bend</td>
<td>0.3</td>
<td>-49.7</td>
</tr>
<tr>
<td>10° twist</td>
<td>-0.02</td>
<td>0.8</td>
</tr>
<tr>
<td>20° twist</td>
<td>-0.1</td>
<td>3.4</td>
</tr>
</tbody>
</table>

Table 8.2: The normalized stiffness values resulting from the bending and twisting of cantilever beams.

<table>
<thead>
<tr>
<th>cantilever condition</th>
<th>probe tip</th>
<th>$\hat{k}$</th>
<th>$\hat{k}_{\text{eff}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0° bend</td>
<td>yes</td>
<td>1.000</td>
<td>1.061</td>
</tr>
<tr>
<td>4° bend</td>
<td>yes</td>
<td>0.997</td>
<td>1.040</td>
</tr>
<tr>
<td>10° bend</td>
<td>yes</td>
<td>1.001</td>
<td>1.014</td>
</tr>
<tr>
<td>20° bend</td>
<td>yes</td>
<td>1.032</td>
<td>0.998</td>
</tr>
<tr>
<td>0° bend</td>
<td>no</td>
<td>1.000</td>
<td>1.044</td>
</tr>
<tr>
<td>4° bend</td>
<td>no</td>
<td>1.002</td>
<td>1.028</td>
</tr>
<tr>
<td>10° bend</td>
<td>no</td>
<td>1.014</td>
<td>1.012</td>
</tr>
<tr>
<td>20° bend</td>
<td>no</td>
<td>1.058</td>
<td>1.012</td>
</tr>
<tr>
<td>10° twist</td>
<td>yes</td>
<td>1.000</td>
<td>1.065</td>
</tr>
<tr>
<td>20° twist</td>
<td>yes</td>
<td>1.011</td>
<td>1.074</td>
</tr>
</tbody>
</table>
in Equation 8.1 to the normal stiffness values, and comparing the results to the effective stiffnesses of the simulated cantilevers. When performing these adjustments for the nominal, flat cantilever with a 5 µm probe tip and a 12° loading angle, the error was found to be 2.1 % as discussed previously. For the twisted cantilevers, adjusting the \( k \) values for cantilevers with twists of 10° and 20° yields \( k_{eff} \) values of 1.040 N/m and 1.051 N/m, respectively. Comparing these results to the effective stiffnesses in Table 8.2 reveals errors of 2.4 % and 2.2 %, respectively. Subtracting the error of 2.1 % for the nominal case leaves errors of 0.3 % for a 10° twist and 0.1 % for a 20° twist, which can be attributed to the twisting. These results suggest that the geometrical effects of cantilever twisting as high as 20° does not impact stiffness calibrations as much as the presence of a 5 µm probe tip.

\[
\hat{k}(\phi) = \frac{k}{k_{eff} \cos^2 \phi} \left( \frac{1 - \frac{2d}{l} \tan \phi}{1 - \frac{3d}{\pi l} \tan \phi} \right)^2. \tag{8.2}
\]

For small amounts of warping, cantilever bending has more of an impact on stiffness than twisting does. To investigate the geometric calibration errors stemming from cantilever bending, the normal and the effective stiffnesses were compared using the normalization presented in Equation 8.2. The normalized stiffnesses are presented in Figure 8.3 for two correction methods. Correcting the stiffness for the cantilever base angle of \( \phi = 12° \) reveals that the error steadily increases at a rate of 0.5 % error per 1° of cantilever bending. Correcting the stiffness for the cantilever angle at the tip, \( \phi = 12° - \theta_b \) (where \( \theta_b \) is the degree of cantilever bending), reveals error less than 1 % for bending that does not exceed 10°. The second correction, where the cantilever end angle is used to correct for the effective stiffness, is generally unavailable to an AFM user since the degree of cantilever bending is unknown. Using the base angle to calculate the effective stiffness results in an error that roughly cancels out
the 2.1% error associated with the probe tip for angles around 5°.

8.2 Static and Dynamic Deflection Shapes

As discussed in Section A.3, the relationship between the angle of the free end of a cantilever and the displacement of the end of a cantilever is different for static or dynamic deflections. Since the cantilever position is often monitored by observing a laser beam reflected off the end of the cantilever, a correction is needed to relate dynamic displacements to static displacements, as is the case when the thermal noise method is used to calibrate a cantilever before conducting quasistatic pulling experiments. The correction factor is predicted by theory to be $\chi = 1.0897$ [19]. Static loading and modal analysis was conducted to establish the ratio between the cantilever end slope and the end position. The models were validated by simulating a cantilever beam with no twisting. Measuring the slopes and displacements of the simulations resulted in $\chi = 1.0897$. 

Figure 8.3: Normalized cantilever stiffnesses for warped cantilevers of various bending angles.
To evaluate the impact of twisting and bending on the $\chi$ factor, extreme cases were modeled that resulted in angles of $20^\circ$ at the free ends of the cantilevers. Side and end views of $20^\circ$ twisted and bent cantilevers are presented in Figures 8.4 and 8.5. Calculating $\chi$ for a beam with a $20^\circ$ twist resulted in $\chi = 1.0891$. Since the model produced a change in $\chi$ for a cantilever a $20^\circ$ twist of only $0.05\%$, then the impact to cantilevers with twists on the order of $0-4^\circ$ would be negligible in comparison to other sources of uncertainty. This procedure was repeated for a bent cantilever with a $20^\circ$ angle at the end, yielding $\chi = 1.0917$, a change of $0.2\%$ from that of a flat beam.
8.3 Fluid Interaction with Warped Cantilevers

Potential calibration error could also stem from the interaction of warped cantilever beams with fluid environments. A change in geometry would yield a change in the hydrodynamic loading function, an effect that could be explored through FEM-CFD simulations as proposed for V-shaped cantilevers in Chapter 7. Using the techniques presented in Chapter 6, transient fluid-cantilever simulations could be used to identify system properties at distinct Reynolds numbers which could be used to create an empirical hydrodynamic function. Due to limited computational resources, this work is therefore proposed as future research.

8.4 Summary and Conclusions

In this chapter, FEM methods were used to investigate how cantilever warping impacts stiffness and calibration. AFM cantilevers are commercially available with a maximum of 2° bending specified. Loads were applied normal to cantilevers and at a 12° angle to evaluate changes in normal stiffnesses and effective stiffnesses. For tipless cantilevers, ANSYS calculations agreed with theory to within 0.1 %. Simulating a 5 µm tip at the end of a 200 µm cantilever did not change the normal stiffness, however, the effective stiffness was increased 2.1 % above theory. Based on this finding, further investigation into the effect of AFM probe tips on effective stiffnesses is warranted.

The warping of cantilevers can impact calibration if the normal and effective stiffnesses scale differently as a result of the warping. Twisting on the order of 10° would yield changes to the normal and effective stiffnesses resulting in a 0.3 % calibration error. Bending produces a calibration error of roughly 0.5 % per 1° of bending.

The warping of cantilevers also has the potential to impact calibration by changing
the $\chi$ value, which relates the ratio of cantilever slope and position for static and dynamic displacements. Twisting and bending a cantilever beam by 20° changes the $\chi$ value by 0.05 % and 0.2 %, respectively.

Changes to the hydrodynamic function also have the potential to impact AFM calibration. Methods to determine the hydrodynamic functions for arbitrary geometries were presented in Chapters 6 and 7, however, due to the computational resources required, this research is proposed as future work.
Chapter 9

Conclusions

Atomic force microscopy is a conceptually direct way to make force measurements subject to a significant number of complicating factors that make improvements in accuracy an elusive target. After over twenty years of development, knowledge of SI-traceable accuracy in AFM experiments has only been available to a small number of researchers, primarily through government research specifically dealing with the subject of AFM accuracy. Several calibration techniques have been developed through the years with a variety of strengths and weaknesses. Comparisons between cantilever stiffness values performed using the most popular techniques typically differ by about 10–20 %. Variation has also been observed between calibrations performed on the same cantilevers in air and in water. In deciding what method to use to calibrate an AFM cantilever, a researcher must weigh the reported stiffness comparisons of various techniques, the limitations and difficulty of implementing each technique, and the desired degree of measurement uncertainty.

The new techniques presented in this research expand the set of options available to a researcher conducting tests in liquid environments. In the design of the techniques, emphasis was placed on using only parameters and measurements that are easily accessible to researchers with typical AFM systems or would be available to someone designing a lab-on-a-chip.
9.1 Contributions to the Field

The research presented demonstrated new techniques that enable the calibration of AFM cantilevers under heavy fluid loading using the thermal vibration of the cantilever. This work resulted in the following specific contributions to the field of AFM calibration:

- Demonstrated that the use of alternate cantilever-fluid system dynamics and noise models reduce the difference between calibrations of the same cantilever in air and in water using the thermal noise method
- Presented a new noncontact stiffness calibration technique specifically designed for use in heavy fluid loading conditions
- Combined the new heavy fluid loading technique with the modified thermal noise method to perform noncontact stiffness and displacement calibrations in water
- Demonstrated through simulation that the geometric effects of cantilever warping do not impact calibration significantly, relative to current calibration uncertainty

9.1.1 Fitting Models and the Thermal Noise Method

The thermal noise method is one of the most popular methods for calibrating cantilevers, but calibrations performed on the same cantilever in air and in water can vary by as much as 20%. Implementation of the thermal noise method commonly involves fitting a model of the cantilever-fluid system to the thermal noise to reduce the impact of system noise. Thermal noise calibrations were performed on a set of six cantilevers by the common technique of using an SHO model to estimate the
dynamics of the cantilever-fluid system and white noise to estimate the system noise. The calibrations were performed in air and in DI water resulting in a maximum difference of 20.2%. The calibrations were repeated using a Lorentzian to estimate the cantilever-fluid system dynamics and a variable-slope noise model, which reduced the maximum difference between air and water calibrations to 9.5%.

9.1.2 Stiffness Calibration under Heavy Fluid Loading

Sader's calibration method enables the calibration of rectangular cantilevers based on the thermal response of a cantilever and knowledge of the length and width of the cantilever and the density and viscosity of the fluid. Since Sader's method is based on dynamic properties of the thermal response other than magnitude, it does not require the cantilever displacements to be calibrated, therefore, it can be performed using only noncontact data. Similarly, the new technique presented in this research does not require calibrated magnitudes and requires knowledge of the same cantilever and fluid parameters as Sader's method. Calibrations performed on eight cantilevers with length-to-width ratios greater than 3.5 using Sader's method in air and the new technique in water varied by a maximum of 9.4%.

9.1.3 Noncontact Displacement Calibration under Heavy Fluid Loading

Since the thermal noise method relates the magnitude of thermal vibration to cantilever stiffness, if the stiffness can be identified through a noncontact method, the thermal noise method can then be used to perform a noncontact displacement calibration. Under light fluid loading, this can be done using Sader's method to calibrate the stiffness before employing the thermal noise method to calibrate displacement. Since the new heavy fluid loading technique is also a noncontact technique, it can be used
in a similar fashion to perform noncontact stiffness and displacement calibrations in liquid environments. The thermal noise method was implemented using Lorentzian and variable-slope noise models in combination with the new heavy fluid loading stiffness calibration technique to produce noncontact stiffness and displacement calibrations for a set of ten cantilevers. The noncontact displacement calibrations varied by a maximum of 11.3 % from calibrations involving contact loading. The stiffness and displacement calibration results were combined to reveal what would happen if the two methods were used to measure forces. Sader’s method, conducted in air, was combined with contact-based displacement calibrations, while the new heavy fluid loading method was combined with the alternate-model thermal noise method. For the set of ten cantilevers, the two sets of force measurements would have varied by a maximum of 5.0 %.

9.1.4 Cantilever Warping

FEM simulations were used to investigate the effects of bending and twisting on stiffness calibration. Both bending and twisting caused minimal changes to the $\chi$ factor used to correct for the difference between the shapes of cantilevers under quasistatic and dynamic deflection, which is needed to correct for calibrations based on thermal vibrations that are used for quasistatic loading experiments. Twisting produced little change to the normal and effective cantilever stiffnesses that would result in calibration error. Bending impacted normal and effective stiffnesses in a way that would introduce 0.5 % error per 1° of cantilever bending.
9.2 Recommendations and Future Work

9.2.1 Continued Focus on Reducing Uncertainty

The development of AFM calibration techniques have been, to a significant degree, efforts in improving consistency between calibration techniques as a way to better understand what factors impact calibration. Currently, the consistency between techniques sets a lower bound for the uncertainty involved in measurements. As SI-traceable methods advance and access to standards proliferate through the AFM community, researchers will be more able to quantify the uncertainty of AFM measurements and better able to evaluate the calibration techniques themselves.

In Chapter 8, FEM was used to investigate the impact of warping on cantilever stiffness and effective stiffness. Models were simulated both with and without probe tips. Simulations of a 200 µm long beam, with a 5 µm probe tip revealed a 2.1 % difference between the effective stiffness of the simulated cantilever and the effective stiffness predicted by theory. The effects of probe tips on effective cantilever stiffness are therefore one of the factors that warrants further attention.

In Chapter 5, the new heavy fluid loading technique was compared to Sader’s method and a noncontact InvOLS method was compared to the commonly used contact method. Stiffness calibrations on cantilevers with length-to-width ratios less than 3.5 varied significantly between the two methods. Presumably, the use of the hydrodynamic function, which assumes high length-to-width ratios, played a significant role in the variation between the calibrations for shorter cantilevers. If the entire set of cantilevers are considered, the largest difference between stiffness calibrations is 17.9 % and the largest difference between InvOLS calibrations is 11.3 %. However, even when the cantilevers violate the assumptions implicit to the use of the hydrodynamic function, forces measured using Sader’s method in combination
with InvOLS determined by contact-forced displacements vary by a maximum of 5.0 \% from forces measured using a combination of the noncontact heavy fluid loading calibration methods. The combination of calibration factors yielding an increase in consistency does not imply that there is an improvement in accuracy, but it does suggest that there is some element of bias that cancels between the two sets of calibration factors. Further research is warranted to identify the source of these biases.

### 9.2.2 Identifying the Hydrodynamic Function for Arbitrary Cantilever Shapes

Sader identified an empirical method for finding the hydrodynamic function for cantilevers of known stiffness and arbitrary geometry. Practical limitations restrict this method to Reynolds numbers that are too low to enable calibrations in liquid environments. Coupled FEM-CFD simulations can generate transient responses of cantilever-fluid systems. Techniques demonstrated in this research can be used to estimate the system parameters needed to calculate the empirical hydrodynamic function. By selecting fluid properties that do not exist in nature, cantilever-fluid systems can be simulated that yield high Reynolds numbers and high quality factor resonances needed for curve fitting. These methods could be used to create hydrodynamic functions that would enable the calibration of V-shaped cantilevers in liquid environments. Alternately, these techniques could be used to investigate the effects of cantilever warping on cantilever-fluid system dynamics.
Appendix A

Measurement Issues

The variety of AFM calibration techniques available to researchers have different strengths and weaknesses and require different considerations to account for geometrical or other factors that result in adjustments that must be made to account for the specific conditions of an experiment. Additionally, there are several good practices that a user should be aware of in calibrating cantilevers. While good summaries of some of the considerations an AFM user should be aware of exist [64], this section aims to offer specific calibration guidance that stems from experience with the Asylum Research MFP-3D along with descriptions and references to issues that researchers should be aware of in the event that data is exported and analyzed outside of the Asylum MFP-3D software package.

A.1 Virtual Deflection

For reasons unknown to Asylum, actuating the piezo impacts the photodiode measurement when the cantilever is not in contact with the substrate. To account for this, Asylum provides a method for calibrating the ‘virtual deflection.’ To open the window that enables virtual deflection calibration, select the Programming tab, then Start User Panel, then Spring Constant Tutor. The selection of this option brings up the Spring Constant Tutor user panel and an illustrated Asylum guide that explains the procedure for performing a thermal noise calibration. To check for this phenomenon in other AFM systems, the Z-stage should be cycled through its entire range while the cantilever tip is far from any substrate.
A.2 InvOLS

Calibrating a cantilever using the thermal noise method requires finding the inverse optical lever sensitivity (InvOLS). The process involves bringing a cantilever into contact with the piezoelectric stage and ramping the stage position, plotting the photodiode voltage against the stage position, and finding the slope of the plotted line. It is good practice to fit the slope of both the approach and retraction of the stage several times and average the results. In air the InvOLS values of the approach and the retraction are generally the same, but in water, there can be a significant difference. Additionally, the data used for fitting the InvOLS should be selected near the zero point of the photodiode where the sensitivity is most linear.

A.3 Accounting for Static and Dynamic Bending Shapes

The shape of a cantilever with a static load applied at the tip is different from the shape of a cantilever that is freely vibrating. Since the optical lever method of detecting cantilever deflection monitors the angle of the cantilever tip rather than the position, the relationship between the tip angle and the tip displacement must be considered in the interpretation of AFM data. The ratio of the slopes of the appropriate beam shapes are identified by Proksch et al., and the resulting correction factor is identified as $\chi = 1.09$ [19]. On the Asylum, $\chi$ is referred to as ‘Kappa’.

The issue concerning this correction value is that the $\chi$ factor is 1.09 in the limit of an infinitely small ‘laser’ spot positioned at the very end of a rectangular cantilever. (The ‘laser’ in the Asylum MFP-3D is actually a superluminescent diode.) Proksch et al. present $\chi$ curves for specific spot-size-to-cantilever-length ratios evaluated as a function of position. Since the spot-size-to-cantilever-length is an uncontrollable
parameter in many experiments, identifying the required $\chi$ value often requires evaluating the presented theory or trying to interpolate between curves. Figure A.1 presents the findings of Proksch et al. as a function of the percentage of the cantilever beam illuminated by the spot, but only under the condition that 80% of the laser illumination is reflected. This enables identification of the $\chi$ value for arbitrary cantilever lengths, provided the laser spot is positioned appropriately.

Figure A.1 presents $\chi$ as a function of the ratio of the laser spot size to the cantilever length, therefore, the size of the laser spot does not necessarily need to be known. The ratio can be identified by comparing arbitrary units, such as the number of pixels of the cantilever length and the size of the laser spot counted from a screen capture during testing. The spot-size-to-cantilever-length ratio describes the fraction of the cantilever length illuminated by the laser spot. The laser spot size should be measured along the length of the cantilever. The cantilever is held at an angle to the camera which scales dimensions differently along and across the cantilever. The entire laser spot size should be measured. While the testing conditions call for a portion of the spot to fall off the end of the cantilever, the entire spot size is considered in the formulation.

A screen-grab of the laser spot and cantilever can be copied to the clipboard using the command `arv_Clipboard()`. Then an image file can be created by pasting into Microsoft Paint and the National Institutes of Health program, ImageJ, can be used to find the ratio by setting the cantilever length equal to one and measuring the spot length. Doing it this way, the laser spot is compared to the effective cantilever length and the proper ratio is found.

To find the correct spot location, the laser spot must be positioned so that the resulting sum voltage on the photodetector is 80% of the maximum possible sum voltage. To achieve this, a user must first position the laser spot so that it is entirely
on the cantilever and alternate adjusting its position along the width and along the length of the beam until the maximum sum voltage is identified. Then the spot can be moved to so that part of the spot is lost off the end of the cantilever and the sum voltage is 80 % of the maximum. It is a good practice to reposition the spot along the width of the cantilever, finding the maximum sum voltage for that $X$-position ($X$ being the axis running along the length of the beam) as a way to re-center the spot at the cantilever end. The laser spot should be iteratively repositioned along the length to find $V_{\text{sum}} = 0.8 \times V_{\text{max}}$ and along the width to find the maximum for that $X$ position.

### A.4 Modal Energy Distribution

Equipartition theorem describes the time-averaged potential energy stored in a cantilever beam excited only by a thermal bath. Each modal degree of freedom is excited and the cantilever motion is the sum of the modes. It is common practice to transform the thermal vibration of a cantilever from a function of time to a function of frequency, and isolate the frequency range that is dominated by the motion of the first bending mode. This practice results in the omission of the contributions from higher frequency modes. This matter is addressed in [19] and is treated at length in [59]. In terms of the displacement and the normal stiffness of the end of a rectangular cantilever, the contribution of the first bending mode accounts for 97.1 % of the time-averaged potential energy stored in the beam. When performing a thermal noise calibration, this factor is taken into account in the MFP-3D software and does not require the attention of users unless the data is exported and analyzed outside of the MFP-3D software.
Figure A.1: \( \chi \) or “Kappa” factor relating the ratio of static to dynamic angular deflections for rectangular cantilevers when the laser spot is positioned at the end of the cantilever such that 80\% of the maximum reflected light is reflected.
A.5  Sader’s Method

It is also possible to perform a calibration using Sader’s method on the MFP-3D using the command `Print kSader(w, l, Q, f_r)`, where \( w \) is the cantilever width in meters, \( l \) is the cantilever length in meters, \( Q \) is the quality factor, and \( f_r \) is the resonance frequency in Hertz. The quality factor and resonance frequency can be identified by performing a thermal noise calibration. It is good practice to calibrate a cantilever using both methods as a means to check against errors that would impact one calibration method but not the other. For example, static charge between a substrate and a cantilever can result in additional, position sensitive forces that would impact the thermal noise method and Sader’s method differently.

### A.5.1 Cantilever Angle

One of the important differences between Sader’s method and the thermal noise method is that Sader’s method identifies the intrinsic stiffness of the beam, \( k \), while the thermal noise method identifies the effective stiffness, meaning the stiffness of the cantilever in the direction of the piezo-actuator motion. These values are different primarily because the cantilever is held at an angle to the substrate. To account for this, calibrations performed using Sader’s method must be converted to the effective cantilever stiffness through the following relation:

\[
    k_{\text{eff,angle}} = \frac{k}{\cos^2 \theta}
\]

where \( \theta \) is the tilt angle of the cantilever.

### A.5.2 Cantilever Tip Position

In addition to the cantilever angle issue, the intrinsic stiffness is different from the effective difference because the cantilever tip is usually not positioned at the end of
the cantilever, rather, it is a few µm from the end. For a rectangular cantilever, stiffness is a function of the length cubed, therefore, the position of the tip can be accounted for with the following relation:

\[ k_{\text{eff,t-pos}} = \frac{k}{\cos^2 \theta} \frac{l^3}{l_2^3} \]

where \( l \) is the length of the entire cantilever, and \( l_2 \) is the distance from the base of the cantilever to the tip location.

A.5.3 Cantilever Probe Tip Length

The length of the cantilever probe tip also impacts the loading of a cantilever as described by Hutter [61]. The correction factor to account for the probe tip height, \( d \), is:

\[ k_{\text{eff,t-len}} = k \left( \frac{1 - \frac{2d}{l} \tan \theta}{1 - \frac{3d}{2l} \tan \theta} \right)^2 \]

Measuring the probe tip length of a cantilever by optical microscope can be difficult since an objective with sufficient magnification will generally have a small focal length and depth of focus and the AFM chip has to be turned on its side, which limits how close the objective can get to the probe tip. Additionally, as discussed in Chapters 8 and 9, ANSYS simulations revealed a 2.1 % difference in the effective stiffnesses predicted by theory and by simulating a 5 µm probe tip on a 200 µm long cantilever. Further investigation into the effects of cantilever probe tips is proposed as future work.

Combining these three correction factors yields the following relation:

\[ k_{\text{eff}} = \frac{k}{\cos^2 \theta} \frac{l^3}{l_2^3} \left( \frac{1 - \frac{2d}{l} \tan \theta}{1 - \frac{3d}{2l} \tan \theta} \right)^2 \]
This is the stiffness estimation that should be compared to calibrations performed using the thermal method.

A.6 Testing in Liquids without a Fluid Cell

Testing in liquids with the Asylum MFP-3D can be difficult when capillary action draws the liquid droplet under the clamp that holds the cantilever. This can be avoided by placing a drop of liquid on the cantilever itself before putting the AFM head in position and one drop on the substrate, displaced slightly from the expected cantilever contact point roughly half a droplet width in the direction slightly opposite the cantilever clamp.

A.7 Leveling

Before any data is taken, after loading the cantilever and placing the AFM head in position, a bullseye bubble level should be placed on the the base of the AFM and on top of the AFM head and the three AFM head leg screws should be adjusted to position the cantilever near the substrate while keeping the AFM head parallel to the base. Regardless of the calibration method used, if forces are to be measured, the angle between the cantilever and the pushing or pulling direction must be known.
Appendix B

Code

In this section, three of the programs used to analyze measured and simulated cantilever data are presented. The first program uses the thermal response data of a cantilever under heavy fluid loading to estimate the stiffness. The second program uses state space models to estimate cantilever-fluid system parameters from transient responses simulated in ANSYS. The third program performs the thermal noise calibration using a Lorentzian to estimate cantilever-fluid system noise and a variable-slope noise model to account for the noise present in the measurement system.

B.1 Heavy fluid loading calibration

The heavy fluid loading calibration technique requires fitting the FSI model to data in a way that reduces the sensitivity of the result to the specific fitting parameter values while assuming cantilever data was produced under heavy fluid loading conditions. There are several potential ways that a researcher could do this. This section describes the details of the automated algorithm to facilitate its use by the AFM community.

B.1.1 User Inputs

Before the calibration can be performed, there are several input steps, the first of which, is loading the thermal response data of the unloaded cantilever. The data should be in the form of a '.mat' file containing two data vectors, 'freq', the values of the frequency bins in Hz and 'Syy', the values of the PSD of the random vibration of the free cantilever beam corresponding to those frequency bins. The cantilever
length and width must be provided, and these values can either be measured with an optical microscope or, when the manufacturing tolerances are small, the values provided by cantilever suppliers can be used. The fluid must be selected because the hydrodynamic model requires knowledge of the density and viscosity of the fluid. Room temperature fluid properties are assumed. Adding additional density and viscosity values to reflect additional fluids or different temperatures would require altering the MATLAB code, and should be a simple task for a programmer of modest ability.

There are two dynamics models used in the automated calibration process, an SHO and a fluid-structure interaction model. The initialization of the two dynamic models is achieved through two sets of slider bars with accompanying text boxes. The slider bars follow weighted exponential curves appropriate for the parameters which can vary over several orders of magnitude for the set of commercially available cantilever probes. It is important that the user line up the resonance peaks of the models and the data to prevent the curve fitting algorithm from selecting an incorrect local minimum. In most cases, the initial model fits can vary significantly from the data and the program will still converge, however, the better the initial fit is, the faster the program will run.

B.1.2 Data Range Selection

The FSI model considers only the motion corresponding to the first bending mode of the cantilever. The measured cantilever thermal response contains this data along with data representing any higher bending modes or twisting modes that were detected along with any electrical, mechanical, or acoustic noise that was present during testing. For this reason, the program has been written to select only the frequency range where the motion of the first bending mode dominates the signal. To quantify
the amount of the peak that has been selected, a ‘selection ratio’, \( R \), is calculated. This parameter is the ratio of the maximum value to the minimum value over the selected frequency range. For illustrative purposes, the frequency range covering what is commonly referred to as the full-width at half-maximum (FWHM) would have a selection ratio of two. In this implementation, the selection ratio is five, therefore the magnitude of the minimum signal included in the curve fitting is only 20 \% of the maximum. To avoid the impact of noise on the data selection process, the selection ratio is calculated for an SHO model fit to the measured data over the selected frequency range.

To achieve this, a nested set of cost functions are minimized by MATLAB’s \textsc{fminsearch} command. The two cost functions are as follows:

\[
J_R = (R - 5)^2 \quad (B.1)
\]

\[
J_{\text{SHO}} = \|S_{\text{SHO}} - S_{\text{data}}\|_2 \quad (B.2)
\]

where \( R \) is the selection ratio and \( S_{\text{SHO}} \) is the power spectral density of the SHO model. The frequency range selected for curve fitting extends above and below the resonance frequency a specific fraction of the resonance frequency determined by the parameter \( A \) (the range starts at \([1 - A] \omega_r\) and ends at \([1 + A] \omega_r\), where \( A \) is less than 1.) The nested search commands identify the frequency range to be used for curve fitting by adjusting the frequency selection parameter, \( A \), to minimize the selection ratio cost function, \( J_R \), while continuously refitting the SHO model to the selected data by adjusting the parameters \( P_{DC}, \omega_n, \) and \( Q \) to minimize the SHO fit cost function, \( J_{\text{SHO}} \).
B.1.3 Algorithm Execution

The calibration algorithm identifies a series of parameters that provides a good fit to a measured cantilever response. The search for each set of parameters is forced to progressively heavier fluid loading conditions until the resulting stiffness estimation converges. The search identifies points that lie along the low cost function locus for the FSI model fit. Points on the cost function map are identified by two parameters, a natural frequency, \( \omega_n \), and a mass loading ratio, \( \gamma \).

There is an additional parameter involved in the curve fitting that allows the magnitude of the FSI model to be rescaled. Without this rescaling parameter, the algorithm would have to fit the magnitude of the FSI model to the magnitude of the measured data as dictated by equipartition theorem. This additional requirement would restrict the range of initialization parameters that would result in convergence. More importantly, any error in the magnitude of the data (e.g., error in the photodiode calibration of a ‘laser lever’ style AFM) has a significant impact on the resulting parameter fit [48].

The initial search point is provided by the user by adjusting a set of slider bars to perform a rough curve fit. To move from these parameters to a point along the low cost function locus (which is the darker region in Figure B.1) an `FMINSEARCH` routine is called to minimize the FSI model fit cost function, \( J_{FSI} \), by adjusting \( \gamma \) and rescaling the data while holding the \( \omega_n \) value fixed. Using the the values for \( \omega_n \) and \( \gamma \) identified by this process, an estimation for the stiffness, \( k \), is established by the following relation:

\[
k = \frac{3m' \omega_n^2}{(\alpha l)^4 \gamma}.
\]  

To ‘walk’ along the low cost function locus, a second point is selected by increasing \( \omega_n \) and using the estimate for \( k \) along with Equation B.3 to solve for a new estimate
for $\gamma$ (see Figure B.1.) The new point is then treated similarly to the previous set of parameter estimations; the `FMINSEARCH` routine is called again to find the value of $\gamma$ that minimizes $J_{FSI}$ for the higher value of $\omega_n$ and a new estimate for $k$ is generated. The two $k$ estimates are then compared for convergence. This process repeats until $k$ converges according to:

$$ (k_{old} - k_{new})^2 < 1e - 10 \quad (B.4) $$

### B.1.4 Stiffness Initialization

In most cases, the algorithm execution as described in the previous section will successfully find points in the heavy fluid loading region where the resulting $k$ estimation
is not sensitive to parameter changes that remain within the low cost function locus. However, in some cases, if the model initialization occurs at a relatively light fluid loading point, the first ‘step’ is not sufficiently aligned with the low cost function locus and the resulting FSI model fit converges to a local minima outside of the low cost function locus (see Figure B.2.)

This possible misstep is avoided by providing a better stiffness estimate for the first step. Fortunately, with an SHO model fit already performed during the frequency range selection, all the necessary information required to perform Sader’s stiffness estimation method is available [17]. Sader’s method, while not intended for use in heavy fluid loading situations, is generally accurate to within 30%. This is accurate enough to prevent the search algorithm from stepping towards the undesired local minimum.
Figure B.2: Without providing an initial stiffness estimate, the first step of the search algorithm can converge to a local minima outside of the desired low cost function locus. A low initial estimate of the mass loading results in a poor initial stiffness estimate as indicated by the slope of the dashed constant stiffness line in A). In B), stepping to a new set of parameters based on this stiffness estimate yields a search that starts too far from the low cost function locus.
function varargout = calibrate_1_1(varargin)

% CALIBRATE_1_1 M-file for calibrate_1_1.fig
% This program is free software: you can redistribute it and/or modify
% it under the terms of the GNU General Public License as published by
% the Free Software Foundation, either version 3 of the License, or
% (at your option) any later version.
%
% This program is distributed in the hope that it will be useful,
% but WITHOUT ANY WARRANTY; without even the implied warranty of
% MERCHANTABILITY or FITNESS FOR A PARTICULAR PURPOSE. See the
% GNU General Public License for more details.
%
% You should have received a copy of the GNU General Public License
% along with this program. If not, see <http://www.gnu.org/licenses/>.

% CALIBRATE_1_1, by itself, creates a new CALIBRATE_1_1 or raises the existing
% singleton*.
%
% H = CALIBRATE_1_1 returns the handle to a new CALIBRATE_1_1 or the
% handle to the existing singleton*.
%
% % CALIBRATE_1_1(‘CALLBACK’,hObject,eventData,handles,...) calls the local
% % function named CALLBACK in CALIBRATE_1_1.M with the given input argu-

130
% CALIBRATE_1_1(’Property’,’Value’,...) creates a new CALIBRATE_1_1 or raises
% the existing singleton*. Starting from the left, property value pairs are
% applied to the GUI before calibrate_1_1_OpeningFcn gets called. An
% unrecognized property name or invalid value makes property application
% stop. All inputs are passed to calibrate_1_1_OpeningFcn via varargin.
%
% *See GUI Options on GUIDE’s Tools menu. Choose ”GUI allows only one
% instance to run (singleton)”.
%
% See also: GUIDE, GUIDATA, GUIHANDLES

% Edit the above text to modify the response to help calibrate_1_1

% Last Modified by GUIDE v2.5 08-Jan-2009 16:18:27
% calibrate_1_1 by Scott Kennedy, Duke University
%
% This program implements two AFM cantilever calibration methods. Sader’s
% method for calibration under light fluid loading and the new method I
% recently developed for heavy fluid loading. There are 5 steps involved
% in using this GUI.
%
% First, a data file is loaded. It should contain 2 vectors, freq, the
% frequency vector in Hz, and Syy, the thermal response data in volts
% squared per Hertz.
Second, the known cantilever and fluid properties are entered. The length
and width of the cantilever in micrometers should be entered into the
text boxes and the fluid selected using the radio buttons. The SHO and
fluid-structure interaction models update as the parameters are changed.

Third, the SHO model guesses are initialized using the appropriate slider
bars and text boxes to select values for the natural frequency, Q factor,
and scale of the SHO model. The slider values can be used to quickly
adjust the model while the text boxes can be used to fine tune the model
if needed.

Forth, the fluid-structure interaction model guesses are initialized
using the appropriate slider bars and text boxes to select values for the
natural frequency, mass ratio, and scale of the fluid-structure
interaction model. The slider values can be used to quickly adjust the
model while the text boxes can be used to fine tune the model if needed.

Finally, pressing the ‘Calibrate’ button initiates algorithms to perform
the two calibration procedures. The user inputs and initial model
parameters are used in automatic fittings of the two models to the
data. The SHO model is repeatedly fit to various data ranged to
determine the appropriate data range (as defined by the parameter
SR_setpoint) before a final SHO fitting is performed. The parameters
from the final SHO fitting are used to generate a stiffness estimate with
Sader’s method. The new calibration technique for heavy fluid loading
% employs an iterative loop. The fluid-structure interaction model is fit
% to the data using the initial natural frequency guess as a fixed variable.
% The fitting is then performed a second time using a higher natural frequency
% guess. The stiffness estimates derived from these two fittings are
% compared. If the resulting stiffness estimations are sufficiently close
% to each other, the loop ends. If they are not, the higher of the two
% natural frequencies is used as the low natural frequency guess and a new
% higher natural frequency guess is made to repeat the process. In this
% way, the algorithm fits the fluid-structure interaction model to the data
% in a way that restricts the search space to heavy fluid loading
% situations. While each calibration technique is only appropriate in the
% proper loading domain, both results are displayed. Care should be taken
% to only use the appropriate stiffness estimation.
%
% Begin initialization code - DO NOT EDIT

gui_Singleton = 1;

gui_State = struct('gui_Name', mfilename, ...
    'gui_Singleton', gui_Singleton, ...
    'gui_OpeningFcn', @calibrate_1_1_OpeningFcn, ...
    'gui_OutputFcn', @calibrate_1_1_OutputFcn, ...
    'gui_LayoutFcn', [], ...
    'gui_Callback', []);

if nargin && ischar(varargin1)
    gui_State.gui_Callback = str2func(varargin1);
end

if nargout

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[varargout1:nargout] = gui_mainfcn(gui_State, varargin);
else
gui_mainfcn(gui_State, varargin);
end
% End initialization code - DO NOT EDIT
% — Executes just before calibrate_1_1 is made visible.
function calibrate_1_1_OpeningFcn(hObject, eventdata, handles, varargin)
% Initialize and set global variables
clear
disp(' This program is free software: you can redistribute it and/or modify')
disp(' it under the terms of the GNU General Public License as published by')
disp(' the Free Software Foundation, either version 3 of the License, or')
disp(' (at your option) any later version.

disp('

disp(' This program is distributed in the hope that it will be useful,')
disp(' but WITHOUT ANY WARRANTY; without even the implied warranty of')
disp(' MERCHANTABILITY or FITNESS FOR A PARTICULAR PURPOSE. See')
disp(' the')
disp(' GNU General Public License for more details.

disp('

disp(' You should have received a copy of the GNU General Public License')
disp(' along with this program. If not, see <http://www.gnu.org/licenses/>.

axes(handles.cbimms_logo);
cbimms=imread('cbimms.jpg');
image(cbimms);
axis off
axes(handles.duke_logo);
duke=imread('logo_copy1.jpg');
image(duke);
axis off

global b L rho_f eta sr_setpoint fr A k k_New C1 wn gamma_mass scale

global kbt wn_SHO Q_SHO Pdc_SHO A Pw_SHO Pw beta

b=30*10^-6; % Cantilever width
L=100*10^-6; % cantilever length
rho_f=1.205; % air density @ 20C
eta=18.21e-6; % air viscosity at 20C
sr_setpoint=5; % the desired ratio of the SHO max to min over the fit
% frequency range. At sr_setpoint = 2, the FWHM is fit
Pdc_SHO=1e-11;
Pw_SHO=1e-28;
fr=2e4;
wn_SHO=fr*2*pi;
Q_SHO=3;
A=.3;
k=.1;
k_New=.05;
C1=1.875104;
wn=2*pi*fr;
gamma_mass=3*wn^2*L*pi*rho_f*b^2/(4*C1^4*k);
scale=1e12;
Pw=Pw_SHO;
kbt=4.143e-21; @300 K
beta=2;

% End initialization of global variables

set(hObject,'toolbar','figure')
% This function has no output args, see OutputFcn.
% hObject handle to figure
% eventdata reserved - to be defined in a future version of MATLAB
% handles structure with handles and user data (see GUIDATA)
% varargin command line arguments to calibrate_1_1 (see VARARGIN)
% Choose default command line output for calibrate_1_1
handles.output = hObject;
% Update handles structure
guidata(hObject, handles);
% UIWAIT makes calibrate_1_1 wait for user response (see UIRESUME)
% uiwait(handles.figure1);
% — Outputs from this function are returned to the command line.

function varargout = calibrate_1_1_OutputFcn(hObject, eventdata, handles)
varargout1 = handles.output;

% — Executes on button press in LoadFile.
function LoadFile_Callback(hObject, eventdata, handles)

global Syy freq Syy1 freq1 Pw_SHO Pw
uiopen('load')
if freq(1)==0
    freq=freq(2:length(freq));
    Syy=Syy(2:length(Syy));
end
freq1=freq;
Syy1=Syy;
Pw=max(Syy1)*10^6;
axes(handles.axes1);
loglog(freq,Syy);

% — Executes on selection change in popupmenu1.
function popupmenu1_Callback(hObject, eventdata, handles)

% — Executes during object creation, after setting all properties.
function popupmenu1_CreateFcn(hObject, eventdata, handles)
if ispc && isequal(get(hObject,'BackgroundColor'),...
    get(0,'defaultUicontrolBackgroundColor'))
    set(hObject,'BackgroundColor','white');
end

% — Executes on button press in select_air.
function select_air_Callback(hObject, eventdata, handles)

    global rho_f eta A
rho_f=1.205; %air density @ 20C
eta=18.21e-6; %air viscosity at 20C
A=.04; %initial guess
update_models(handles);

% — Executes on button press in select_water.
function select_water_Callback(hObject, eventdata, handles)

global rho_f eta A

clear global rho_f eta A;
rho_f=1000; %water density 1000
eta=.001002; %.001002 viscosity of water @ 20 C in Ns/m^2
A=.3; %initial guess
update_models(handles);

function Length_Callback(hObject, eventdata, handles)

global L

L=str2double(get(hObject,'String'))*10^-6;
update_models(handles);

% — Executes during object creation, after setting all properties.
function Length_CreateFcn(hObject, eventdata, handles)

if ispc && isequal(get(hObject,'BackgroundColor'),...

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get(0,'defaultUicontrolBackgroundColor'))
    set(hObject,'BackgroundColor','white');
end

function Width_Callback(hObject, eventdata, handles)

global b

    b=str2double(get(hObject,'String'))*10^-6;
    update_models(handles);

    % — Executes during object creation, after setting all properties.
    function Width_CreateFcn(hObject, eventdata, handles)

        if ispc && isequal(get(hObject,'BackgroundColor'),...
                            get(0,'defaultUicontrolBackgroundColor'))
            set(hObject,'BackgroundColor','white');
        end

    function update_models(handles)
    global b L rho_f eta C1 wn gamma_mass scale omega H_rect Pw beta
    global kbt Syy freq wn_SHO Q_SHO Pdc_SHO Pw_SHO SHO_model freq1 Syy1

        % Hydrodynamic function
        omega=2*pi*freq;
        Re=omega*b^2*rho_f/(4*eta);
H_rect=Sader_hydro(Re);
s=j*omega;
G=wn^2./(s.^2+wn^2);
K=gamma_mass*(s.^2/wn^2).*H_rect;
P=G./(1+G.*K);
mu=pi*rho_f*b^2/4/gamma_mass;
m=mu*L;
k1=wn^2*m;
k_Cole_fit=3*k1/C1^4;
Sqq=Pw./omega.^beta-4*kbt*imag(P)./omega/k1*4;
Cole_model=(Sqq*scale);
SHO_model = Pdc_SHO*(wn_SHO/2/pi)^4./((freq.^2-(wn_SHO/2/pi)^2).^2+freq.^2*...
(wn_SHO/2/pi)^2/Q_SHO^2);
axes(handles.axes1);
plotrange=get(handles.axes1,'Xlim');
loglog(freq,Syy,freq,Cole_model,freq,SHO_model,freq1,Syy1,...
freq.scale*Pw./omega.^beta);
set(handles.axes1,'Xlim',plotrange);
xlabel('Frequency (Hz)')
ylabel('Mean Response (V^2/Hz)')
legend('Measured Response','F-S Interaction Model','SHO Model')
legend('boxoff')

% — Executes on slider movement.
function nat_freq_bar_Callback(hObject, eventdata, handles)
global wn

wn=exp((get(hObject,'Value'))*10)*1000;
set(handles.nat_freq,'String',wn/2/pi);
update_models(handles);

% — Executes during object creation, after setting all properties.
get(hObject,'Value');

function nat_freq_bar_CreateFcn(hObject, eventdata, handles)

if isequal(get(hObject,'BackgroundColor'),...
    get(0,'defaultUicontrolBackgroundColor'))
    get(0,'defaultUicontrolBackgroundColor'))
    set(hObject,'BackgroundColor',[.9 .9 .9]);
end

% — Executes on slider movement.
function gamma_bar_Callback(hObject, eventdata, handles)

global b L rho_f k k_New C1 wn gamma_mass

gamma_mass=(exp((get(hObject,'Value'))*10)-.9999999)/10;
k=3*wn^2*L*pi*rho_f*b^2/(4*C1^4*gamma_mass);
k_New=k/2;
set(handles.gamma,'String',gamma_mass);
update_models(handles);
get(hObject, 'Value');

% — Executes during object creation, after setting all properties.
function gamma_bar_CreateFcn(hObject, eventdata, handles)

if isequal(get(hObject, 'BackgroundColor'), ...
    get(0, 'defaultUicontrolBackgroundColor'))
    set(hObject, 'BackgroundColor', [.9 .9 .9]);
end

% — Executes on slider movement.
function scale_bar_Callback(hObject, eventdata, handles)

global scale

scale = exp((get(hObject, 'Value'))*50)*1e4;
set(handles.scale, 'String', scale);
update_models(handles);

% — Executes during object creation, after setting all properties.
function scale_bar_CreateFcn(hObject, eventdata, handles)

if isequal(get(hObject, 'BackgroundColor'), ...
    get(0, 'defaultUicontrolBackgroundColor'))
    set(hObject, 'BackgroundColor', [.9 .9 .9]);
end
function nat_freq_Callback(hObject, eventdata, handles)

global wn

wn=str2double(get(hObject,'String'))*2*pi;
update_models(handles);

% — Executes during object creation, after setting all properties.
function nat_freq_CreateFcn(hObject, eventdata, handles)

if ispc && isequal(get(hObject,'BackgroundColor'),...
    get(0,'defaultUicontrolBackgroundColor'))
    set(hObject,'BackgroundColor','white');
end

function gamma_Callback(hObject, eventdata, handles)

global b L rho f k k_New C1 wn gamma_mass

gamma_mass=str2double(get(hObject,'String'));
k=3*wn^2*L*pi*rho_f*b^2/(4*C1^4*gamma_mass);
k_New=k/2;
update_models(handles);

% — Executes during object creation, after setting all properties.
function gamma_CreateFcn(hObject, eventdata, handles)

if ispc && isequal(get(hObject, 'BackgroundColor'), get(0,'defaultUicontrolBackgroundColor'))
    set(hObject, 'BackgroundColor', 'white');
end

function scale_Callback(hObject, eventdata, handles)

global scale

scale=str2double(get(hObject, 'String'));
update_models(handles);

% — Executes during object creation, after setting all properties.
function scale_CreateFcn(hObject, eventdata, handles)

if ispc && isequal(get(hObject, 'BackgroundColor'), get(0,'defaultUicontrolBackgroundColor'))
    set(hObject, 'BackgroundColor', 'white');
end

% — Executes on slider movement.
function nat_freq_bar_SHO_Callback(hObject, eventdata, handles)

global wn_SHO

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wn_SHO = \exp((\text{get(hObject,'Value')} \times 10)\times 1000;
set(handles.nat_freq_SHO,'String',\text{wn} \_ \text{SHO}/2/\pi);
update\_models(handles);

% — Executes during object creation, after setting all properties.
function nat\_freq\_bar\_SHO\_CreateFcn(hObject, eventdata, handles)

if isequal(get(hObject,’BackgroundColor’),

    set(hObject,’BackgroundColor’, [.9 .9 .9]);
end

% — Executes on slider movement.
function Q\_bar\_SHO\_Callback(hObject, eventdata, handles)

global Q\_SHO

Q\_SHO = (\exp((\text{get(hObject,’Value’)} \times 10)\times .9999999)/10;
set(handles.Q\_SHO,’String’,Q\_SHO);
update\_models(handles);

% — Executes during object creation, after setting all properties.
function Q\_bar\_SHO\_CreateFcn(hObject, eventdata, handles)

if isequal(get(hObject,’BackgroundColor’),...
get(0,'defaultUicontrolBackgroundColor'))
set(hObject,'BackgroundColor',[.9 .9 .9]);
end

% — Executes on slider movement.
function Pdc_bar_SHO_Callback(hObject, eventdata, handles)

global Pdc_SHO

Pdc_SHO=exp((get(hObject,'Value'))*50)*1e-24;
set(handles.Pdc_SHO,'String',Pdc_SHO);
update_models(handles);

% — Executes during object creation, after setting all properties.
function Pdc_bar_SHO_CreateFcn(hObject, eventdata, handles)

if isequal(get(hObject,'BackgroundColor'),...
    get(0,'defaultUicontrolBackgroundColor'))
    set(hObject,'BackgroundColor',[.9 .9 .9]);
end

function nat_freq_SHO_Callback(hObject, eventdata, handles)

global wn_SHO

wn_SHO=2*pi*str2double(get(hObject,'String'));

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update_models(handles);

function nat_freq_SHO_CreateFcn(hObject, eventdata, handles)

if ispc && isequal(get(hObject, ’BackgroundColor’), ...
    get(0,’defaultUicontrolBackgroundColor’))
    set(hObject, ’BackgroundColor’, ’white’);
end

function Q_SHO_Callback(hObject, eventdata, handles)

global Q_SHO

Q_SHO=str2double(get(hObject,’String’));
update_models(handles);

function Pdc_SHO_Callback(hObject, eventdata, handles)
global Pdc_SHO

Pdc_SHO=str2double(get(hObject,'String'));
update_models(handles);

% — Executes during object creation, after setting all properties.
function Pdc_SHO_CreateFcn(hObject, eventdata, handles)

if ispc && isequal(get(hObject,'BackgroundColor'),...
        get(0,'defaultUicontrolBackgroundColor'))
        set(hObject,'BackgroundColor','white');
end

% — Executes on button press in Calibrate.
function Calibrate_Callback(hObject, eventdata, handles)

global b L rho f eta A k k New C1 wn gamma mass scale Pw omega H rect

global Syy freq wn SHO Q SHO Pdc_SHO Pw_SHO SHO_model beta

global Index1 freq1 Syy1 SHO_model1 selection_ratio

A = fminsearch(@(A) A_cst(A,handles),A,optimset('Display','off'));

% select frequency range to fit
Index1=find(SHO_model==max(SHO_model));
freq1=freq(floor(Index1*(1-A)):ceil(Index1*(1+A)));
Syy1=Syy(floor(Index1*(1-A)):ceil(Index1*(1+A)));

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SHO_model1 = Pdc_SHO*(wn_SHO/2/pi)^4./((freq1.^2-(wn_SHO/2/pi)^2).^2+...
  freq1.^2*(wn_SHO/2/pi)^2/Q_SHO^2);
selection_ratio=max(SHO_model1)/min(SHO_model1);

set(handles.nat_freq_SHO,'String',wn_SHO/2/pi);
set(handles.Q_SHO,'String',Q_SHO);
set(handles.Pdc_SHO,'String',Pdc_SHO);

%Sader Calibration
omega=wn_SHO;

%Hydrodynamic function at a single point
Re=omega*b^2*rho_f/(4*eta);
H_rect=Sader_hydro(Re);
k_Sader=-0.1906*rho_f*b^2*L*Q_SHO*wn_SHO^2*imag(H_rect);
% Sader’s calibration, while not intended for heavy fluid loading, provides a good
% initialization value for the search algorithm.
% it is negative because H_rect is in Cole’s convention for modeling
% purposes
k_New=k_Sader;

k_New2=k_Sader;
k2=k;

%Hydrodynamic function
omega=2*pi*freq1;
Re=omega*b^2*rho_f/(4*eta);
H_rect=Sader_hydro(Re);
while (k_New-k)^2>1e-10
  k=k_New;
end
wn=wn*10;
gamma_mass=3*wn^2*L*pi*rho_f*b^2/(4*C1^4*k);
param_g_s=[gamma_mass,scale,Pw,beta];
param_g_s = fminsearch(@(param_g_s) Cole_cost_g_s(param_g_s,handles),...
                    param_g_s,optimset('Display','off'));
gamma_mass=param_g_s(1);
scale=param_g_s(2);
Pw=param_g_s(3);
beta=param_g_s(4);
mu=pi*rho_f*b^2/4/gamma_mass;
m=mu*L;
k1=wn^2*m;
k_New=3*k1/C1^4;
end

% plot Cole fit
set(handles.nat_freq,'String',wn/2/pi);
set(handles.gamma,'String',gamma_mass);
set(handles.scale,'String',scale);
set(handles.k_New_out,'String',k_New);
set(handles.setpt_out,'String',selection_ratio);
update_models(handles);

function J_A = A_cost(A,handles)

global sr_setpoint Index1 freq1 Syy1 SHO_model1 selection_ratio
global Syy freq wn_SHO Q_SHO Pdc_SHO Pw_SHO SHO_model
Index1 = find(SHO_model == max(SHO_model));

freq1 = freq(floor(Index1*(1-A)):ceil(Index1*(1+A)));

Syy1 = Syy(floor(Index1*(1-A)):ceil(Index1*(1+A)));

% find SHO fit to selected data

param_SHO(1) = Pdc_SHO;

param_SHO(2) = wn_SHO / 2 / pi;

param_SHO(3) = Q_SHO;

param_SHO = fminsearch(@(param_SHO) SHO_cost(param_SHO, handles), param_SHO, ... optimset('Display', 'off'));

Pdc_SHO = param_SHO(1);

wn_SHO = param_SHO(2) * 2 * pi;

Q_SHO = param_SHO(3);

SHO_model = Pdc_SHO * (wn_SHO / 2 / pi)^4 ./ ((freq^2 - (wn_SHO / 2 / pi)^2)^2 + ... freq^2 * (wn_SHO / 2 / pi)^2 / Q_SHO^2);

SHO_model1 = Pdc_SHO * (wn_SHO / 2 / pi)^4 ./ ((freq1^2 - (wn_SHO / 2 / pi)^2)^2 + ... freq1^2 * (wn_SHO / 2 / pi)^2 / Q_SHO^2);

selection_ratio = max(SHO_model1) / min(SHO_model1);

J_A = (sr_setpoint-selection_ratio)^2;

function J_SHO = SHO_cost(param_SHO, handles)

global wn_SHO Q_SHO Pdc_SHO Pw_SHO freq1 Syy1 SHO_model1 Syy_error

Pdc_SHO = param_SHO(1);

wn_SHO = param_SHO(2) * 2 * pi;
Q_SHO=param_SHO(3);

SHO_model1 = Pdc_SHO*(wn_SHO/2/pi)^4./((freq1.^2-(wn_SHO/2/pi)^2).^2+...
    freq1.^2*(wn_SHO/2/pi)^2/Q_SHO^2);

Syy_error = ( SHO_model1 - Syy1 );

J_SHO = norm(Syy_error);

function J_g_s = Cole_cost_g_s(param_g_s,handles)

    global b L rho_f wn gamma_mass scale Pw kbt Syy1 Syy_error omega H_rect beta

    gamma_mass=param_g_s(1);
    scale=param_g_s(2);
    Pw=param_g_s(3);
    beta=param_g_s(4);
    % Cole Model
    s=j*omega;
    zeta=0;
    G=wn^2./(s.^2+2*zeta*wn*s+wn^2);
    K=gamma_mass*(s.^2/wn^2).*H_rect;
    P=G./(1+G.*K);
    mu=pi*rho_f*b^2/4/gamma_mass;
    m=mu*L;
    k1=wn^2*m;
    Sqq=Pw./omega.^beta-4*kbt*imag(P)./omega/k1*4; % 4 is the modal amplitude
    Syy_est=Sqq*scale;
    Syy_error = ( Syy_est - Syy1 );
\[ J_{gs} = \text{norm}(Syy\_error); \]

return;

function \([H\_rect]= \text{Sader\_hydro}(Re)\)

% The hydrodynamic forces on a cylindrical beam as given in
% eqns 18, 20, and 21 of J. E. Sader, Frequency response of cantilever beams
% immersed in viscous fluids with applications to the atomic force microscope.,
% Journal of Applied Physics, Vol. 84, No. 1, July 1998
x1=sqrt(i.*Re);
H=1+(4*i.*besselk(1,-i*x1))./(x1.*besselk(0,-i*x1));
tau=log10(Re);
Omega_r=(0.91324-0.48274*tau+0.46842*tau.^2-0.12886*tau.^3+0.044055*tau.^4-...
  0.0035117*tau.^5+0.00069085*tau.^6).*(1-0.56964*tau+0.48690*tau.^2-...
  0.13444*tau.^3+0.045155*tau.^4-0.0035862*tau.^5+0.00069085*tau.^6).^-1;
Omega_i=(-0.024134-0.029256*tau+0.016294*tau.^2-0.00010961*tau.^3-...
  0.00064577*tau.^4-0.000044510*tau.^5).*(1-0.59702*tau+0.55182*tau.^2-...
  0.18357*tau.^3+0.079156*tau.^4-0.014369*tau.^5+0.0028361*tau.^6).^-1;
Omega=Omega_r+i*Omega_i;
H\_rect=conj(Omega.*H); % The conjugate is needed to account for differences in
% notation
B.2 Transient Response Parameter Identification

In Chapter 6, FEM-CFD simulations were presented that produced transient responses to momentary loading. The simulations produced displacement data as a function of time. To identify cantilever-fluid system characteristics from this time response, a state space model was created and excited with the same loading profile applied to the ANSYS model. The state space parameters were adjusted until the resulting transient minimized a cost function that compared it to the ANSYS transient.

Several variations on this strategy were tested. The strategy presented includes four SHO models that are fit to the ANSYS response in two stages. In the first stage, the natural frequencies are scaled relative to each other to maintain the ratios predicted by beam theory. In the second stage, the restrictions are removed and each mode is fit independently.
B.2.1  MATLAB Program: ID_Omega_from_transient_Ver_2.m

%Fits 4 SHO models to a transient response. The user must initialize %fn, Q, k, and d on lines 19 through 22. The input file is named on line %23 followed by parameters allowing the user to select a specific portion of %the transient to fit. The sample rate, simulation length and forcing %characteristics are input on lines 24-38. The cantilever beam width and %the fluid properties and input on lines 40-44. They are not required for %parameter identification, but they are needed to identify the hydrodynamic %loading.

%The 4 SHO models are first fit to the transient in a way the locks the %ratios of the natural frequencies to the ratios predicted by theory. That %is followed by a second unrestrained fitting.

close all
clear all
clc

%user initialization
d=1; %a factor to account for the frequency shift induced by damping
fn=16100; %Hz
Q=100;
k=.02; %N/m
load filename
n1=1;
n2=length(resp);
% n2=120;

\[ t_{\text{sample rate}} = \text{time}(2) - \text{time}(1) \]
\[ t_{\text{end}} = \text{time}(n2) \]

% A zero should be added to the beginning of the response. ANSYS does not include the initial condition in the response.

response(1)=0;
response(2:n2+1)=resp(1:n2);
\[ t = 0 : t_{\text{sample}} : (\text{length(response)}-1) \cdot t_{\text{sample}} \]
\[ u = \text{zeros}(1, \text{length(response)}) \]
\[ u(1:33) = 2e-2 \]; % u is the forcing function
\[ u(34) = u(1)/2 \]

b=20e-6; % beam width
% rho_f=997.5; % water density kg/m^3
% eta=.0009453; %.001002 viscosity of water @ 20 C in Ns/m^2
rho_f=1.185; % kg/m^3 air ANSYS
eta=1.831e-5; % Pa sec air ANSYS

% end user initialization

a1=3.52; %(\alpha*l)^2
a2=22.0;
a3=61.7;
a4=121.0;
a5=200.0;
fn2=fn*a2/a1/d;
fn3=fn*a3/a1/d;
fn4=fn*a4/a1/d;
wn=2*pi*fn;
wn2=2*pi*fn2;
wn3=2*pi*fn3;
wn4=2*pi*fn4;
Q2=Q;
Q3=Q;
Q4=Q;
k2=k*(a2/a1)^2;
k3=k*(a3/a1)^2;
k4=k*(a4/a1)^2;
k_known=k;

% a zero should be added to the beginning of the response. ANSYS does not
% include the initial condition in the response.
response(1)=0;
response(2:n2+1)=resp(1:n2);
M=k/wn^2;
M2=M;
M3=M;
M4=M;
B=M*wn/Q;
B2=M2*wn2/Q2;
B3=M3*wn3/Q3;
B4=M4*wn4/Q4;

f=1; %f allows for some adjustment of the frequency spacing of the modes while
%maintaining the ratio between frequencies.

param=[M,B,k,B2,B3,B4,f];
param = fminsearch(@(param) find_responseSS_cost_ver_2(param,t,u,response,n1,n2),...
    param,optimset('TolX',1e-12,'MaxFunEvals',2500));

M=abs(param(1));
B=abs(param(2));
k=abs(param(3));
B2=abs(param(4));
B3=abs(param(5));
B4=abs(param(6));
f=param(7);
k2=k*(a2/a1)^2*f; %the use of ‘f’ is explained above (line 79)
k3=k*(a3/a1)^2*f;
k4=k*(a4/a1)^2*f;
M2=M;
M3=M;
M4=M;
y=find_responseSS_ver_2(M,B,k,M2,B2,k2,M3,B3,k3,M4,B4,k4,t,u);
figure(3)
plot(t,response,t,y)
title('Restricted Fitting Results')

param=[M,B,k,M2,B2,k2,M3,B3,k3,M4,B4,k4];
param = fminsearch(@(param) find_responseSS_cost_ver_2b(param,t,u,response,n1,n2),...
param, optimset(‘TolX’, 1e-12, ’MaxFunEvals’, 2500));

M = abs(param(1))
B = abs(param(2))
k = abs(param(3))
M2 = abs(param(4));
B2 = abs(param(5));
k2 = abs(param(6));
M3 = abs(param(7));
B3 = abs(param(8));
k3 = abs(param(9));
M4 = abs(param(10));
B4 = abs(param(11));
k4 = abs(param(12));
y = find_responseSS_ver_2(M, B, k, M2, B2, k2, M3, B3, k3, M4, B4, k4, t, u);

wn = sqrt(k/M);
wn2 = sqrt(k2/M2);
wn3 = sqrt(k3/M3);
wn4 = sqrt(k4/M4);

fn = wn/2/pi
fn2 = wn2/2/pi
fn3 = wn3/2/pi
fn4 = wn4/2/pi

Q = M*wn/B
Q2 = M2*wn2/B2
Q3 = M3*wn3/B3
Q4 = M4*wn4/B4
L0=b/2;
Re=rho_f*L0^2*wn/eta
Omega=k_known/(rho_f*L0^3*wn^2*Q)
Omega_ksolve=k/(rho_f*L0^3*wn^2*Q)
y2=find_responseSS(fn,Q,k,t,u);
y3=find_responseSS(fn2,Q2,k2,t,u);
y4=find_responseSS(fn3,Q3,k3,t,u);
y5=find_responseSS(fn4,Q4,k4,t,u);
figure(4)
plot(t,response,t,y,t,y2,t,y3,t,y4,t,y5)
legend(‘data’,’fit’,’m1’,’m2’,’m3’,’m4’)
B.2.2 MATLAB Function: find_responseSS_cost_ver_2.m

function J = find_responseSS_cost_ver_2(param,t,u,resp,n1,n2)

d=2;
a1=3.52;
a2=22.0;
a3=61.7;
a4=121.0;
a5=200.0;

M=abs(param(1));
B=abs(param(2));
k=abs(param(3));
B2=abs(param(4));
B3=abs(param(5));
B4=abs(param(6));
f=param(7);
k2=k*(a2/a1)^2*f;
k3=k*(a3/a1)^2*f;
k4=k*(a4/a1)^2*f;
M2=M;
M3=M;
M4=M;
A=[0 1 0 0 0 0 0 0;...
   -1*k4/M4 -1*B4/M4 0 0 0 0 0 0;...
   0 0 0 1 0 0 0 0;...
   0 0 -1*k3/M3 -1*B/M3 0 0 0 0;...
   ...];
\[
\begin{bmatrix}
0 & 0 & 0 & 0 & 1 & 0 & 0;
0 & 0 & 0 & 0 & -1 & B_2/M_2 & 0 & 0;
0 & 0 & 0 & 0 & 0 & 0 & 1;
0 & 0 & 0 & 0 & 0 & 0 & -1 & k/M & -1 & B/M;
\end{bmatrix};
\]

\[
B = \begin{bmatrix}
0; & 1/M_4; & 0; & 1/M_3; & 0; & 1/M_2; & 0; & 1/M;
\end{bmatrix};
\]

\[
C = [1 & 0 & 1 & 0 & 1 & 0 & 1 & 0];
\]

\[
D = 0;
\]

\[
H = SS(A,B,C,D);
\]

\[
y = lsim(H, u, t); \text{'y=lsim for the data'}
\]

\[
err = (\text{resp}(n1:n2) - y(n1:n2))';
\]

\[
\text{figure(2)}
\]

\[
\text{plot(t,resp,t,y)}
\]

\[
J = \text{norm(err)};
\]

### B.2.3 MATLAB Function: find_responseSS_cost_ver_2b.m

```matlab
function J = find_responseSS_cost_ver_2b(param,t,u,resp,n1,n2)
M = abs(param(1));
B = abs(param(2));
k = abs(param(3));
M2 = abs(param(4));
B2 = abs(param(5));
k2 = abs(param(6));
M3 = abs(param(7));
B3 = abs(param(8));
k3 = abs(param(9));
```

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M4=abs(param(10));
B4=abs(param(11));
k4=abs(param(12));

A=[0 1 0 0 0 0 0;
   -1*k4/M4 -1*B4/M4 0 0 0 0 0;
   0 0 1 0 0 0 0;
   0 0 -1*k3/M3 -1*B/M3 0 0 0;
   0 0 0 0 1 0 0;
   0 0 0 0 -1*k2/M2 -1*B2/M2 0 0;
   0 0 0 0 0 1;
   0 0 0 0 0 0 -1*k/M -1*B/M];
B=[0;1/M4;0;1/M3;0;1/M2;0;1/M];
C=[1 0 1 0 1 0 1 0];
D=0;
H=SS(A,B,C,D);
y=lsim(H,u,t);%y=lim for the data
err=(resp(n1:n2)-y(n1:n2)');
figure(2)
plot(t,resp,t,y)
J=norm(err);

B.2.4 MATLAB Function: find_responseSS_ver_2.m

function y = find_responseSS_ver_2(M,B,k,M2,B2,k2,M3,B3,k3,M4,B4,k4,t,u)
A=[0 1 0 0 0 0 0;... 
   -1*k4/M4 -1*B4/M4 0 0 0 0 0;... 
   0 0 1 0 0 0 0;... 
   0 0 -1*k3/M3 -1*B/M3 0 0 0;... 
   0 0 0 0 1 0 0;... 
   0 0 0 0 -1*k2/M2 -1*B2/M2 0 0;... 
   0 0 0 0 0 0 0 1;... 
   0 0 0 0 0 0 -1*k/M -1*B/M];
B=[0;1/M4;0;1/M3;0;1/M2;0;1/M];
C=[1 0 1 0 1 0 1 0];
D=0;
H=SS(A,B,C,D);
y=lsim(H,u,t);%y=lim for the data

B.2.5 MATLAB Function: find_responseSS.m

function y = find_responseSS(fn,Q,k,t,u)

wn=2*pi*fn;
A=[0 1;-1*wn^2 -1*wn/Q];
B=[0;wn^2/k];
C=[1 0];
D=0;
H=SS(A,B,C,D);
y=lsim(H,u,t);%y=lim for the data

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B.3 Thermal Noise Calibration

The thermal noise calibration method is a popular technique, readily available in the software that accompanies commercial AFM systems such as the Asylum Research MFP-3D. As discussed in Chapter 4, the difference in calibrations performed in air and in water can be reduced significantly by using alternate dynamics models in the curve fitting of the thermal vibration. The presented software uses a Lorentzian line function rather than an SHO to approximate the cantilever-fluid system dynamics. Rather than constant-magnitude white noise, the magnitude and the slope of the noise model are curve fit to the data. Additionally, a 160 Hz, high-pass filter is included in the model to reflect the RC filter in the MFP-3D.
B.3.1 MATLAB Program: Thermal_Noise.m

% This program allows the user to calibrate a cantilever using the thermal
% noise method. The model for fitting the thermal vibration data is a
% Lorentzian line function and variable-slope noise.

% Usage Summary
% 1) Adjust the parameters in the USER INPUTS section and run the m-file.
% 2) On the first plot that pops up, click the data cursor button and select the
% center of the resonance peak.
% 3) Right-click the yellow data window and select 'Export Cursor Data to
% Workspace...'
% 4) Click ok.
% 5) Select the main MATLAB window and press any key.
% 6) On the second plot that pops up, click the data cursor button and select a
% point at the low frequency limit of the data you would like to use in the
% curve fitting.
% 7) Right-click the yellow data window and select 'Export Cursor Data to
% Workspace...'
% 8) Click ok.
% 9) Select the main MATLAB window and press any key.
% 7) After the program finishes, check the plot to confirm that the SHO
% model provides a good fit.
% 8) In the MATLAB window, confirm that the Lorentzian parameters are positive.

% 9) It is sometimes helpful to check Figure 1 after the program has finished to
% verify that the initial conditions get the model somewhat close to the data.
After the user selects the first mode resonance peak, this program selects a frequency range to curve fit by manipulating the variable ‘A’. A large value for A results in a large frequency range centered on the resonance peak being fit, small A results in a small frequency range being used. This version of the code fits an SHO to the data and then uses the SHO fit to determine if the frequency range used is the full width at half maximum (FWHM). If it is not, A is adjusted and the SHO fit again. Future versions of this code may simply smooth the measured data and find the FWHM from the smoothed data.

On rare occasions, the Lor fit will yield nonsensical results, such as negative values of Q or fr. If this is the case, changing the Lor initialization values so that they are close to the absolute values of the previous set of results will usually correct the problem.

Example:

If this program yields Pdc = 3.4e-012, fr = 3.3e+004, and Q = -3.2, change the initialization to Pdc = 3e12; fr = 3e4, Q = 3.2

Required Functions:

lor_cost_fb_filt.m
integrate.m

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clc
close all
clear all
format compact
format long

%**************************************************************************
% USER INPUTS
%**************************************************************************

load sept15_t20
freq=freq;
Syy=Syy;
b=36*10^-6; % Cantilever width
L=85*10^-6; % cantilever length
%asylum uses a 160Hz RC filter
h=1./(1-i*(160./freq));
fullfreq=linspace(1,1e6,1e5);
sr_setpoint=5;
%select air or water - note: Sader’s method is inaccurate in water

rho_f=1000; %water density 1000
eta=.001002; %.001002 viscosity of water @ 20 C in Ns/m^2 .00089 @ 25 C
Q=1.5;
A=.2;
gamma\_mass=3;
beta=1;

% rho\_f=1.205; %air density @ 20C
% eta=18.21e-6; %air viscosity at 20C
% Q=30;
% A=.03;
% gamma\_mass=.01;

%**************************************************************************
% END USER INPUTS
%**************************************************************************

kbt=4.143e-21; %@300 K
C1=1.875104;

%initialize SHO parameters and A
%Obtains the data indexes for the minimums before and after the resonant
figure(1)
loglog(freq,Syy)
xlabel('Frequency (Hz)')
ylabel('nm^2/Hz')
title('Power Spectral Density - identify the peak')
fprintf('click points take data

');
pause
ind1=cursor\_info(1).DataIndex;
fr= freq(ind1);
fri=fr;
Qi=Q;
figure(2)
loglog(freq,Syy)
xlabel('Frequency (Hz)')
ylabel('nm^2/Hz')
title('Power Spectral Density - identify the noise')
fprintf('click points take data\n\n');
pause
indn=cursor_info(1).DataIndex;
Pp=Syy(indn)*freq(indn).^beta/2;
Pdc=Pp/freq(indn);
Ppi=Pp;
Pdci=Pdc;
%temp plot to check initial conditions if needed
Syy_Lortemp = abs(h).^2.*(abs(Ppi./freq.^beta)+Pdci*fr.*2./(4.*(freq-fr).^2+fr.^2/Qi.^2));
%using the second click to pick A
A=abs(ind1-indn)/ind1;
freq1=freq(floor(ind1*(1-A)):ceil(ind1*(1+A)));
Syy1=Syy(floor(ind1*(1-A)):ceil(ind1*(1+A)));
h1=h(floor(ind1*(1-A)):ceil(ind1*(1+A)));
figure(1)
loglog(freq,Syy,'k',freq,Syy_Lortemp,'--k','LineWidth',2)
xlabel('frequency (Hz)')
ylabel('mean response (m^2/Hz)')
legend('measured response','Lor model')
Syy = \text{l}\text{or}\text{only} = |h|^2 \frac{P_{dc}\cdot fr^2}{(4\cdot(freq-fr)^2 + fr^2/Q^2)};

P_{dc} = \max(Syy_{\text{l}\text{or}\text{only}})/\max(Syy_{\text{l}\text{or}\text{temp}})\cdot P_{\text{dc}};

\text{param} = [P_{dc}, f_{ri}, Q, P_{\text{pi}}, \beta];

\text{param} = \text{fminsearch}(@(\text{param}) \text{lor\_cost\_fb\_filt}(\text{param}, \text{freq1}, Syy1, h1), \text{param});

P_{dc} = \text{param}(1);

f_{ri} = \text{param}(2);

Q = \text{param}(3);

P_{\text{pi}} = |\text{param}(4)|;

P_{\text{lor}} = P_{\text{pi}};

\beta_{\text{l}\text{or}} = |\text{param}(5)|;

Syy_{\text{l}\text{or}} = |h|^2 \frac{P_{\text{pi}}/freq^\beta + P_{dc}\cdot fr^2}{(4\cdot(freq-fr)^2 + fr^2/Q^2)};

Syy_{\text{l}\text{or}1} = |h1|^2 \frac{P_{\text{pi}}/freq1^\beta + P_{dc}\cdot fr^2}{(4\cdot(freq1-fr)^2 + fr^2/Q^2)};

Syy_{\text{l}\text{or}only} = P_{dc}\cdot fr^2/(4\cdot(freq-fr)^2 + fr^2/Q^2);

\text{figure(2)}

\text{loglog(freq1, Syy_{\text{l}\text{or}1}, 'r', 'LineWidth', 4)}

\text{hold on}

\text{loglog(freq, Syy, 'k', freq, Syy_{\text{l}\text{or}}, ':k', freq, Syy_{\text{l}\text{or}only}, ':k', 'LineWidth', 2)}

\text{xlabel('frequency (Hz)')}

\text{ylabel('mean response (m^2/Hz)')}

\text{legend('selected data range', 'measured response', 'Lor model w/ noise', 'Lor model')}

\text{legend('boxoff')}

\text{title('Lor fit')}

\text{hold off}

df = freq(2) - freq(1);
slor = integrate(Syy_Loronly(2:length(freq)),df);
klor = kbt/(slor/.9707)
Syy_lor1 = Syy_Lor(floor(ind1*(1-A)):ceil(ind1*(1+A)));
Syy_error = ( Syy1 - Syy_lor1 );
Jlor = norm(Syy_error)

B.3.2 MATLAB Function: lor_cost_fb_filt.m

function J = lor_cost_fb_filt(param,freq,Syy,h)
%
% calculate the estimated power spectrum
%
Pdc = abs(param(1));
fr = abs(param(2));
Q = abs(param(3));
Pp = abs(param(4));
beta = abs(param(5));
% calculate the pointwise normalized error
Syy_error = ( Syy_est - Syy );
% calculate the norm of the normalized error
J = norm(Syy_error);
return;
function [S]= integrate(curve,step)

%Simpson’s rule
%curve must have an even number of points
n=length(curve);
feven=0;
fodd=0;
for ii = 1:(n/2),
  feven=feven+curve(2*ii);
  fodd=fodd+curve(2*ii-1);
end
S=(step/3)*(-curve(1)-curve(n)+2*fodd+4*feven);
Bibliography


Biography

Scott J. Kennedy was born November 13, 1977 in Ann Arbor, Michigan, to Steven M. and Christine D. Kennedy. After graduating from Williamston High School in Williamston, Michigan, in 1996, he obtained a B.S. in mechanical engineering from the University of Michigan in 2001. This dissertation fulfilled the requirements for his Ph.D. in mechanical engineering from Duke University, which he obtained in 2010 under the advisement of Dr. Robert L. Clark and Dr. Daniel G. Cole. His research interests include dynamic testing and modeling, micro- and nanofabrication techniques, and technologies with applications in the harvesting, storage, and transmission of energy.