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Fundamentals and applications of fluid-structure interactions in compliant microchannels

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FUNDAMENTALS AND APPLICATIONS OF
FLUID-STRUCTURE INTERACTIONS IN COMPLIANT
MICROCHANNELS

by

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Beware of the young doctor and the old barber.

Benjamin Franklin
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FUNDAMENTALS AND APPLICATIONS OF FLUID-STRUCTURE INTERACTIONS IN COMPLIANT MICROCHANNELS

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ABSTRACT

The development of soft lithography techniques for fabricating microfluidic channels has enabled the study of microscale flows. These studies have become an essential component of experimental research in biology, fluid dynamics, engineering and related fields. A systematic understanding of microscale flows requires that the characteristics of the flow fields be determined accurately. However, as microchannels are scaled down, the size of most experimental probes becomes comparable to or even bigger than the micro-flows themselves, making the measurement of the distribution of flow fields problematic. In this work, we take advantage of the fact that most microfluidic channels are made up of soft materials and can deform during flow. We develop a non-invasive optical measurement technique to correlate the channel deformation with the pressure field inside the microchannel; we then apply this technique to studies of biological flows and flows on superhydrophobic surfaces.

Our analytical technique for extracting the pressure distribution in a deformable micro-channel is based on an initial measurement of the channel deflection profile
as a function of applied hydrostatic pressure. This initial measurement generates constitutive curves for the deformable channel. The deflection profile under flow is then matched to these constitutive curves, providing the hydrodynamic pressure distribution. The method is validated by comparing measurements on planar microfluidic channels with results from analytic and numerical models. The accuracy of the technique is independent of the nature of the wall deformations and does not degrade even in the limit of large wall deflections.

In order to test the applicability of our measurement technique to biological flows, we have extracted the interstitial fluid pressure (IFP) field within hydrogels in silicone (PDMS)-based microfluidic devices. Here, we have optically measured extremely slight deformations (< 1 μm) of the PDMS wall when a device filled with porous media is pressurized under static and dynamic flow conditions. The distension fields under uniform static pressure provide a map of the local device stiffness, which can be used to obtain the two-dimensional IFP field under flow conditions. We have validated this method numerically and applied it toward determining the hydraulic properties of cell aggregates and monolayers that are cultured within micropatterned gels. Using IFP maps in conjunction with computational models, we estimate the hydraulic permeability of tumor cells and the hydraulic conductivity of an epithelium monolayer; we find good agreement with values listed in the literature. Measurement of the IFP along with flow velocity could enable direct calculation of permeability for porous media, such as tissues and gels. Extracting IFP in biological media can be useful in understanding and predicting the effects of flow parameters on biological processes, particularly in those in which physical forces determine function.
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## List of Abbreviations

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<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>BLMEC</td>
<td>Bovine Lung Microvascular Endothelial Cells</td>
</tr>
<tr>
<td>CCD</td>
<td>Charge-Coupled Device</td>
</tr>
<tr>
<td>CL</td>
<td>Coherence Length</td>
</tr>
<tr>
<td>CSI</td>
<td>Coherence Scanning Interferometry</td>
</tr>
<tr>
<td>DOF</td>
<td>Degree of Freedom</td>
</tr>
<tr>
<td>DSP</td>
<td>Digital Signal Processing</td>
</tr>
<tr>
<td>HeNe</td>
<td>Helium-Neon</td>
</tr>
<tr>
<td>HMDS</td>
<td>Hexamethyldisilazane</td>
</tr>
<tr>
<td>IFP</td>
<td>Interstitial Fluid Pressure</td>
</tr>
<tr>
<td>KOH</td>
<td>Potassium hydroxide</td>
</tr>
<tr>
<td>LLC</td>
<td>Mouse Lewis Lung Carcinoma cells</td>
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<tr>
<td>MDCK</td>
<td>Madin-Darby Canine Kidney cells</td>
</tr>
<tr>
<td>OPD</td>
<td>Optical Path Difference</td>
</tr>
<tr>
<td>PDMS</td>
<td>Polydimethylsiloxane</td>
</tr>
<tr>
<td>PID</td>
<td>Proportional-Integral-Derivative</td>
</tr>
<tr>
<td>RIE</td>
<td>Reactive Ion Etching</td>
</tr>
<tr>
<td>RPM</td>
<td>Revolutions per Minute</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>Si</td>
<td>Silicon</td>
</tr>
<tr>
<td>SiN</td>
<td>Silicon Nitride</td>
</tr>
<tr>
<td>SNR</td>
<td>Signal to Noise Ratio</td>
</tr>
<tr>
<td>SWLI</td>
<td>Scanning White Light Interferometry</td>
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<tr>
<td>TCR</td>
<td>Temperature coefficient of electrical resistance</td>
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Chapter 1

Introduction

Fluid interactions with deformable bodies are ubiquitous in nature and observable in daily life (Dickenson et al., 2000). A fish swimming (Drucker and Lauder, 1999), an insect flying (Willmott et al., 1997), a flag flapping (Shelley and Zhang, 2011) or paper falling (Belmonte et al., 1998) are just a few examples. Flow-structure interactions can manifest themselves in the reconfiguration of deformable bodies under flow. The structure deforms in a way to minimize its cross-sectional area exposed to the flow. Shape changes under flow are crucial for the survival of many organisms. Broad tree leaves reconfigure themselves under high winds (Vogel, 1989), or many animal and plant species living in river and sea ecosystems have evolved to tolerate the high speed flow (Koehl, 1984). In all these examples, the general problem consists of coupling a solid structure's inertia or elastic response to the hydrodynamic forces created by the flow around it. In this thesis, we are interested in studying fluid-structure interactions. We conduct sensitive measurements of flows in micro channels with compliant walls. The flow is pressure driven, viscous and steady, and the compliant wall is a thin elastic solid. Fluid inertia in all cases is small compared to viscous forces, hence neglected.

1.1 Physiological Flows

Fluid-structure interactions are common in physiological flows. Due to its clinical importance, blood flow in arteries (Ku, 1997) and microvessels (Popel and Johnson, 2005) has been the topic of extensive research in the last three decades. Arteries are
large vessels that are not embedded in a tissue or organ. They carry blood pumped by the heart to other parts of the body. Microvessels form a vascular network which is responsible for the transfer of nutrients and gases between the blood and the tissue. Blood flow through vessels with diameters of 300 microns or less is different from arterial blood flow (Pries et al., 1996). Blood behaves as a non-Newtonian fluid in microvessels. Flow properties of blood in microvessels depend strongly on the interactions of blood components with the flow, with each other and with the vessel walls. For instance, in small capillaries (less than or equal to single cell size), red blood cells can undergo large deformations to pass through constrictions (Skalak and Branemark, 1969; Noguchi and Gompper, 2005). It’s well known that mechanical properties of red blood cells have a significant effect on blood viscosity (Bayliss, 1959; Albrecht et al., 1979; Pries et al., 1992; Coupier et al., 2008; Freund and Orescanin, 2011). Properties of blood flow in arteries and veins are different from the microcirculation system. In these flows, fluid behaves as a Newtonian fluid in an elastic tube which can adapt to changes in the flow and the pressure inside (Ku, 1997). Pumping of the heart creates flow in arteries. Flow resistance differs in different parts of the arterial system. Therefore, pressure and flow shapes are not uniform across the arterial system.

In vitro measurements and modeling of flow induced structural deformations in arterial blood flow developed in conjunction with experiments in deformable tubes (Heil and Jensen, 2003). In these experiments thin-walled axisymmetric soft rubber tubes are frequently used (Grotberg and Jensen, 2004) (e.g. Penrose tube (Holt, 1959)). The main parameter of the system at a given flow rate is the pressure difference between the inside and the outside of the tube (transmural pressure). In a number of experiments, it’s observed that changes in transmural pressure are accompanied by changes in the cross-sectional area of the tube (Holt, 1959; Katz et al., 1969; Conrad,
1969; Bertram and Godbole, 1995; Pedley et al., 1996; Brondum et al., 2009). This, in turn, affects flow geometry inside the tube. The final shape of the cross-section of the tube is determined by this coupled effect. It has been commonly observed that cross-sections of these tubes evolve from a buckled and collapsed state at zero flow rate to uniform circular cross-sections at high flow rates while tube walls are getting stiffer. Before cross-sections have become completely circular along the entire tube at medium flow rates, the cross-section is a function of axial position. At a given flow rate, cross-sectional area decreases with increasing distance from the upstream end of the tube. The tube's cross-sectional shape transitions from uniform circular to buckled and collapsed gradually with decreasing flow rate (i.e. transmural pressure). Collapse occurs in the tube when the local transmural pressure is around or below zero (Grotberg and Jensen, 2004). The interaction between the flow and the deformable tube wall provides a measure of the changes in transmural pressure as a function of changes in cross-sectional area at low flow rates (i.e. small transmural pressure). At high flow rates changes in axial wall curvature occur too due to higher transmural pressure. One dimensional time dependent models (Pedley and Lou, 1998) based on these arguments are frequently used in a wide range of collapsible tube experiments (Bertram, 2009).

1.2 Flexible Microfluidics

Similar to collapsible tubes, most microfluidic devices are made up of flexible materials, e.g., silicone (PDMS)(Whitesides and Stroock, 2001). As a result, the channel walls distend under flow, increasing the cross-sectional area with increasing flow rate (i.e. transmural pressure). This provides the possibility of probing a flow by monitoring the response of the confining micro-channel to the flow. In other words, the local (position-dependent) deflection \( \zeta \) of the deformable walls of a micro-channel
may enable the accurate determination of the pressure field (or the velocity field) under flow. The challenge in this approach, of course, is characterizing the interactions between a deformable body and a flow (Holmes et al., 2013; Hosoi and Mahadevan, 2004; Shelley et al., 2005). This is not a simple task, especially in the limit of large deflections. To accurately predict a flow bounded by a deformable wall, one must determine the hydrodynamic fields as well as the wall deformations consistently. This requires solving coupled fluid-structure equations (Heiland Jensen, 2003; Pedley and Lou, 1998), often in situations where constitutive relations or parameters describing fluid-structure interactions are not available. Even if these relations and parameters are known, numerical approaches are often expensive. The recent interest in fluid-structure interactions in microfluidic systems stems from the possibility of measuring pressure distribution in flexible microfluidic devices ((Gervais et al., 2006; Hardy et al., 2009; Orth et al., 2011; Song and Psaltis, 2011)).

Flows through flexible microfluidic channels are common in experiments relating to biological processes. For instance, in the study of cell biology, many groups are interested in using microscale tissues to understand how physical forces affect tissue function (Nelson and Gleghorn, 2012; Paszek et al., 2005). In many cases (e.g., tumor progression), it is well-known that changes in interstitial fluid pressure (IFP) accompany changes in tissue function, but cause-and-effect remains difficult to determine in vivo (Swartz and Fleury, 2007; Jain, 1999). Likewise, other groups have investigated the dependence of the transport processes through microscale tissues on pressure gradients (Choi et al., 2007; Helm et al., 2005). Along these lines, extracting the IFP in flows in microscale hydrogels is important for several applications. Microfluidic devices that contain microscale tissues can provide the opportunity to examine the effects of IFP on biological processes in a more controlled environment. Direct measurements of the flow fields (e.g., the pressure field) are of paramount importance for
understanding and predicting such flows.

1.3 Superhydrophobicity

Fluid-structure interactions manifest themselves not only in deformations of solid bodies but at the microscopic scale as well. In the case of flow over a solid body, fluid-structure interactions at the solid-fluid boundary depend on the microscopic interactions of fluid molecules with the solid surfaces and cannot be universal. In flow past rigid bodies, it's typically assumed that the no-slip boundary condition is valid, in which the fluid velocity is equal to the solid velocity. In real fluid systems, however, when fluid has a tangential velocity at the solid-fluid boundary, there is some degree of slip over the solid. It is well-established that the no-slip boundary condition can be violated (Lauga et al., 2007) on both hydrophobic (Vinogradova, 1995; Bouzigues et al., 2008; Tyrrell and Attard, 2001; Zhang et al., 2007) and superhydrophobic surfaces (Cottin-Bizonne et al., 2003; Choi and Kim, 2006; Joseph et al., 2006; Davis and Lauga, 2009). The consequences of a shift in boundary condition from no-slip to partial-slip are vast. Many natural organisms benefit from water repellent body parts (Neinhuis and Barthlott, 1997; Bush et al., 2007; Gao and Jiang, 2004). Slip flows are expected to impact technology by enabling drag reduction in both laminar (Bouzigues et al., 2008) and turbulent flows (Daniello et al., 2009).

1.4 Organization

In order to familiarize ourselves with the material and geometry of deformable walls used in Chapter 3, initially, we study membrane elasticity in experiments with wrinkled membranes. In our experiments, we use silicon nitride (SiN) membranes because of their easy fabrication and well-known material properties. The membranes have the same lateral dimensions and thicknesses as the ones used in our other experi-
ments in Chapter 3. The difference is that, in this experiment, membranes are not flat but wrinkled. We monitor deformations of wrinkled membranes during inflation with hydrostatic pressure. In Chapter 2, we first derive the elastic energy of a thin plate. Then, we find the membrane limit of this energy by considering large deformations of the plate. In this limit, the bending rigidity of the membrane is negligible and deformation is dominated by stretching (Ventsel and Krauthammer, 2001). Our experiments on inflation of wrinkled membranes show a different behavior from flat membranes. We attribute this deviation to the non-zero bending modulus of the wrinkled membrane. We support our argument with a geometric model that considers the total radius of curvature to be constant while the wrinkles are opening. Our experiments agree with the geometric scaling relation obtained from this model. Our results indicate that the transition from wrinkled to the unwrinkled state happens without stretching, and that bending is the dominant mode of deformation. Similar deformations on wrinkled shell structures have been observed (Aumaitre et al., 2013).

In Chapter 3, we demonstrate our method (Ozsun et al., 2013) for measuring the internal pressure distribution in a flexible microfluidic channel by exploiting channel wall deformations under flow. In this work, we employ standard planar microchannels, which have top walls made of thin membranes of silicon nitride or PDMS and rigid bottom walls. Our method is based on the measurement of the membrane distension as a function of applied uniform hydrostatic pressure; this initial measurement generates “calibration curves” for the deformable channel. The distension profile under flow is then matched to these calibration curves, providing the hydrodynamic pressure distribution. In Chapter 4, we extend our non-invasive technique to the measurement of the IFP field within hydrogels in PDMS-based microfluidic devices.

In chapter 5, we perform drag measurements on porous superhydrophobic mem-
branes both in high frequency oscillatory flows (Rajauria et al., 2011) and pressure driven flows in deformable microchannels. First, we present our experiments on Stokes’ second flow over porous superhydrophobic membranes with different solid fractions at atmospheric pressure conditions. Our results indicate that starting around 90% solid fraction, drag friction decreases anomalously with decreasing solid fraction. We consider using these porous structures as superhydrophobic walls in microchannels. We show our drag measurement results in pressure driven flows in microchannels with porous superhydrophobic membrane walls and conclude with future directions.

In chapter 6, we study fluid-structure interactions in the context of pressure dependent heat transfer from a heated oscillating beam. We propose a non-contact gas pressure sensing mechanism based on the resonance frequency shift of a fixed-fixed bilayer beam with changes in its temperature.
Chapter 2

Inflation of Wrinkled Membranes under Hydrostatic Pressure

2.1 Introduction

Wrinkles form on the surface of a material as a result of mechanical instabilities. The characteristic parameters of wrinkles, i.e., the amplitude and the wavelength, depend upon the linear dimensions and the material properties of the layer and applied forces. Wrinkle patterns with greatly different length scales in different material systems appear to follow the same scaling relationships. Recent experiments and analyses suggest that this scaling for wrinkles remains valid over a length scale extending over $\sim 10$ orders of magnitude — observable in an engineered nano structure (Vandeparre et al., 2011) as well as the earth’s crust (Slim et al., 2009). Recent interest in wrinkles stems from the fundamentally interesting nature of the problem as well as from applications of wrinkled structures in technology. Wrinkles have allowed for creative approaches in designing a variety of novel structures (Mei et al., 2010). Simply measuring the characteristic parameters of wrinkles in a layer provides information about the material properties of the layer and insight into the underlying forces in the medium.

A first-pass discussion of wrinkling starts with the Euler buckling instability (Roman and Pocheau, 1999). Consider an unconstrained thin elastic plate with linear dimensions $l \times w \times t$ ($l, w \gg t$) subjected to a compressive load along its length. Under
a large enough load, the plate will tend to deform in the out-of-plane direction with a wavelength twice as long as its length. Buckling allows the plate to reduce its overall elastic energy because bending a thin elastic structure requires much less energy than compressing it. If large-amplitude long-wavelength deformations are prohibitive due to constraints, a more complicated buckled state with shorter-wavelength wrinkles will emerge. To fully describe such a wrinkled state, one must consider the Föppl-von Karman equations (Audoly and Pomeau, 2010), which take into account large deformations of a flat plate with both bending and stretching. The Föppl-von Karman equations have been employed, for example, to accurately describe the deformations of a clamped rectangular sheet subjected to biaxial in-plane loads (Audoly et al., 2002). In most cases, however, these coupled nonlinear equations are impossible to solve analytically; thus, researchers have resorted to semi-analytical and numerical approaches. An intuitive approach is to minimize the elastic energy of the plate subject to constraints. This approach by Cerda and Mahadevan (Cerda and Mahadevan, 2003) has provided simple scaling laws, which have been observed on wrinkles in different material systems. Wrinkles on stretched elastic sheets (Géminard et al., 2004), on films on soft substrates, on bilayer materials (Concha et al., 2007), on reinforced membranes (Takei et al., 2011) and on floating thin films acted on by capillary forces (Huang et al., 2007) all appear to obey these scaling laws obtained from energy minimization.

While the onset of wrinkling has received much attention from researchers, relatively few studies exist on the evolution of wrinkles under applied mechanical forces. Recent efforts to characterize the response of a wrinkled state to forces, especially to pressure, come from several different disciplines. In analyzing the response of an elastic shell to a point force, Vella and co-workers (Vella et al., 2011) have shown that the point-force gives rise to a deformation, which transitions from a polygonal local-
ization to wrinkles, as the pressure increases. At the high pressure limit, the scaling relationships of Cerda and Mahadevan (Cerda and Mahadevan, 2003) is recovered. Complementary to this study on spherical shell rigidity, the shape-dependent rigidity of non-spherical shells has been studied experimentally (Lazarus et al., 2012) and theoretically (Vella et al., 2012) under similar loading conditions. Wrinkles on thin shell structures such as microcapsules (Walter et al., 2001) and vesicles (Knorr et al., 2010) may form and evolve under externally applied forces. Here, efforts have mostly been focused on understanding the time-dependent evolution of wrinkled structures in a flow due to velocity gradients (Kantsler et al., 2007; Turytsin and S., 2008). It has been shown that wrinkles can be used in estimating the bending stiffness of elastic bubbles and capsules with different geometries and material compositions (Knoche et al., 2013). In these experiments, water bubbles coated with protein molecules form an elastic film, and wrinkles appear on their surfaces with compression (Aumaitre et al., 2013).

Similar to these studies, here we study the mechanical response of thin wrinkled membranes under hydrostatic pressure. In particular, we measure the structural parameters, such as the average deflection of the membrane, and the wavelength and the amplitude of the wrinkles — all as a function of the applied pressure. Similar to the arguments of Aumaitre and co-workers (Aumaitre et al., 2013), we argue that during the transition from the wrinkled to the unwrinkled state, the surface area stays constant and the membrane does not stretch. We support our argument with a geometric model that considers total radius of curvature change of the membrane during inflation. Geometric scaling relations emerging from our model agree well with our experimental measurements. Our experiments and analysis show that wrinkled membranes open out through isometric deformations, providing a material architecture at the micro/nano scale for devices and an experimental venue for fundamental
2.2 Theory

2.2.1 Elastic Energy of a Thin Plate

A cubic solid element with stress components $\sigma_{ij}$ ($i, j = x, y, z$) is shown in figure 2.1. The total mechanical force for the entire solid can be written as a sum of the interior forces, $F$, and the body forces, $G$, (Audoly and Pomeau, 2010)

$$F_i + G_i = \int \int_{S} \sigma_{ij} n_{ij} dS + \int \int_{V} \rho g_i dV, \quad (2.1)$$

such that using the divergence theorem,

$$F_i + G_i = \int \int \int_{V} \left( \frac{\partial \sigma_{ij}}{\partial x_j} + \rho g_i \right) dV. \quad (2.2)$$

In equilibrium, the sum of all forces should be equal to zero. This condition is satisfied for each solid element if the integrand in equation (2.2) is zero:

$$\frac{\partial \sigma_{ij}}{\partial x_j} + \rho g_i = 0. \quad (2.3)$$

The work done by the interior force, $W_F$, on the cubic element can be calculated from the work needed to displace the cubic element by an infinitesimal virtual displacement, $\delta_i$. Using methods of virtual work (Hibbeler, 2010) and the divergence theorem we write

$$W_F = \sigma_{ij} n_{ij} \delta_i \quad \begin{equation} \begin{aligned} = \left( \frac{\partial (\sigma_{ij} \delta_i)}{\partial x_j} \right) \\
= \frac{\partial \sigma_{ij}}{\partial x_j} \delta_i + \sigma_{ij} \frac{\partial \delta_i}{\partial x_j}. \end{aligned} \end{equation} \quad (2.4)$$
In the absence of body forces, the first term is equal to zero in mechanical equilibrium from equation (2.3). From the symmetry of the stress tensor (Timoshenko and Goodier, 1951), $\sigma_{ij} = \sigma_{ji}$ and the elastic energy for the cubic element in figure 2.1a can be written as:

$$e_{el} = W_F = \frac{1}{2} \sigma_{ij} \varepsilon_{ij}, \quad (2.5)$$

where

$$\varepsilon_{ij} = \frac{1}{2} \frac{\partial \delta_i}{\partial x_j} + \frac{\partial \delta_j}{\partial x_i} \quad (2.6)$$

is the linear strain. Equation (2.5) can be reduced to two dimensions in the case of a thin plate with thickness $t$ (figure 2.1b). The lower and upper surfaces of the plate are stress free:

$$\sigma_{xz}(x, y, \pm t/2) = \sigma_{yz}(x, y, \pm t/2) = \sigma_{zz}(x, y, \pm t/2) = 0. \quad (2.7)$$

From the membrane theory of plates, the normal shear and transverse stress components should be close to their values on free surfaces (Audoly and Pomeau, 2010) (figure 2.1b)

$$\sigma_{xz}(x, y, z) \approx \sigma_{yz}(x, y, z) \approx \sigma_{zz}(x, y, z) = 0. \quad (2.8)$$

In order to distinguish between the three-dimensional and two-dimensional equations, the indices are changed from $i, j$ to $\alpha, \beta$ in the two-dimensional case. The elastic energy density in equation (2.5) reads:

$$e_{el} = \frac{\sigma_{\alpha\beta} \varepsilon_{\alpha\beta}}{2} = \frac{\sigma_{xx} \varepsilon_{xx} + \sigma_{yy} \varepsilon_{yy} + 2\sigma_{xy} \varepsilon_{xy}}{2}. \quad (2.9)$$
From two-dimensional constitutive relations the stress-strain relation for linear isotropic materials reads (Timoshenko and Goodier, 1951):

\[
\sigma_{xx}(x, y) = \frac{E}{1 - \nu^2} (\epsilon_{xx}(x, y) + \nu \epsilon_{yy}(x, y)), \\
\sigma_{yy}(x, y) = \frac{E}{1 - \nu^2} (\epsilon_{yy}(x, y) + \nu \epsilon_{xx}(x, y)), \\
\sigma_{xy}(x, y) = \frac{E}{1 + \nu} \epsilon_{xy}(x, y).
\] (2.10)

Eliminating stress in favor of the strain in equation (2.10), we write the energy density:

\[
e_{el} = \frac{E}{2(1 - \nu^2)} ((\epsilon_{xx} + \nu \epsilon_{yy})\epsilon_{xx} + (\epsilon_{yy} + \nu \epsilon_{xx})\epsilon_{yy} + 2(1 - \nu)\epsilon_{xy}^2) \\
= \frac{E}{2(1 - \nu^2)} ((\epsilon_{xx} + \epsilon_{yy})^2) - 2(1 - \nu)(\epsilon_{xx}\epsilon_{yy} - \epsilon_{xy}^2).
\] (2.11)

We can calculate the energy of the thin plate by integrating the energy density, equation (2.11), first with respect to z and then over the surface. Before that, we
must find the strain on the center surface, \( \varepsilon_{\alpha \beta}^g \), by considering how far two material points drift apart from each other during stretching of the center surface.

**2D Strain: \( \varepsilon_{\alpha \beta}^g(x, y) \)**

Consider the two points on the center surface separated by a distance \( x \) in figure 2.2a. Stretching in the \( x \) direction moves the points apart by a distance \( u \). The total linear strain becomes \( \Delta u / \Delta x \). In figure 2.2b, the stretching causes the points to drift apart both in the \( x \) and transverse directions. In this case, from Pythagorean theorem, the strain reads, \( \frac{\Delta u}{\Delta x} + \frac{1}{2} (\frac{\Delta \zeta}{\Delta x})^2 \), where \( \Delta u = u_2 - u_1 \) and \( \Delta \zeta = \zeta_2 - \zeta_1 \). The second term is the additional nonlinear term due to out of plane deflection. The same applies in
the y - z plane, too. Then, the total strain of the center surface can be written as:

\[
\varepsilon_{\alpha\beta}(x, y) = \frac{1}{2} \left( \frac{\partial u_\alpha}{\partial x_\beta} + \frac{\partial u_\beta}{\partial x_\alpha} \right) + \frac{1}{2} \left( \frac{\partial u_x}{\partial x_\alpha} \frac{\partial u_x}{\partial x_\beta} + \frac{\partial u_y}{\partial x_\alpha} \frac{\partial u_y}{\partial x_\beta} + \frac{\partial \zeta}{\partial x_\alpha} \frac{\partial \zeta}{\partial x_\beta} \right),
\]

(2.12)

where \( u_x(x, y) \) and \( u_y(x, y) \) and \( \zeta(x, y) \) are in plane and transverse displacements, respectively.

In equation (2.12), the first two non-linear terms can safely be neglected since they are quadratic functions of the linear term \( \partial u_\gamma / \partial x_\alpha \). However, the third non-linear term depends on the deflection, \( \zeta \). We can’t neglect it and the stretching strain reads:

\[
\varepsilon_{xx}^{es}(x, y) = \frac{\partial u_x(x, y)}{\partial x} + \frac{1}{2} \left( \frac{\partial \zeta(x, y)}{\partial x} \right)^2,
\]

\[
\varepsilon_{xy}^{es}(x, y) = \frac{1}{2} \left( \frac{\partial u_x(x, y)}{\partial y} + \frac{\partial u_y(x, y)}{\partial x} \right) + \frac{1}{2} \frac{\partial \zeta(x, y)}{\partial x} \frac{\partial \zeta(x, y)}{\partial y},
\]

(2.13)

\[
\varepsilon_{yy}^{es}(x, y) = \frac{\partial u_y(x, y)}{\partial y} + \frac{1}{2} \left( \frac{\partial \zeta(x, y)}{\partial y} \right)^2.
\]

**Bending Strain:** \( \varepsilon_{\alpha\beta}^{es}(x, y) \rightarrow \varepsilon_{\alpha\beta}(x, y, z) \)

During bending, the distance between two points on the center surface does not change. However, the distance between two neighboring points increases above the center surface and decreases below it (figure 2.2d). This leads to a non-zero energy density even though the center surface stays unaffected. Since the plate is thin, this energy is not likely to shear the plate in the transverse direction and the linear strain \( \varepsilon_{\alpha z} = 0 \):

\[
\varepsilon_{\alpha z} = \frac{1}{2} \left( \frac{\partial u_x}{\partial z} + \frac{\partial u_z}{\partial x} \right) = 0,
\]

(2.14)

where \( u_z \) is independent of \( z \) such that \( u_z(x, y, z) \approx u_z(x, y, 0) = \zeta(x, y) \) and \( \partial u_z / \partial z = -\partial \zeta(x, y) / \partial x \). First, integrating along \( z \), then taking the gradient along the in-plane
directions, the total strain becomes:

$$\epsilon_{\alpha \beta}(x, y, z) = \epsilon_{\alpha \beta}^c(x, y, 0) - z \frac{\partial^2 \xi}{\partial x_\alpha \partial x_\beta}.$$  

(2.15)

Bending enters into the total strain by establishing the connection between the variation of the in-plane strain, $\epsilon_{\alpha \beta}$, across the thickness and the curvature of the central surface.

**Total Elastic Energy: Integral of the Energy Density**

Let $\mathbf{\epsilon}$ be the vector containing the in-plane strain components such that $\mathbf{\epsilon} = \{\epsilon_{xx}, \epsilon_{yy}, \epsilon_{xy}\}$. Over a domain $z \in [-t/2, t/2]$, the in-plane strain vector takes the form (Audoly and Pomeau, 2010):

$$\mathbf{\epsilon}(z) = \mathbf{\epsilon}_0 + z \mathbf{\epsilon}_1,$$  

(2.16)

where $\mathbf{\epsilon}_0$ is the average value of $\mathbf{\epsilon}$ and $\mathbf{\epsilon}_1$ is its linear part with $z$. A symmetric matrix $Q$ maps the in-plane strain $\mathbf{\epsilon}$ to the energy density given in equation (2.11) such that:

$$\epsilon_{el} = \mathbf{\epsilon}(z) \cdot Q \cdot \mathbf{\epsilon}(z).$$  

(2.17)

Then the integral of the energy density over thickness takes the form:

$$\int_{-t/2}^{t/2} dz \mathbf{\epsilon}(z) \cdot Q \cdot \mathbf{\epsilon}(z) = \int_{-t/2}^{t/2} dz (\mathbf{\epsilon}_0 + z \mathbf{\epsilon}_1) \cdot Q \cdot (\mathbf{\epsilon}_0 + z \mathbf{\epsilon}_1)$$

$$= \mathbf{\epsilon}_0 \cdot Q \cdot \mathbf{\epsilon}_0 \int_{-t/2}^{t/2} dz + \mathbf{\epsilon}_1 \cdot Q \cdot \mathbf{\epsilon}_1 \int_{-t/2}^{t/2} z^2 dz$$

$$= t \mathbf{\epsilon}_0 \cdot Q \cdot \mathbf{\epsilon}_0 + \frac{t^3}{12} \mathbf{\epsilon}_1 \cdot Q \cdot \mathbf{\epsilon}_1.$$  

(2.18)

Note that the cross terms $\mathbf{\epsilon}_0 \cdot Q \cdot \mathbf{\epsilon}_1$ are linear in $z$ and cancel due to symmetry of the integration domain. Hence, the stretching and bending energies of a plate are
uncoupled. The constant term, $\varepsilon_0$, appearing in the first term on the right hand side of equation (2.18) contains the in-plane strain components along the center surface $z = 0$, $\varepsilon_0 = \{\varepsilon_{xx}, \varepsilon_{yy}, \varepsilon_{xy}\}_{z=0}$. On the other hand, the second term has the linear component $\varepsilon_1$, which contains the curvature terms, $\varepsilon_1 = \{-\partial \zeta_{xx}, -\partial \zeta_{yy}, -\partial \zeta_{xy}\}$. Using equations (2.11, 2.17, 2.18) and the definition of $\varepsilon_0$ and $\varepsilon_1$, the integral of the volumetric energy density across the thickness of the plate takes the form:

$$
\int_{-t/2}^{t/2} \varepsilon_{el} dz = \frac{Et}{2(1-\nu^2)} \left[ (\varepsilon_{xx} + \varepsilon_{yy})^2 - 2(1-\nu)(\varepsilon_{xx}\varepsilon_{yy} - \varepsilon_{xy}^2) \right]_{z=0} + \frac{Et^3}{24(1-\nu^2)} \left[ \left( \frac{\partial^2 \zeta}{\partial x^2} + \frac{\partial^2 \zeta}{\partial y^2} \right)^2 - 2(1-\nu) \left( \frac{\partial^2 \zeta}{\partial x^2 \partial y^2} - \left( \frac{\partial^2 \zeta}{\partial x \partial y} \right)^2 \right) \right].
$$

(2.19)

Integrating equation (2.19) over the in-plane variables $x$ and $y$ gives the total elastic energy $\varepsilon_{el}$ as a sum of stretching and bending energies, $\varepsilon_{el} = \varepsilon_s + \varepsilon_b$. Finally, the integral form of the stretching and bending energies are given below in equation (2.20):

$$
\varepsilon_s = \frac{Et}{2(1-\nu^2)} \int \int dxdy \left[ (\varepsilon_{xx} + \varepsilon_{yy})^2 - 2(1-\nu)(\varepsilon_{xx}\varepsilon_{yy} - \varepsilon_{xy}^2) \right], \quad (2.20a)
$$

$$
\varepsilon_b = \frac{Et^3}{24(1-\nu^2)} \int \int dxdy \left( (\Delta \zeta)^2 - 2(1-\nu) \left[ \frac{[\zeta, \zeta]}{2} \right] \right), \quad (2.20b)
$$

where $\Delta = (\partial^2 / \partial x^2 + \partial^2 / \partial y^2)$ is the two-dimensional harmonic operator and $[.,.]$ is the differential operator which is defined for two arbitrary functions $U(x,y)$ and $V(x,y)$ as:

$$
[U, V] = \left( \frac{\partial^2 U}{\partial x^2} \frac{\partial^2 V}{\partial y^2} + \frac{\partial^2 V}{\partial x^2} \frac{\partial^2 U}{\partial y^2} - 2 \frac{\partial^2 U}{\partial x \partial y} \frac{\partial^2 V}{\partial x \partial y} \right).
$$

(2.21)
2.2.2 Membrane Approximation

In this section we consider the out-of-plane deflection of a rectangular plate under the action of a uniform pressure load normal to the plate surface. A mode shape describing the deflected profile $\zeta$ of a thin bounded plate in the $x-y$ coordinate system can be written as:

$$
\zeta(x, y) \approx \zeta_{\text{max}} \sin \frac{\pi y}{w} \sin \frac{\pi x}{l},
$$

where $\zeta_{\text{max}}$ is the maximum deflection and $l, w$ are the length and width of the plate. In equation (2.20b) the first term in parenthesis is the square of the mean curvature, $\kappa_\zeta^2 = (\Delta \zeta)^2$, and the second term is the Gauss curvature $K = [\zeta, \zeta]/2$. The Gauss curvature term is associated with the stretching occurring during bending. It becomes important in bending cases where high curvature content is localized in a small region on the plate, such as during the process of creasing (Aharoni and Sharon, 2010). In the case of the bending of a flat plate without creasing, $K \ll \kappa_\zeta^2$ and can be neglected.

Then, the surface density of the bending energy $\varepsilon_b \sim D\kappa_\zeta^2$, where $D = E\ell^3/12(1 - \nu^2)$ is the bending modulus. Similarly, from equation (2.20a), the surface density of the stretching energy $\varepsilon_s \sim S\epsilon_s^2$ where $S = E\ell/2(1 - \nu^2)$ is the stretching stiffness and $\epsilon_s$ is the stretching strain. Finally, the potential energy (work) per unit area due to the applied pressure is $\varepsilon_p \sim p\zeta$. Then the energy equilibrium per unit area reads:

$$
p\zeta \sim \frac{D}{R_\zeta^2} + S\epsilon_s^2,
$$

where, $R_\zeta = 1/\kappa_\zeta$ is the radius of curvature. We estimate the order of magnitude of $\epsilon_s$ from figure 2·2 using equation (2.12). We assume that there is an initial constant strain in the plate $\gamma_t$. During pressurization, the plate deflects and the curvature of the plate changes dramatically resulting in a displacement between the two points in the out-of-plane direction. We write the stretching strain as a sum of the constant and
nonlinear strains, \( \epsilon = \gamma + \frac{1}{2} \left( \frac{\partial \gamma}{\partial t} \right)^2 \sim (\gamma + \frac{\gamma^2}{L^2}) \). We note that the non-linear strain is geometric and material properties are still in the linear elastic regime. The curvature of the plate \( \kappa = \frac{\partial^2 \xi}{\partial x^2} \sim \frac{\xi}{L^2} \). Here \( L \) is a planar dimension of the plate geometry (in this case it’s the width of the membrane since \( l/w \approx 10 \) in our experiments).

In principle, the equilibrium equations for the plate can be obtained by minimizing the free energy with respect to \( \zeta \), i.e., \( \frac{\delta E}{\delta \xi} + \frac{\delta E}{\delta \xi} + \frac{\delta E}{\delta \xi} = 0 \). Then the equation of mechanical equilibrium between the applied pressure and the \( \zeta \) (averaged over the entire plate area \( l \times w \)) can be written in non-dimensional form:

\[
\frac{pw}{Et} \sim \frac{1}{1 - \nu^2} \left( \frac{t^2}{w^2} + \gamma \right) \frac{\bar{\zeta}}{w} + \frac{1}{(1 - \nu^2) w^3}.
\]  

(2.24)

Here, the first term stands for bending and the second term for stretching. In the case of \( \gamma = 0 \), bending effects are important when \( \frac{t^2}{\xi^2} \gg 1 \). However, for thin films, \( \xi \gg t \) throughout the entire inflation. Therefore, inflation of thin plates is dominated by stretching and can be treated with the membrane approximation (Vlassak and Nix, 1992). Membranes are thin plates with vanishing flexural rigidity. They carry load through axial tensile stresses at their mid-plane (Ventsel and Krauthammer, 2001). The membrane theory of plates considers the energy of the center surface and assumes elastic energy is independent of the transverse direction. We will illustrate this fact with a simple example.

Before the pressurization starts, initially the average deflection \( \bar{\zeta} \approx 0 \) indicating radius of mean curvature \( R \approx \frac{1}{\kappa} \approx \infty \). With increasing pressure, the membrane attains a non-zero curvature with \( \bar{R} \) having a finite value. At this point, the flat membranes become a shell and bending cannot occur without stretching for closed shells (Landau and Lifshitz, 1986). Consider the circular cross-section of a thin closed shell with radius \( R \) as illustrated in figure 2-3a. Let the shell expand with a radial increase \( r \). Then the strain due to this expansion will be on the order of \( r/R \). Even
Figure 2-3: A model that describes the inflation of wrinkled shells without stretching. (a) A shell without wrinkles. An increase $r_\xi$ in the shell’s radius causes a strain on the order of $r_\xi/R_\xi$. (b) A shell with wrinkles. Material transfer is allowed between the shell and wrinkles. The same increase $r_\xi$ in shell’s radius is accompanied by a total decrease of $nr_w$ in all wrinkles’ radius with a net strain $\approx 0$. 
a tiny expansion will cause a non-zero strain unless \( R_\xi \approx \infty \). Therefore, the bending contribution in equation (2.24) can be neglected for thin plates with small \( \gamma_i \) and the pressure-deflection relation becomes \( p \sim \bar{\zeta}^3 \) in general.

### 2.2.3 Inflation of Wrinkled Membranes

Open shells, however can bend without stretching as in isometric deformations of developable surfaces (Audoly and Pomeau, 2010). The simplest, most common example is rolling a flat sheet into a cone or cylinder. In some special cases closed shells can bend without stretching too. Examples of this situation include a closed shell with a hole cut in it or a closed shell whose curvature changes sign across its surface (Landau and Lifshitz, 1986). The latter is studied here in the case of membranes with alternating curvatures on their surfaces, such as membranes decorated with wrinkles. The membrane can continue to deform without stretching until the wrinkles open out. The only way this can happen is if the change in the radius of mean curvature of the membrane, \( R_\xi \), is compensated for by the change in the wrinkles' total radius of curvature, \( R_w \propto \frac{n}{\kappa_w} \), where \( n \) is the number of wrinkles and \( \kappa_w \) is the curvature of a single wrinkle. We illustrate this situation in figure 2.3b. Consider the same shell in figure 2.3a, but this time its surface is decorated with wrinkles (solid circles) of radius \( R_w \). When the shell goes through a radial expansion \( R_\xi + r_\xi \), the wrinkles contract radially with a new radius \( R_w - r_w \). The total circumference change will be \( \sim r_\xi - nr_w \). Hence, expansion without stretching is possible if

\[
r_\xi \sim nr_w.
\]  

(2.25)

This relation can be written in terms of bending strains from the second term on the right in equation (2.15) with \( z = t \), the thickness of the membrane

\[
\frac{t}{R_\xi} \sim \frac{t}{nR_w}.
\]  

(2.26)
Equations (2.25) and (2.26) predict that inflation of a wrinkled membrane happens at constant strain until the wrinkles open out. We will test this prediction experimentally on thin rectangular wrinkled membranes in section 2.4.

2.3 Experimental System

2.3.1 Sample Structure

The structure under study is a thin rectangular membrane suspended from a thicker and larger solid frame as shown in figure 2.4a. The membrane occupies the middle region of the chip and is semi-transparent to light. All of the structures are made of silicon nitride (SiN) on silicon (Si). The structures are fabricated using a standard recipe, in which photolithography is followed by a KOH wet etch (Williams and Muller, 1996). The linear dimensions of the membranes are \( l \times w = 15.5 \text{ mm} \times 1.7 \text{ mm} \) with two different thicknesses of \( t = 1 \mu\text{m} \) and \( t = 200 \text{ nm} \); the solid frame is a 500-\( \mu\text{m} \)-thick Si substrate. As-fabricated plates are typically under tension and follow the membrane approximation (Rajauria et al., 2011).

These initially flat membranes are integrated into a custom built microfluidic chamber with inlet and outlet ports. Figure 2.4b shows the schematic of the microfluidic chamber. The inlet and outlet ports provide access for pressure gauges and pumps. Wrinkles are formed on the membranes by bending the originally-flat Si frame during clamping between the top and bottom plates of the microfluidic assembly. An image of a frame, after the chip has been deformed in a vise, is shown in figure 2.4c. Wrinkle patterns, which emerge on the membrane as a result of the bending, are shown in figure 2.4d. Both images of figure 2.4c and d are obtained using SWLI.

We obtain different wrinkle patterns by changing the linear dimensions (e.g., thickness) of the membrane. Our experimental system allows for tuning the wrinkle patterns by varying the strain levels through adjusting the tightness of the clamping
Figure 2.4: (a) Schematic of the cross-section and top view of sample structure. (b) Microfluidic chamber with inlet and outlet ports. (c) An optical image of the deformed Si frame. (d) The wrinkle pattern, which emerges as a result of the frame deformation. (e) A typical line scan along the x axis showing the deformation profile. The deflection of the membrane is $\zeta$, the wrinkle amplitude is $A$, wavelength is $\lambda$ and occupation length is $\Lambda$. 
Table 2.1: Initial structural parameters of the shells used in this study. All the shells are rectangular with $l \times w = 15.5 \text{ mm} \times 1.7 \text{ mm}$. The thicknesses $t$ vary. Also displayed are the number $n_i$, the wavelength $\lambda_i$ and the amplitude $A_i$ of the wrinkles.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$t$ (µm)</th>
<th>$n_i$</th>
<th>$\lambda_i$ (µm)</th>
<th>$A_i$</th>
<th>$p_i$ (Pa)</th>
<th>$\zeta_i$ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$S_1$</td>
<td>0.2</td>
<td>18</td>
<td>250</td>
<td>5.2</td>
<td>1320</td>
<td>1</td>
</tr>
<tr>
<td>$S_2$</td>
<td>1</td>
<td>4</td>
<td>910</td>
<td>17</td>
<td>1460</td>
<td>0.5</td>
</tr>
<tr>
<td>$S_3$</td>
<td>1</td>
<td>7</td>
<td>800</td>
<td>15.1</td>
<td>1300</td>
<td>-0.1</td>
</tr>
<tr>
<td>$S_4$</td>
<td>1</td>
<td>10</td>
<td>660</td>
<td>14.4</td>
<td>1575</td>
<td>-2.6</td>
</tr>
<tr>
<td>$S_5$</td>
<td>0.2</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1990</td>
<td>0.17</td>
</tr>
<tr>
<td>$S_6$</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1621</td>
<td>-0.4</td>
</tr>
</tbody>
</table>

screws in figure 2·4b (i.e., the deformation of the substrate). The parameters of the initial states — namely, the number $n_i$, the wavelength $\lambda_i$, the amplitude $A_i$ of the wrinkles for the the studied structures are displayed in Table 2.1. All the membranes have the same area $l \times w$. The first state is obtained on a thinner membrane; the next three states are obtained on the same structure by changing the strain; the fifth and sixth states are structures without wrinkles.

2.3.2 Measurements and Measurable Parameters

In the experiments, we apply hydrostatic pressure on the wrinkled membranes quasi-statically and record the position-dependent surface profile as a function of the applied pressure $p$ using SWLI.

There are three significant pressure-dependent structural parameters, which can be correlated with the mechanical behavior of this system. The first two are the average wavelength $\lambda$ and the average amplitude $A$ of the wrinkle patterns on the membranes. $A(p)$ and $\lambda(p)$ are extracted by typically analyzing line scans, such as the one shown in figure 2·4e. The final parameter is the deflection of the shell, $\zeta$, which is also shown in the line scan of figure 2·4e, where the dashed line corresponds to the deflection. After the wrinkles are ironed out, $\zeta$ corresponds to the maximum deflection (see figure 2·5h).
One point to note is that due to buckling of our membranes, the initial value of the deflection averaged along the entire line scan $\bar{\zeta}_i \neq 0$. The tangential stress in the membrane depends only on the curvature which is a function of $\zeta$ locally, or $\bar{\zeta}$ on average. To ensure that we start pressurization on flat sheets, we start our experiments at $\bar{\zeta}_i \approx 0$. At this initial state the gauge pressure readout $p_i > 0$. In order to measure the stress increase with curvature (i.e., $\bar{\zeta}$), we subtract this offset pressure from the measured pressures such that $p = p - p_i$. By doing so we set the initial condition for all experiments to be the same (at $p = 0$, $\bar{\zeta}(p) \approx 0$). The values of $p_i$ and $\bar{\zeta}_i$ for all the structures studied are given in Table 2.1.

The coordinate axes, which are used in our analysis are shown in figure 2.5. The $x-y$ axes are parallel to the sides of the rectangular structure as shown. The principal axes $X-Y$, on the other hand, are determined by the wrinkles, with $X$ being the direction of tension and $Y$ being the direction of compression.

2.4 Results and Discussion

Results from a typical measurement, where the pressure-dependent surface profile of a wrinkled membrane is captured optically, are displayed in figure 2.5a-g. At the lowest pressure, the deformation of the substrate determines the wrinkle patterns; the wrinkles appear uniformly distributed over the entire area of the rectangular structure, i.e., over $l \times w$. As the pressure is increased, the wrinkles tend to move to the central region of the structure, now occupying an area of $\Lambda(p) \times w$ and with changes to both wrinkle amplitude $A$ and wavelength $\lambda$, as seen in figure 2.5h. This new length scale, $\Lambda(p)$ (see figure 2.4a), which can be called occupation length, initially spans the length of the structure, becoming progressively smaller with increasing pressure — as displayed in figure 2.5h. After a critical pressure $p_c$ is exceeded, the wrinkles are completely ironed out (figure 2.5h).
Figure 2.5: (a)-(g) Typical surface profiles obtained during the pressurization of a wrinkled shell. The pressure increases from (a) to (g). (h)-(k) Line scans of the deflection profile along the $x-y$ and the $X-Y$ directions for various pressures. Note that the height scales in the plots are greatly exaggerated. In reality, all the deflections are quite small as compared to the linear dimensions of the structure.
At pressures higher than this critical pressure $p_c$, the structure appears to inflate — stretching similar to a membrane.

The cross-sectional line scans shown in figure 2.5h-k are obtained from the optical surface profiles. The height scales in the plots are greatly exaggerated. Along the $x$ direction (figure 2.5h), line scans show the following features. Initially, the wrinkle amplitude and wavelength do not change with pressure. In this limit, due to buckling, the membrane is extremely sensitive to pressure. Even tiny changes in pressure cause the membrane to deflect up. As the pressure keeps increasing the membrane becomes taut, and wrinkles start to unfold with an accompanied increase in deflection. During the process the wrinkles are pushed into the central region of the structure, occupying an area $\sim \Lambda \times w$. Both the amplitude and the wavelength of the wrinkles decrease, and eventually all the wrinkles become completely ironed out. Line scans along the $y$ (figure 2.5i) and $Y$ (figure 2.5k) directions show the ironing out of the wrinkles, but do not offer much insight into the physics because of the interference of the boundaries. Figure 2.5j shows a line scan taken along the tension direction $X$. The surface height increases monotonically with increasing $p$. All the line scans along the $X$ direction, including those taken at the minima of a wrinkle (e.g., inset), show this general trend.

Analyzing line scans similar to those in figure 2.5h-k, we can extract the structural parameters of the inflating membrane as a function of pressure $p$. A typical line scan used in our analysis is shown in figure 2.4a. The deflection of the membrane is $\zeta$, the wrinkle amplitude is $A$, the wavelength is $\lambda$ and the occupation length is $\Lambda$. Figure 2.6a shows membrane deflection averaged over the entire line scan, $\tilde{\zeta}(p)$, as a function of $p$. The insets in the plot show line scans along the $x$ direction at selected pressures. At the high pressure limit, the data converge to a power law, $\tilde{\zeta}(p) \propto p^{0.30}$ close to the analytically predicted value $\tilde{\zeta}(p) \propto p^{1/3}$. With reducing pressure, wrinkles
Figure 2.6: (a) average deflection $\tilde{\zeta}(p)$ (b) average amplitude and (c) the average wavelength of wrinkle patterns as a function of pressure. Note that the pressure range in (b) and (c) correspond to the range $p < p_c$ in (a). The insets in the plots show line scans along $x$ direction at different pressures.

appear, and the $\frac{d\tilde{\zeta}}{dp}$ changes. The average deflection $\tilde{\zeta}$, for this particular sample appears to follow another power law, $\tilde{\zeta} \propto p^m$, where $m = 0.78 \pm 0.02$. The error, however, is large at this limit due to the experimental difficulties in obtaining small pressure steps and resolving changes in average surface deflections in the presence of large wrinkles. In figure 2.6b&c, the average amplitude $A(p)$ and the average wavelength $\lambda(p)$ of the wrinkles are plotted, respectively. Note that the displayed pressure ranges in figure 2.6b&c, i.e., $p \lesssim 10^4$ correspond to the lower end of the pressure range in figure 2.6a. At the low pressure limit, both $A$ and $\lambda$ converge to flat lines, with very small changes. As $p \to p_c$, both $A$ and $\lambda$ decay to zero. Data from our other samples ($S_1 - S_4$) listed in Table 2.1 display similar trends. The amplitudes and wavelengths of the wrinkle patterns shrink in a qualitatively similar way to the behavior shown in figure 2.6b&c.

We first study the average deflection, $\tilde{\zeta}$, of the wrinkled membrane as a function of pressure, $p$. Since we have samples with two different thicknesses, from equation (2.24), we plot non-dimensional average deflection $\frac{\tilde{\zeta}}{w}$ as a function of non-dimensional pressure $\frac{\rho w^2}{EI}$ for all wrinkled samples in figure 2.7a. From the average of all four
samples, we find that in the high pressure limit wrinkled membrane deflection follows \( \bar{\zeta} \sim p^{0.3 \pm 0.01} \) in agreement with equation (2.24). Hence stretching is the dominant mode of deformation in this regime. In the low pressure limit, wrinkled membrane deflection follows another power law \( \bar{\zeta} \sim p^{0.88 \pm 0.15} \), and equation (2.24) still holds indicating bending to be the dominant mode of deformation in this regime.

We repeat the same experiment with two more SiN membranes without wrinkles on them (\( S_5 \) and \( S_6 \), see Table 2.1). As usual, the membranes buckle during clamping into the fluidic cell, but they are not further compressed and stretched to avoid formation of any wrinkles. A plot of the \( \bar{k}_w \) as a function of \( \frac{p w}{E t} \) of flat membranes are given in figure 2-7b. In the high pressure limit, similar to the case of wrinkled membranes, flat membranes follow \( \bar{\zeta} \sim p^{0.36 \pm 0.02} \) in agreement with equation (2.24). Hence, stretching is the dominant mode of deformation in this regime for flat membranes, too. However, in the low pressure regime, the data shows a lower slope for
Figure 2.8: (a) Number of wrinkles, $n$, as a function of pressure. Non-dimensional scaling relations given in equations (2.27) and (2.26) are plotted in (b) and (c) respectively. Arrows in (b) indicate where the unwrinkling starts.

deflection of flat membranes $\zeta \sim p^{0.74\pm0.07}$ indicating a higher stiffness. This is a reasonable result since the flat membranes are only slightly buckled during clamping and they are not as compressed as the wrinkled ones. This is more obvious in figure 2.7c where we compare all the wrinkled states with all the flat states. The dashed lines in figure 2.7c correspond to two asymptotes in equation (2.24). In both high and low pressure limits, both wrinkled and flat membranes tend to converge to these power laws. However where the transition starts and how the transition happens are different for these two sets. The arrow indicates roughly where the transitions start for wrinkled membranes. Flat membranes seem to transition to stretching earlier than the wrinkled membranes. The observed difference can be due to higher compressive stresses present in wrinkled membranes. Therefore, the wrinkled membranes are less stiff than the flat membranes until the wrinkles open out completely. Observation of the evolution of wrinkled membrane parameters in equation (2.25) during inflation can shed light into how the transition from the wrinkled state to the unwrinkled state occurs.

We start by observing the number of wrinkles, $n$, as a function of pressure in figure 2.8a for all wrinkled membranes $S_1 - S_4$. We see that $n$ stays constant throughout
the pressurization with a sudden drop towards the end. We approximate the number of wrinkles, \( n \), as a ratio of the occupation length to single wrinkle wavelength, \( n \approx \Lambda/\lambda \). Constant \( n \) implies that both \( \lambda \) and \( \Lambda \) either stay constant or change proportionally with pressure. We can understand what really goes on by observing line scans of figure 2.5h. Initially, both \( \lambda \) and \( \Lambda \) do not change at low pressures and \( n \) stays constant. Later, when the wrinkles start to merge into the mid-section of the membrane, both \( \lambda \) and \( \Lambda \) decrease proportionally still keeping \( n \) constant. However, later in the final stages of unwrinkling, occupation length decreases much faster than the single wrinkle wavelength. This can be seen in figure 2.8a as the number of wrinkles are decreasing sharply in final stages.

Next, we consider arc length conversation during unwrinkling discussed in section 2.2.3. We are particularly interested in seeing if our data agrees with the geometric scaling relations given in equations (2.25) and (2.26). These equations are obtained from a geometric consideration which suggests that during unwrinkling the total arc length of any cross section stays constant. In equation (2.25) we plug \( r_\zeta \sim R_\zeta \sim \frac{\lambda^2}{\zeta} \), \( r_w \sim R_w \sim \frac{\Lambda^2}{A} \), and \( n \sim \frac{\Lambda}{\lambda} \). We obtain the relation \( \frac{\lambda^2}{\zeta} \sim \frac{\Lambda^2 \lambda}{\Lambda} \). Rearranging gives us a nondimensional relation between the geometric parameters of the inflating wrinkled membrane:

\[
\frac{A}{\zeta} \sim \frac{\Lambda \lambda}{l^2},
\]

(2.27)

where \( A/\zeta \) is the non-dimensional amplitude and \( \Lambda/l \) and \( \lambda/l \) are two non-dimensional lengths of the system whose ratio determines the number of wrinkles. We plot equation (2.27) in figure 2.8b for \( S_1 - S_4 \). All the curves show a flat region in the low pressure limit when \( A/\zeta \rightarrow \infty \). Here, in the low pressure limit, initially the wrinkle parameters do not change, but the average deflection increases at low pressure (see line scans of figure 2.5h). Later when unwrinking starts, our data follow the linear relation given in (2.27). From figure 2.8a, we conclude that the occupation length
decreases faster than the wavelength. Using this information while evaluating figure 2.8b, we conclude that, similar to the occupation length, non-dimensional amplitude, $A/\zeta$, must be decreasing faster than non-dimensional wavelength, $\lambda/l$. In fact, from figures 2.6b&c, we see that in the same pressure range the wrinkle amplitude decays three times more than the wrinkle wavelength. We note that in figure 2.8a, the thinner membrane (sample $S_1$, $t = 200$ nm) does not collapse on to the data of 1 $\mu m$-thick membranes. This brings another geometric quantity, the thickness of the membrane, into consideration. Membrane thickness can be included into the geometric scaling relation in equation (2.27). This is simply done by considering (2.26) where we compare the bending strain of opening wrinkles with the bending strain associated with the decrease in membrane's mean radius of curvature. Using $R_\zeta$, $R_w$, $n$ and $t$ in equation (2.26), the non-dimensional geometric scaling relation including membrane thickness is written as

$$\frac{IA}{t\zeta} \sim \frac{\Lambda\lambda}{lt}.$$  \hspace{1cm} (2.28)

We plot equation (2.28) in figure 2.8c. The dashed line corresponds to linear slope. As familiar now, initially the wrinkle parameters do not change. After initial pressurization, unwrinkling starts and our data agrees well with equation (2.28) collapsing into one line. This gives us the confidence that we successfully included the thickness effect into the geometric scaling. Implications of the agreement between our data and equation (2.28) are two fold. First, this confirms our argument that unwrinkling happens at constant strain until the wrinkles are completely ironed out. Second, equation (2.28) provides a unique method of thickness measurement which we will discuss in the next section.
2.5 Conclusion

We studied the elastic response of wrinkled membranes experimentally under hydrostatic loading. Among the observed parameters, we considered average deflection, wrinkle amplitude and wrinkle wavelength as physical parameters of our model. We obtained the solution for the pressure vs. deflection profile from minimization of total energy. The solution contained two different power regimes, one representing the bending \( p \sim \zeta \) and the other representing stretching of the membrane \( p \sim \zeta^3 \). We showed that the solution is dominated by the stretching term for thin membranes.

In our experiments with thin membranes, we observed a deviation between deflection vs. pressure profiles of wrinkled and flat membranes. We attributed this deviation to the high compressive stress occurring during wrinkling. We argued that during unwrinking, wrinkled membranes do not stretch until all the wrinkles disappear since they are under high compression in one direction. This requires the condition that the total surface area stays the same during unwrinking, hence constant strain. With a geometric model we showed that constant strain condition at each cross section is satisfied when the change in the membrane's mean radius of curvature, \( \kappa_{\xi} \), is equal to the change in wrinkles total radius of curvature, \( \kappa_w/n \). This ensures that the arc length of the wrinkled membrane cross-section stays the same during unwrinking.

The scaling relation that emerged from this condition agrees well with our data. This indicates that transition from the wrinkled state to the unwrinkled state happens through isometric deformations without stretching. We further validate this result from the comparisons of bending strains and we successfully include the effect of thickness into the geometric scaling relation.

Our measurements can be a useful tool in determining thickness ratios of two samples made out of the same material. For instance, in our case, the thickness ratio of the thinner membrane to thicker membranes is \( t_1/t_2 = 200 \text{ nm} / 1 \mu\text{m} = \)
0.2. Simply by comparing the non-dimensional amplitudes, $A/\zeta$, at the onset of unwrinkling, indicated by arrows in figure 2.8b, we determine the thickness ratios to be $\approx 0.20\pm0.01$ which is in good agreement with the actual value. Similar calculations can be done by comparing the ordinate, $\Delta \lambda/l^2$, values at the onset of unwrinkling. In that case, we find a thickness ratio of $\approx 0.23 \pm 0.05$ which is still in good agreement with the actual value. We note that in the case of spherically symmetric shells as in figure 2.3, $n(p) \rightarrow n \approx constant$. This condition implies that $t \sim \lambda^2$ in agreement with calculation, measurements and predictions of Ref. (Wong and Pellegrino, 2006; Knoche et al., 2013; Erni et al., 2012). Measuring in plane length scales such as $\Lambda$ or $\lambda$ are easier than measuring out of plane length scales $A$ and $\zeta$. Without the need of interferometric equipment, they can be captured with a camera and analyzed by image processing. This approach can find applications in determining thicknesses of complex membrane structures such as membranes of bio-capsules (Walter et al., 2001; Erni et al., 2012). A final note is we can write the average deflection $\bar{\zeta}$ in terms of wrinkle parameters from geometric scaling. Using this in deflection vs. pressure relation and provided that we know the initial constant strain, $\gamma_i$, our measurements and analysis on wrinkled membranes can be useful in bending modulus estimation of thin flexible structures (Aumaitre et al., 2013; Knoche et al., 2013).

In the Chapters 3, 4 & 5, we will study fluid interactions with deformable bodies during pressure driven flows in deformable microchannels. In Chapter 3, we start with presenting our non-invasive method for measuring pressure distributions in deformable microchannels.

### 2.6 Instrumentation

We used scanning white light interferometry (SWLI) to measure the surface profile of the membranes. SWLI uses the same basic principle used in coherence scanning in-
Figure 2.9: A typical configuration of a scanning white light interferometer with illumination optics and Michelson type interferometric objective connected to a height scanner.

A broadband light source with a short coherence length illuminates the pupil of a Michelson interferometric objective through a beam splitter. This type of objective has a built-in beam splitter and a reference mirror. The light entering the objective is split into two parts with the built-in beam splitter. One beam reflects from the sample surface, the other reflects from a flat reference surface in the objective. Both beams are then recombined and imaged on a CCD camera. White light is composed of many wavelengths and has a short coherence length (CL), so constructive interference occurs only when the path lengths of the two beams are nearly equal. For
each pixel on the CCD, the modulation intensity of the interference signal is maximum when the two path lengths are exactly the same. Hence, scanning the sample objective vertically and recording the modulation intensity allows one to construct a profile map of the surface.

Since white light is composed of many wavelengths, the total interference signal on the CCD is a superposition of all the interference signals from all wavelengths. The contribution of each wavelength to the total interference signal is

$$g(\beta, k, z) = g_{DC}(1 + \cos(K(\beta, k)(\zeta - z))),$$

(2.29)

where $g_{DC}$ is a constant intensity, $K(\beta, k) = 2k\beta$ is called the fringe frequency (rad/$\mu$m), $k = 2\pi/\lambda$ is the wavenumber (1/m), $\beta = \cos(\Psi)$ is the directional cosine and $\Psi$ is the angle of incidence as shown in figure 2.9. Finally, $\zeta$ is the surface height that we want to measure and $z$ is the scan height of the objective. Superposition consists of summing all individual interference contributions over all angular wavenumbers and directional cosines with appropriate weighting functions that take into account factors such as illumination and detection spectrum, $V(k)$ and distribution of light in the pupil, $U(\beta)$. The interference intensity signal becomes

$$I(z) = \int_{0}^{\infty} \int_{0}^{1} g(\beta, k, z)U(\beta)V(k)\beta d\beta dk.$$  

(2.30)

A schematic of this superposition is illustrated in figure 2.10. Each wavelength of the source spectrum is mutually incoherent and has different fringe spacings. The sum of the individual intensities will have a maximum when all of them are in phase. The location at which this can happen is when the optical path difference (OPD) between the sample and reference arms is zero. This is the best focus position on the sample. Away from the zero OPD point, the total fringe intensity decays quickly localizing the interference to a narrow region. The processing of a signal of the form in figure can
Figure 2.10: White light consists of many wavelengths that are mutually incoherent and have different fringe spacings. Superposition of these wavelengths have a maximum when the optical path difference between the sample and reference arms is zero.

be achieved by treating it as a rapidly oscillating interference signal in an envelope in a narrow region \((\zeta - z)\). Then the normalized interference signal can be written as (Schmit et al., 2011):

\[
I(z) \propto 1 + \gamma(z)\cos(K_0 z),
\]

where \(\gamma(z)\) is called the modulation or the coherence (fringe) envelope and \(K_0\) is the central fringe frequency. The coherence length of the light source is determined by the width of the fringe envelope, \(\Delta\lambda\) such that \(CL \approx \frac{\lambda^2}{\Delta\lambda}\) (Schmit et al., 2011). In a typical SWLI, taking \(\lambda_0 = 500\) nm and \(\Delta\lambda = 100\) nm gives a CL of 2.5 \(\mu\)m.

A light source with a short coherence length confines the interference fringes to a narrow surface height range, and, therefore, is essential both for signal generation and efficient signal processing. A light source with a broad spectrum like white light is therefore suitable for this microscopy.

During a vertical scan, each pixel on the CCD receives an intensity with its highest
Figure 2.11: Image build-up on a lateral array of pixels on the CCD. The solid line is the surface profile to be measured. Each pixel on the CCD has a coherence envelope whose center is located at a height where the surface is in best focus.

Point determined by the fringe envelope. Figure 2.11 illustrates the fringe intensities measured by an array of pixels. Each pixel is assumed to receive the same signal at different vertical positions as the topography of the sample changes from pixel to pixel. The intensity information at each pixel is analyzed using signal processing algorithms to compute the envelope of the fringes. After computing the envelope, its position, and hence the surface height, \( h \), is found from calculation of the envelope's center of mass (Schmit et al., 2011):

\[
h = \frac{\sum_{i=1}^{N-1} \gamma_s \zeta_i}{\sum_{i=1}^{N-1} \gamma_s}.
\]  

(2.32)

Along with the other factors such as the coherence length, the vertical resolution of SWLI depends on the algorithm used to compute the fringe envelope (Deck and de Groot, 1994). In our case, the minimum vertical resolution is \(~ 20 \) nm. The lateral resolution depends on the Rayleigh criteria, \( 0.6 \lambda/NA \) and in our experiments, we used lateral resolutions in the range of \( 2 - 5 \mu m \).
Chapter 3

Non-Invasive Measurement of the Pressure Distribution in a Deformable Micro-Channel

3.1 Introduction

Since the early experiments of Poiseuille (Sutera and Skalak, 1993) more than two centuries ago, the craft of measuring flow fields in tubes and pipes has been perfected. Even so, the resolution limits of these exquisite experimental probes — such as the pitot tube (McKeon, 2007) or the hot wire anemometer (Bruun, 1995) — are quickly being approached, given recent advances in micron and nanometer-scale technologies. One frequently encounters micro- (Stone et al., 2004; Popel and Johnson, 2005) and nano-flows (Schoch et al., 2008), which come with smaller length scales (Lissandrello et al., 2012) and shorter time scales (Ekinci et al., 2008; Ekinci et al., 2010) than can be resolved by the commonly available probes. For instance, in a pressure-driven micro-flow, one must insert micron-scale pressure transducers in test sections in order to determine the local pressure drops (Hardy et al., 2009; Akbarian et al., 2006; Kohl et al., 2005; Orth et al., 2011; Song and Psaltis, 2011; Srivastava and Burns, 2007). As the size of a probe becomes comparable to or even bigger than the flow scale itself, measurement of the distribution of flow fields becomes problematic. Although the tools of macroscopic fluid mechanics may not easily be scaled down, the materials and techniques of micro-fluidics offer unique measurement approaches. Most
micro-channels in lab-on-chip systems, for instance, are made up of flexible materials (Whitesides and Stroock, 2001; Gervais et al., 2006) and can deform under flow.

Consider a steady pressure-driven flow between an infinite rigid plate at \( y = 0 \) and a deformable wall at \( y = 2h_0 + \zeta(x) \), where \( \zeta(x) \) is the local deflection of the deformable wall due to the local pressure \( p(x) \) as shown in figure 3.1a. The equations for incompressible steady flow \( (\partial_x u + \partial_y v = 0) \),

\[
\begin{align*}
\rho u \partial_x u + \rho v \partial_y u &= -\partial_x p + \eta (\partial_x^2 + \partial_y^2) u, \\
\rho u \partial_x v + \rho v \partial_y v &= -\partial_y p + \eta (\partial_x^2 + \partial_y^2) v,
\end{align*}
\] (3.1)

are to be solved subject to boundary conditions \( u|_B = v|_B = 0 \). All the variables in (3.1) have their usual meanings (see figure 3.1a), with \( \rho \) and \( \eta \) being the density and dynamic viscosity respectively. In general, solution to these equations, accounting for inlet and outlet effects is impossibly difficult. However, if the channel is long such that \( 2h_0 + \zeta_{\text{max}} \ll 1 \), where \( \zeta_{\text{max}} \) is the maximum deflection and \( l \) is the length of the channel, we can write the local solution for the average velocity \( \bar{u}(x) \) as

\[
\bar{u}(x) \approx \frac{1}{12\eta} [2h_0 + \zeta(p(x))]^2 \partial_x p.
\] (3.2)

Here, \( \zeta(x) = \zeta(p(x)) \) is a local constitutive relation, which determines the dependence of the wall deflection \( \zeta(x) \) on \( p(x) \). No particular form for this dependence (e.g., elastic) is assumed \textit{a priori}. In order to find the flow rate and the wall stresses, we need accurate information on \( p(x), \zeta(x) \), and the constitutive relation \( \zeta(x) = \zeta(p(x)) \). If the flow rate is given, the problem becomes somewhat simplified, but still remains rather complex to be handled numerically or analytically.

In this chapter, we describe a method to close (3.1) in a deformable channel using \textit{independent static} measurements of \( \zeta = \zeta(p) \). Using this method, we extract the pressure distribution in a planar channel flow and validate our measurements against
the analytic approximation in (3.2) for a long channel. Our method does not depend upon the particulars of the local constitutive relation $\zeta(x) = \zeta(p(x))$. In other words, it remains independent of the nature of the wall response, providing accurate results for buckled walls and elastically stretching walls alike.

### 3.2 Slowly Varying Channel Approximation

In the case of a pressure driven two dimensional steady flow in between two plates separated by a distance $2h_0$, fluid velocity vector, $u \hat{i} + v \hat{j}$, is unidirectional and independent of the flow direction (Batchelor, 2000). Then $u \to u(y)$ and $v = 0$. In this case, the non-linear inertial terms on the left hand side of Navier-Stokes equations in (3.1) vanish

$$ \rho u \partial_x u = \rho v \partial_y u = \rho u \partial_x v = \rho v \partial_y v = 0, $$(3.3)

and the equation governing the fluid flow becomes

$$ \frac{\partial^2 u}{\partial y^2} = -\frac{1}{\eta} \frac{dp}{dx}. $$(3.4)

Solution of the above equation with no-slip boundary conditions at the plates, $u(y = 0, 2h_0) = 0$, gives the local velocity profile across the distance between the plates:

$$ u(y) = \frac{1}{2\eta} \frac{dp}{dx} y(2h_0 - y). $$(3.5)

The total volume flux, $Q$, across a plane normal to the flow direction is found by integrating the fluid velocity across the distance between the plates:

$$ Q = \frac{1}{2\eta} \frac{dp}{dx} \int_0^{2h_0} y(2h_0 - y) \frac{dp}{dx} \frac{2wh_0^3}{3\eta}, $$(3.6)
where \( w \) is the width of the plane. The average velocity across the plane is given by
\[
\bar{u} = \frac{Q}{w2h_0} = \frac{1}{12\mu} \frac{dp}{dx} (2h_0)^2.
\]  
(3.7)

From equation (3.6), the total pressure difference, \( \Delta p \), between two planes normal to the flow direction and separated by a distance \( l \) is found by integrating the local hydraulic resistances along \( l \),
\[
Q \int_0^l r \, dx = \Delta p,
\]  
(3.8)

where \( r = \frac{3\eta}{2wh_0^3} \) is the total local hydraulic resistance across each plane and \( R = \int_0^l r \, dx = \frac{3\eta}{2wh_0^3} \) is the total hydraulic resistance between the two planes.

If the cross-section along the flow direction is not uniform as in figure 3.1a, \( 2h_0 \rightarrow 2h_0 + \zeta(x) \), then the streamlines are not unidirectional. In addition to axial component of the fluid velocity, \( u \), component of the velocity normal to the plates, \( v \), is non zero and has an order of magnitude \( \alpha u \). Here, \( \alpha \) is the magnitude of angle that the streamlines make with the axial direction and given by \( \frac{d(2h(x))}{dx} \), where \( 2h(x) = 2h_0 + \zeta(x) \) is the position dependent plate separation. Then the magnitude of the inertial forces can be estimated from the nonlinear terms in equation (3.1) such that
\[
\rho u \partial_x u \sim \rho u \partial_x v \sim \alpha u^2 / 2h(x).
\]
Comparing inertial forces with viscous forces, \( \eta \nabla^2 u \sim \eta u / (2h(x))^2 \), in equation (3.1), inertial forces can safely be neglected for slowly varying plate separations with sufficiently small \( \alpha = \frac{d\zeta(x)}{dx} \) such that
\[
\alpha \frac{\rho 2h(x)u}{\eta} \ll 1.
\]  
(3.9)

For a flow in a slowly varying channel, as long as equation (3.9) holds, the flow can be treated as Poiseuille flow locally. Then, the local hydraulic resistance in equation (3.8) and the average local velocity in equation (3.7) become functions of axial position as
2h₀ → 2h(x) such that r → r(x), equation (3.11), and u → u(x), equation (3.2).

3.3 Experimental System

To test these ideas, we have fabricated planar micro-channels with deformable walls and measured the deformations of these micro-channels using optical techniques under different conditions. Figure 3.1b is a rendering of one of our micro-channels under pressure. The inset shows how the channel is formed: a rigid bottom wall and a deformable top wall are held together by clamps, and the channel is sealed by a gasket. The in-plane linear dimensions of the channels are l × w = 15.5 × 1.7 mm². The distance 2h₀ between the undeflected top wall and the rigid bottom wall is set by a precision metal shim (in the range 100 μm ≤ 2h₀ ≤ 250 μm); optical interferometry is employed to independently confirm the 2h₀ values. Different materials with varying thicknesses t are used to make the deformable walls. In three of the channels studied here, the deformable walls are ultrathin SiN membranes fabricated on a thick silicon handle chip (t ≈ 500 μm). In the other channels, the compliant walls are made up of thicker elastomer (PDMS) layers. Various parameters of all the micro-channels used in this study are given in table 3.1.

After the micro-channels are formed, they are connected to a standard microfluidic circuit equipped with pressure gauges. In the hydrostatic measurements, the outlet of the micro-channel is clogged, and a water column is used to apply the desired pressure. In flow measurements, a syringe pump is inserted into the circuit to provide the flow.
Figure 3.1: (a) A one-dimensional channel with a deformable top wall. (b) The two-dimensional deflection $\zeta_{2d}(x, z)$ of S1 ($t=200$ nm, $2h_0 = 175$ $\mu$m) under hydrostatic pressure of $p = 2.3$ kPa as measured optically. The inset shows a cross-sectional view of the channel. Clamps hold the chip (with the thin membrane at the center) and the bottom wall together. The two walls are separated by a precision shim; an O-ring (black ovals) seals the channel. (c) Cross-sectional line scans from the image in (b) showing the parabolic $z$-profile of the deformable wall under pressure. (d) The one-dimensional (average) wall deflection $\zeta(x)$ at several different pressures for the same sample. The indicated values are gauge pressure values. These are the local constitutive curves. (e) The peak deflection $\zeta_p$ of the PDMS deformable walls as a function of pressure $p$. (f) $\zeta_p$ of the SiN deformable walls as a function of $p$; the dashed lines are the $p^{1/3}$ asymptotes. Note that the $\zeta_p$ and the $p$ axes do not cover the same ranges in (e) and (f). Error bars are smaller than the symbols.
3.4 Results and Discussion

3.4.1 Hydrostatic Loading

First, the channels are characterized under hydrostatic loading. In these experiments the inlet port is connected to a water column with the outlet clogged, and hydrostatic pressure \( p \) is applied on the channel by raising the water column. The resulting position-dependent deflection field \( \zeta_{2d}(x, z) \) of the compliant wall is measured using SWLI (Deck and de Groot, 1994) at each pressure. In figure 3·1b, \( \zeta_{2d}(x, z) \) of the deformable top wall of sample S\( \text{I} \) (\( t = 200 \) nm and \( 2h_0 = 175 \) \( \mu \)m; table 3.1 first row) at \( p = 2.3 \) kPa is shown. Cross-sections along the \( x \) and \( z \) axes taken from this profile are shown in figure 3-1c. Because the cross-sections are parabolic in the \( z \)-direction, we define an average or one-dimensional wall deflection \( \zeta(x) \) as

\[
\zeta(x) = \frac{1}{w} \int_{-w/2}^{+w/2} \zeta_{2d}(x, z) dz \approx \frac{2}{3} \zeta_{2d}(x, z = 0).
\]  

(3.10)

Here, \( \zeta_{2d}(x, z = 0) \) is the maximum value of the parabolic cross-section, and the factor \( \frac{2}{3} \) comes from the integration. Similarly defined \( \zeta(x) \) will allow us to perform a one-dimensional analysis in the hydrodynamic case. In figure 3·1d, we plot \( \zeta(x) \) for the same channel at several different hydrostatic pressures, 100 Pa \( \leq p \leq 50 \) kPa. These are the position-dependent (local) constitutive curves. Because of the clamping stresses, the deformable wall is initially in a buckled state. At low \( p \), the wall deformation remains in the negative \( y \) direction.

As \( p \) is increased, the wall response becomes elastic, and the wall stretches like a membrane. Also, a small asymmetry is noticeable in \( \zeta(x) \), caused by the deformation of the silicon chip during clamping. Figures 3·1e and 3·1f shows the peak deflection \( \zeta_p \), which typically occurs at \( (x, z) \approx (\frac{1}{2}, 0) \), as a function of \( p \) for the elastomer (PDMS) and SiN walls, respectively.
<table>
<thead>
<tr>
<th>Sample</th>
<th>Material</th>
<th>Wall thickness $t$ (μm)</th>
<th>Unperturbed height $2h_0$ (μm)</th>
<th>Re</th>
<th>Max. defl. $\zeta_{\text{max}}$ (μm)</th>
<th>Max. flow rate $Q_{\text{max}}$ (ml/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>SiN</td>
<td>0.2</td>
<td>175</td>
<td>70-1200</td>
<td>33</td>
<td>70</td>
</tr>
<tr>
<td>S2</td>
<td>SiN</td>
<td>1</td>
<td>180</td>
<td>100-1200</td>
<td>20</td>
<td>70</td>
</tr>
<tr>
<td>S3</td>
<td>SiN</td>
<td>1</td>
<td>97</td>
<td>250-1300</td>
<td>37</td>
<td>70</td>
</tr>
<tr>
<td>S4</td>
<td>PDMS</td>
<td>200</td>
<td>244</td>
<td>200-800</td>
<td>86</td>
<td>50</td>
</tr>
<tr>
<td>S5</td>
<td>PDMS</td>
<td>605</td>
<td>155</td>
<td>200-900</td>
<td>25</td>
<td>50</td>
</tr>
</tbody>
</table>

Table 3.1: Parameters of the channels (first four columns), the range of Reynolds numbers (Re), and the maximum channel deflection $\zeta_{\text{max}}$ attained under the maximum flow rate $Q_{\text{max}}$. The Re is found by averaging $Re_z = \frac{2Q}{\nu[w+2h(z)]]}$ over the channel.
Each deformable wall in figure 3·1e and 3·1f has a constitutive $\zeta_p$ vs. $p$ curve, determining the behavior of the entire wall. The thin nitride walls shown in figure 3·1f obey the well-known elastic shell model at high $p$, $\zeta_p \sim p^{1/3}$ (Small and Nix, 1992). The elastomer walls in figure 3·1e follow a different power law from the SiN ones, presumably because they are much thicker and bending dominates their deformation. There is no noticeable universality in the $\zeta_p$ vs. $p$ data — i.e., the nature of the wall response and thus the constitutive relations are material and geometry (thickness) dependent. Our flow results below, however, remain independent of the wall response.

3.4.2 Flow Measurements

Next, we perform flow measurements in each micro-channel. The results from all five channels are shown in figure 3·2a. In the experiments, we establish a constant volumetric flow rate $Q$ through each channel using a syringe pump and measure the pressure drop between the inlet and outlet using a macroscopic transducer. We prefer to plot $Q$ as the independent variable because the experiments are performed by varying $Q$ and measuring the pressure drop. In all measurements, a small pressure drop occurs in the rigid inlet and outlet regions of the channel. This is because of the finite size of the connections to the macroscopic pressure transducers. Knowing the geometry of the rigid regions, we determine the pressure drop in these regions from flow simulations. Subsequently, we subtract this “parasitic pressure drop” from the measured pressure drop. In summary, $\Delta p_t$ in the plots in figure 3·2a corresponds to the corrected pressure drop in the compliant section of the channel as measured by a macroscopic transducer (hence, the subscript “t”). Figure 3·2b shows the channel deflection $\zeta(x)$ at several different flow rates for S1 ($t = 200$ nm and $2h_0 = 175$ $\mu$m). Returning to table 3.1, we now clarify that $\zeta_{\text{max}}$ corresponds to the maximum deflection of the channel at the highest applied flow rate.
Figure 3.2: (a) The pressure drop $\Delta p_r$ in the compliant sections of the micro-channels as a function of flow rate $Q$. Error bars are smaller than the symbol sizes. The dotted lines show fits based on the hydraulic resistance of the micro-channel. The inset shows a double logarithmic plot of the same data. (b) The deflection profile $\zeta(x)$ of S1 ($t=200$ nm, $2h_0 = 175$ $\mu$m) at different $Q$. The profile is no longer uniform [cf. figure 3.1d] because of the position-dependent pressure $p(x)$ in the channel.
3.4.3 Simple Fits

Before we present our method for analyzing the flow, we attempt to fit the experimental $\Delta p_t$ vs. $Q$ data to the theory described above in equations (3.1-3.2). Because our channels have a finite width $w$, the result in (3.2) must be modified slightly. The simplest approach is to use a linear approximation for the local pressure drop based on the hydraulic resistance per unit length, $r(x)$, of the channel as outlined in section 3.2. In a long channel at low Reynolds number, $\partial_x p \approx Q r(x)$. The total pressure drop between the inlet and outlet can then be found as $\approx \int_0^l r(x)dx$ (Batchelor, 2000). In our analysis, we approximate our channel as a channel of rectangular cross-section of $w \times 2h(x)$, where $2h(x) = 2h_0 + \zeta(x)$. Then (Bruss, 2008),

$$r(x) \approx \left(\frac{1}{1 - 0.63 \left(\frac{2h(x)}{w}\right)}\right) \frac{12\eta}{w[2h(x)]^3}. \quad (3.11)$$

With the $2h(x)$ data available from optical measurements, we calculate $r(x)$ and integrate it along the length of the channel for all flow rates to find the pressure drop. The calculated $\Delta p_t$ are shown in figure 3-2a as dashed lines. It is difficult to determine the source of the disagreement between the data and the fits in some cases. The flow in the inlet and outlet regions may still be contributing to the error — even after subtraction. Another source of error may be the boundary between the compliant and rigid regions of the channel.

3.4.4 Measurement of the Pressure Distribution

We now turn to the main point of this chapter. Our method is illustrated in figure 3-3. The curves in the background in figure 3-3a are the now-familiar $\zeta(x)$ curves of S1 ($t = 200$ nm and $2h_0 = 175$ $\mu$m) under different hydrostatic pressures (figure 3-1d). These serve as the constitutive curves. On top of these hydrostatic profiles, we overlay two different hydrodynamic profiles (thicker lines) at flow rates of $Q = 5$ ml/min and
Figure 3.3: Determining the pressure gradient inside a microchannel from hydrostatic measurements (S1, $t=200$ nm, $2h_0 = 175$ μm). (a) The channel deflection profiles under two flow rates (upper thick line, $Q = 70$ ml/min; lower thick line, $Q = 5$ ml/min) are overlaid on top of the deflection profiles taken under different hydrostatic pressures [cf. figure 3.1d and figure 3.2b]. (b) The data points correspond to the pressure distribution $p(x)$ in the test section (shaded region) for a flow rate of $Q = 5$ ml/min. To obtain $p(x)$, the pressure values at the intersection points in (a) are plotted as a function of $x$. A nonlinearity is noticeable in $p(x)$ at $x \approx 7.5$ mm, where the slope of the linear fit changes. (c) Similarly determined $p(x)$ for $Q = 70$ ml/min. The dashed line is a simple linear fit. The solid (red) lines in (b) and (c) are the deflection profiles of the channel at the given flow rates. The error bars in the data are estimated to be smaller than the symbol size. Also plotted (+) are results from simple flow simulations.
$Q = 70$ ml/min. The assumption here is that, under equilibrium, $\zeta(x)$ only depends upon $p(x)$, providing us with the constitutive relation $\zeta(x) = \zeta(p(x))$. We determine the positions where the dynamic profiles intersect with the static profiles and read out the pressure values for each intersection position. In figure 3·3b and 3·3c, we plot these read-out pressure values using symbols as a function of position for the two different flow rates; solid (red) lines are the deflection profiles; also plotted (+) are results from simple flow simulations (see below for further discussion). In figure 3·3b, a noticeable deviation from a linear pressure distribution is present, as captured by the two dashed line segments with different slopes. The $p(x)$ in figure 3·3c can be approximated well by a linear fit (dashed line) to within our resolution. In the region near the boundaries ($x = 0$ mm and $x = 15.7$ mm), where significant pressure gradients must be present, it is not possible to obtain pressure readings. Thus, the test section is the shaded regions in figure 3·3b and 3·3c away from the boundaries. We confirm that similar behavior is observed in all measurements on different channels.

Finally, we show that what is found above is indeed the pressure distribution in the channel. First, we turn to the simple flow simulation results (shown by +) in figure 3·3b and 3·3c. Here, we take a two-dimensional channel with two rigid walls, with the top one having the experimentally-measured profile $\zeta(x)$ and the bottom one being flat. We prescribe the velocity $u$ at the inlet based on the experimental $Q$ value. We then calculate the pressure distribution in the channel with the outlet pressure set to zero. In figure 3·3b, a small nonlinearity qualitatively similar to that observed in the experiment is noticeable. Between the experiment and the simulation, there is a small but constant pressure difference ($\sim 300$ Pa), which probably mostly comes from the non-zero outlet pressure in the experiment. In figure 3·3c, we notice a constant pressure difference ($\sim 500$ Pa) between experiment and simulation as well; in addition, there is a larger pressure difference towards the inlet. The excess
pressure observed in the experiment is probably the pressure that is needed to keep the deformable wall stretched — as the deformability of the wall is completely ignored in the simulation. The wall is stretched more towards the inlet — hence the larger pressure difference. (We estimate that this tension is *not* present in the *buckled* wall of figure 3.3b.) Overall, the agreement is quite satisfactory.

We can further validate the extracted pressure drop $\Delta p_e$ across the (entire) deformable test section against the analytical approximation in (3.2). Our method provides $\Delta p_e$ directly for each flow rate. We illustrate this in figure 3.3b: we take the high and low pressure values at the beginning and end of the test section, and calculate the difference to find $\Delta p_e$, i.e., $\Delta p_e = p(x \approx 1.6 \text{ mm}) - p(x \approx 13 \text{ mm})$. Against this $\Delta p_e$ value, we plot $QR$, where $R$ is the hydraulic resistance of the channel for *only* the region where the pressure drop is determined, i.e., the hydraulic resistance of the test section. For the data in figure 3.3b, for instance, $R = \int_{x=1.6\text{mm}}^{x=13\text{mm}} r(x)dx$, where $r(x)$ is the resistance per unit length given in (3.11). $QR$ vs. $\Delta p_e$ data for each channel and flow rate are shown in figure 3.4. The error bars are due to the propagated uncertainties in the measurements of $2h_0 + \zeta(x)$.

### 3.5 Conclusions and Outlook

The agreement in figures 3.3 and 3.4 provides validation for our method and gives us confidence that we can measure $p(x)$ in deformable channels accurately. For the proof-of-principle demonstration, we have applied our method to a flow, which can be approximated by the Poiseuille equation, i.e., (3.2) and (3.11). However, the method should remain accurate independent of the nature of the flow (e.g., turbulent flows, flows with non-linear $p(x)$ or separated flows) because $p(x)$ simply comes from the wall response — as evidenced by the *non-linear* $p(x)$ resolvable in Fig 3.3b. It must also be re-emphasized that the nature of the wall response is not of consequence as...
Figure 3.4: Calculated pressure drop $QR$ in the test section as a function of the extracted pressure drop $\Delta p_e$. The symbols match with those used in figure 3.2a. Representative error bars are due to the uncertainties in $2h_0 + \zeta(x)$. The solid line is the $QR = \Delta p_e$ line.

long as the deflection is a continuous function — any function — of pressure, $\zeta = \zeta(p)$. All these suggest that the method can be applied universally as an accurate probe of flows with micron, and even possibly sub-micron, length scales.

Our results may be related to prior studies on collapsible tubes (Carpenter and Pedley, 2003; Heil and Jensen, 2003). For our system in two dimensions, in the case of small displacements, $\zeta/2h_0 \ll 1$, one can write a “tube law”

$$p = p(\zeta, \zeta'') \approx a\zeta + b\zeta'',$$  \hspace{1cm} (3.12)

which relates the local the gauge pressure $p$ (the so called transmural pressure) to the channel deflection $\zeta$ and its axial derivative $\zeta'' = d^2\zeta/dx^2$. In analogous expressions in the collapsible tube literature, the coefficient $a$ is typically found by considering the changes in the cross-sectional area; however, finding $b$, which determines the effect of the axial tension on $p$, is typically not simple and is possible only for certain tube geometries, e.g., for elliptic tubes (Whittaker et al., 2010). Several points are
noteworthy about our experiments. First, the axial tension term appears to be unim-
portant in test sections, i.e., $p = p(\zeta)$. Second, the method remains accurate even
when $\zeta \sim 2h_0$. Finally, a method similar to the one described here may be useful for
determining $b$ experimentally for different geometries and large deformations.

We mention in passing that the friction drag in a channel with rigid walls separated
by a gap of $2h_0$ is larger than that in a deformable channel with the same unperturbed
gap. To see this, consider a one-dimensional flow with a flux $q$ per unit width. The
stress at the rigid wall is $\tau = -\eta \partial_y u_{\text{wall}} = h_0 \partial_x p$. But $q = \frac{2}{3\eta} \partial_x p$. Therefore,
$\tau = \frac{3\eta^2 q}{2h_0}$. Given that $\frac{q}{h_0^2} \geq \frac{q}{(h_0 + \zeta/2)^2}$, drag is reduced. We also note that no evidence
of transition to turbulence has been observed in our experiments even at the largest
Re $\approx 1200$. 

Chapter 4

Measurement of Interstitial Fluid Pressure Field in Microscale Porous Hydrogels

4.1 Introduction

This study extends our non-invasive technique to the measurement of the interstitial fluid pressure field (IFP) within hydrogels in PDMS-based microfluidic devices. Currently, nearly all of the techniques that can measure IFP within hydrogels in a microfluidic device are invasive, in that they rely on interfacing the device with an external element (e.g., liquid column or air bubble for manometry) (Akbarian et al., 2006; Srivastava and Burns, 2007). While such techniques allow for the precise measurements of the local IFP, they suffer from the requirement that relatively large volumes of liquid be transferred through the device in order to provide observable changes (e.g., in the height of a water column). Since gels are hydraulically resistive, application of these invasive techniques to microscale gels may result in low temporal resolution and flow-induced alterations of IFP. Moreover, as the size of a probe becomes comparable to or even bigger than the flow scale itself, measurement of the distribution of flow fields becomes problematic: the pressure transducers may lack the spatial resolution to map the pressure field and/or may perturb the flow profile and alter the local pressure. A non-invasive technique to measure IFP would allow the device to function in its native state without potentially confounding effects due
to fluid flow to, from, or around a measurement port.

Our method employs interferometric microscopy in order to detect the extremely small distension (< 0.4 \( \mu \)m) of PDMS under typical device pressures. Our interferometric microscope can measure the distension field of PDMS devices with an in-plane resolution of \( \sim 9 \) \( \mu \)m and a height resolution of \( \sim 5 \) nm, corresponding to an error in IFP of \( \sim 10 \) Pa. In contrast to our earlier work (Ozsun et al., 2013), the time required to measure a pressure distribution in a typical sample is determined by the response time of the sample, because fluid movement through such a device is hindered by the resistance of the gel. By ensuring that the displacement of the PDMS remains small so that the accompanying interstitial flow does not limit the response time, we have attained a response time of \( \sim 5 \) min. We further show that this method is compatible with mammalian cell culture and that it can provide the necessary spatial and temporal resolution to generate IFP maps, which can be used in conjunction with computational modeling to estimate the hydraulic properties of microscale engineered tissues.

4.2 Theory

In one-dimensional flow through a thin distensible channel, the pressure \( P \) and distension \( \zeta \) vary along the flow axis \( x \). Their relationship is given by (McClurken et al., 1981)

\[
P(x) = P(\zeta(x)) = a\zeta(x) + b\frac{d^2\zeta(x)}{dx^2},
\]

where \( a \) and \( b \) are constants that characterize the elastic properties of the distensible wall.

We assume that this so-called "tube law" can be generalized to fluid systems, not necessarily thin-walled, in which the pressure and distension vary in two dimensions
Here, the linear term describes bending, and the terms with the curvature content describe stretching. Distension can be positive or negative; we do not limit the analysis to the case of thin membranes for which the resistance to collapse is small. For the devices considered in this study, the distension gradients are extremely small (< 0.4 μm displacement over a distance of 4 mm). This operating condition ensures that the curvature of the top wall due to pressurization is small; that is, the top wall remains nearly flat, and can be treated as an elastic plate.

From the elastic theory of plates, we obtain
\[ P(x) = a\zeta(x, y) + b_x \frac{\partial^2 \zeta}{\partial x^2} + b_y \frac{\partial^2 \zeta}{\partial y^2}. \] (4.2)

In our experiments, the thickness of the PDMS wall is \( t \approx 1 \) mm, and the maximum distention is \( \zeta \approx 500 \) nm, which means that the bending term is a factor of \( \sim 10^6 \) larger than the stretching term. Thus, bending is the dominant mode of deformation, and the relation between pressure and distension simplifies to \( P \approx a\zeta \).

In the current study, the pressure \( P(x, y) \) refers to that exerted by a porous medium that fills the channel, and in principle, includes contributions from interstitial fluid pressure as well as the stress exerted by the solid component (Wang, 2000). In the case of dilute extracellular matrix gels, the solid pressure is negligible; for a strain of < 1 μm/1 mm (as in the devices studied here) and a gel modulus on the order of 1 kPa, the solid stress is on the order of 1 Pa or less. Altogether, these considerations suggest that the local distension of PDMS is proportional to the local fluid pressure in the gel, i.e., IFP.
Knowing the stiffness $a$ allows one to calculate IFP from a measured distension map $\zeta(x,y)$. In a PDMS device, the local thickness of the microfluidic device may vary, so the bending “constant” may be a function of position, but is assumed to be independent of distension and pressure; that is, $a = a(x,y)$. Our proposed method to determine IFP is thus as follows: First, we measure the distension of the device under uniform pressure (or a series of uniform pressures) to obtain a map of the local stiffness $a(x,y)$. Second, we operate the device under conditions that subject the gel to pressure gradients, and use the local stiffness with measured distensions to obtain the pressure field:

$$P(x,y) = a(x,y)\zeta(x,y).$$

(4.3)

We validated the applicability of the proposed method with numerical simulations before applying it to experimental distension data.

4.3 Materials and Methods

4.3.1 Cell Culture

Mouse Lewis lung carcinoma cells (LLC; ATCC) and Madin-Darby canine kidney cells (MDCK; ATCC) were routinely cultured at 5% $CO_2$ in MEM (Invitrogen) that was supplemented with 10% calf serum (Invitrogen) and 1% glutamine-penicillin-streptomycin (Invitrogen). Bovine lung microvascular endothelial cells (BLMEC; VEC Technologies) were routinely cultured at 5% $CO_2$ on gelatin-coated dishes in MCDB131 (Caisson) that was supplemented with 10% fetal bovine serum (Atlanta Biologicals), 80 $\mu$M dibutyryl cyclic AMP (Sigma), 1 $\mu$g/mL hydrocortisone (Sigma), 25 $\mu$g/mL endothelial cell growth supplement (Biomedical Technologies), 2 U/mL heparin (Sigma), 0.2 mM ascorbic acid 2-phosphate (Sigma), and 1% glutamine-penicillin-streptomycin. All cells were used before passage ten.
4.3.2 Preparation of PDMS Devices and Collagen Gels with and without Cells

Figure 4.1a and 4.1b show the basic fabrication steps and configurations of the microfluidic devices. Each device consisted of a PDMS channel (width of 1 mm, height of 1 mm, length of 7 or 32 mm) that was adjacent to 6-mm-diameter wells and that lay on top of a glass slide. The thickness of the PDMS layer that formed the “ceiling” of the channel varied between devices, and was in the range of 1 ± 0.3 mm. We introduced a solution of type I collagen (from rat tail, 7-8 mg/mL; BD Biosciences) into the channels, and we allowed it to gel at ~ 23°C for thirty minutes. In some experiments, we molded the gel around one or two 120-μm-diameter needles (Seirin) that had been inserted into the channels; after gelation, we removed the needle(s) to yield a single open channel that spanned the gel, a single blind-ended channel (the tip of which lay at the middle of the gel; figure 4.1a, or a pair of opposing channels (the tips of which were at the middle of the gel and slightly offset; figure 4.1b), as described previously (Price and Tien, 2009; Wong et al., 2013; Tien et al., 2012). Gels were kept hydrated by adding phosphate-buffered saline (PBS) to each of the adjacent wells.

To form cell-containing samples, we used 7-mm-long gels that contained a single blind-ended channel (for LLC and MDCK cells) or a single open channel (for BLMECs). For LLC and MDCK samples, we first conditioned the gels for at least one hour by adding the appropriate media to the wells. We then added a dense suspension of cells to the well that was adjacent to the open end of the channel, and allowed interstitial flow of media to convect cells into the channel. We seeded LLC cells until they formed a packed bed within the channel, but limited the seeding time for MDCK cells so that only enough cells adhered to eventually organize into a confluent monolayer. We then washed the well with media to remove non-adherent cells,
and added fresh media to both wells to promote interstitial flow and thereby provide nutrients to the cells within the channel. For BLMEC samples, we first cross-linked gels with 20 mM genipin (Wako) for two hours, a step that was required to promote vascular adhesion and stability (Chan et al., ), before conditioning with media and seeding cells. After seeding, flow was established in the BLMEC samples by interfacing the wells with reservoirs of media that was supplemented with 3 % dextran (70 kDa; Sigma) (Chrobak et al., 2006; Leung et al., 2012); the reservoirs were held at pressures of ~800 Pa (8 cm H₂O) and 0 Pa, which yielded flow rates of 1.1-1.5 mL/hr.

4.3.3 Measurement and Analysis of Pressure-Distention Data

One day after forming gels (for cell-free samples), one day after seeding (for LLC samples), or three days after seeding (for MDCK and BLMEC samples), we imaged the mechanical response of the devices. At these time points, the cells in the LLC, MDCK, and BLMEC samples had organized into a packed bed, a blind-ended tubular monolayer, and an open tubular monolayer, respectively. SWLI (Deck and de Groot, 1994) was performed on a Zygo® NewView™ 6300 microscope with a 2.5× Nikon interferometric infinite conjugate objective (NA = 0.075, WD = 10.3 mm).

To image a sample, we first interfaced its two wells with polyethylene tubing (Braintree Scientific) to reservoirs of saline and media for cell-free and cell-containing samples, respectively (Price and Tien, 2009). For cell-free samples, we kept the reservoirs at the same height, and raised and lowered them in tandem to generate uniform hydrostatic pressures of 0-800 Pa within the gel, in steps of 100 Pa. At each pressure, we measured the resulting height profile at the outermost surface of the PDMS in a rectangular region-of-interest (4.24 mm × 5.65 mm area, 480 × 640 pixels CCD resolution) at the central section of the gel (red dotted rectangles in figure 4·1a and 4·1b). These measurements took place every minute; we typically recorded five
Figure 4.1: Experimental design. (a) Schematic of the formation of a microfluidic PDMS device that contains a collagen gel with a blind-ended channel. (b) Similar procedures generate devices that contain a solid gel, a gel with opposing blind-ended channels, or a gel with an open channel. Red dashed rectangles refer to the imaging window. $P_a$ and $P_b$ are the pressures applied to the ends of the gel. (c) Sequence of applied pressures. For cell-containing samples, we limited the pressure conditions to the three boxed ones. (d) Plot of PDMS distension versus time at a single point on a device that was filled with a solid collagen gel and subjected to a series of uniform hydrostatic pressures ($P_a = P_b = 0 - 800$ Pa, in steps of 100 Pa every six minutes). Inset, plot of distension versus pressure.
consecutive measurements at a given pressure before changing the pressure. Next, 
we introduced a pressure difference across the ends of the gel by holding one end at 
0 Pa and changing the pressure at the other end to 200-800 Pa in steps of 200 Pa. 
Again, we imaged the resulting deflection every minute for at least five minutes at 
each pressure condition. Each cell-free sample required up to 2 hr. to complete the 
entire set of pressure conditions. Figure 4-1c lists the complete sequence of pressure 
conditions.

For cell-containing samples, we minimized the imaging time, since the imaging 
was performed at room temperature. Thus, the pressure conditions consisted only of 
uniform 0 Pa, uniform 800 Pa, and a pressure difference of 800 Pa. Imaging these 
samples required only ~20 min. each.

Surface profile data were imported into and analyzed with MATLAB R2010b 
ver. 7.11.0 (Mathworks®). The profile of the PDMS without any pressurization was 
subtracted from all data to yield two-dimensional distension maps \( \zeta(x, y) \) for the 
various pressure conditions. The stiffness map of the PDMS device was calculated 
by a linear fit of pressure versus distension under uniform pressure conditions (for 
samples without cells) or from distension data under 800 Pa uniform pressure only 
(for samples with cells). To calculate the two-dimensional pressure profiles in the case 
of pressure-driven flow, we multiplied the stiffness map by the measured distension.

4.3.4 Numerical Modeling

We constructed finite-element models in COMSOL Multiphysics® ver. 3.5a to solve 
for the pressure distribution within the gel and the elastic deformation of the PDMS 
device. Governing equations consisted of Darcy’s Law for the gel and the constitutive 
equations of linear elastic solids for PDMS; only steady terms were retained. The two 
fields were coupled by assuming that the gel pressure acted as a load at the PDMS-gel 
interface. Since PDMS is nearly incompressible, we used models that treated pressure
as an additional independent parameter in the calculation of the stress field within the PDMS (Holzapfel, 2000). The Young's modulus and Poisson's ratio of PDMS were set to 2.5 MPa and 0.4999, respectively. For small models [< 10⁶ degrees-of-freedom (DOF)], we solved the governing equations simultaneously; for large models (up to ~ 4⁶ DOF), we solved Darcy's Law first and the elasticity equations second.

Adaptive meshing was used to resolve the stress concentration at the corners of the PDMS channel. Tests of mesh independence used two sequentially refined meshes that differed by at least two-fold in DOF; meshes were refined until these comparisons yielded a maximum pressure difference of < 4 Pa at the PDMS-gel interface and a maximum distension difference of < 5 nm at the top surface of the PDMS (i.e., the surface imaged in experiments).

For each gel configuration, we simulated an imaging experiment by cycling through the same series of pressure conditions as those used in experiments. For models that were designed to simulate LLC samples, we treated the blind-ended channel as a porous medium of unknown permeability $K_{LLC}$, and set the ratio of $K_{LLC}$ to the gel permeability $K_{gel}$ to be 0.01-100. For models that were designed to simulate MDCK samples, we assumed the surface of the blind-ended channel acted as a permeable membrane with hydraulic conductivity $L_{MDCK}$, and set $L_{MDCK}/K_{gel}$ to be 0.01-100 cm⁻¹. For models that were designed to simulate BLMEC samples, we assumed the surface of the open channel acted as a permeable membrane with hydraulic conductivity $L_{BLMEC}$, and set $L_{BLMEC}/K_{gel}$ to be 0.01-100 cm⁻¹; at the outlet end of the channel, we set the fluid pressure in the well and gel to be equal, as justified previously (Price et al., 2010). For the BLMEC models, the pressure within the channel in the gel was found by solving the Navier-Stokes equations before determining the pressure within the gel itself.
4.4 Results and Discussion

In a typical experiment, we subjected a gel-containing microfluidic device to a series of homogeneous pressures, and measured the resulting distension fields at the outermost PDMS surface. These measurements showed that gel pressure (i.e., IFP) and PDMS distension were proportional, and thus yielded a two-dimensional stiffness map of the device. We then subjected the device to a pressure difference, thereby inducing two-dimensional IFP variations, and measured the distension field. From the stiffness map, we calculated the non-homogeneous pressure field at the PDMS-gel interface as the product of local stiffness and distension.

4.4.1 Numerical Validation

Since our proposed approach relies on several assumptions (see Theory), we used detailed numerical models to validate the approach and to determine the accuracy of the IFP field that was calculated from PDMS distension (figure 4.2). These models reflected three gel configurations that we examined experimentally: a solid gel (figure 4.2a), a gel with a single blind-ended channel (figure 4.2b), and a gel with two opposing blind-ended channels (figure 4.2c). Solving these models showed that, regardless of the exact gel configuration, the distension of the PDMS was extremely modest; an 800 Pa uniform pressure within the gel led to less than ~ 400 nm distension of the outermost PDMS surface.

Moreover, the distension was linearly proportional to the level of uniform pressure. We then solved for the distension fields when the gels were subjected to a pressure difference (see figure 4.2 for the case of 800 Pa pressure difference). From these distension fields, we deduced the IFP in the gel [IFP (predicted)] in figure 4.2] by treating the computed distensions as if they were actual experimental data sets.
Figure 4.2: Numerical validation of the method for calculating IFP from distension maps. (a) Solid gel. (b) Gel with blind-ended channel. (c) Gel with opposing channels that were offset. In each panel, the upper and lower plots of distension refer to the cases of $P_a = P_b = 800$ Pa, and $P_a = 800$ Pa and $P_b = 0$ Pa, respectively. Arrows refer to the direction of the resulting flow for the latter case. The upper, middle, and lower maps of IFP refer to the pressures predicted from the distension maps, those computed directly from the models, and the difference between computed and predicted pressures, respectively.
These models also yielded the exact pressure fields within the gels ["IFP (computed)" in figure 4-2], which we could compare with the pressures predicted from the distension fields. The predicted and exact IFP were virtually identical for the case of a solid gel and a gel with one channel, with a maximum error of 0.3 and 4 Pa, respectively. For the case of a gel with opposing channels, the maximum error was $\sim 100$ Pa, a larger value that we attribute to the steep pressure gradients in the region between the tips of the channels (figure 4-2c, bottom). These results suggest that our generalized tube law for two-dimensional systems remains extremely accurate under the pressures examined.

### 4.4.2 Distention and Pressure Fields in Cell-Free PDMS Devices

Next, we applied the method to experimental distension data from PDMS devices that contained a gel (without cells). Since the gel is hydraulically resistive and may relax, it is possible that reaching a steady-state level of distension would require a measurable time lag after a sudden change in applied pressure. For this reason, we designed the dimensions of the PDMS device (in particular, the thickness of the wall above the gel) so that the distension would be small. Given a maximum distension of $\zeta \approx 400$ nm for an applied pressure of $P = 800$ Pa, a PDMS channel height and length of $H = 1$ mm and $L = 32$ mm, and a gel hydraulic permeability of $K_{gel} \approx 5 \times 10^{-8}$ cm$^4$/dyn·s (Chan et al., ), we estimated the interstitial flow speed to be on the order of $K_{gel}P/L \approx 1$ $\mu$m/s. Thus, the time required to transport the fluid needed to allow the PDMS to distend is on the order of $(\zeta L/H)/(1$ $\mu$m/s)$\approx 10$ s. Distension versus time data showed that the distension fields reached a steady-state in less than five minutes after changing the pressure condition (figure 4-1d), and the somewhat longer time-constant may result from viscoelastic relaxation of the gel. While using thinner PDMS would result in larger distensions at a given pressure (thus improving the sensitivity of the method), these distensions would also require longer times to
equilibrate. In our experience, an approximately 1-mm-thick PDMS wall led to an acceptable balance of sensitivity and responsiveness.

Under uniform pressures, the measured distensions were proportional to applied pressure (figure 4.1d, inset), which allowed us to generate a stiffness map characteristic of each PDMS device. Measurement of the distension fields under a pressure difference allowed us to calculate the IFP fields as a product of local stiffness and distension for three gel configurations (solid gel, gel with a single blind-ended channel, and gel with two opposing blind-ended channels) (figure 4.3).

Several features suggest that these calculated IFP fields accurately represent the actual pressure distributions at the PDMS-gel interface. Most importantly, the predicted sizes and directions of the IFP contours matched those expected from the gel configuration. For instance, a pressure difference across a solid gel yielded a nearly linear IFP drop along the channel, with a value at the gel midpoint close to 400 Pa (i.e., the average of the two applied pressures) (figure 4.3a). In a single blind-ended channel, the pressure in the gel nearly matched the pressure applied to the open end of the channel, as expected since the channel provides a much smaller hydraulic resistance than the bulk gel (figure 4.3b). In opposing channels, the pressure in the gel exhibited a much larger gradient, since the pressures applied to both open ends were easily transmitted to the central portion of the gel (figure 4.3c). Moreover, reversing the applied pressure difference led to a switch in the direction, but not the magnitude, of the predicted IFP gradients, as expected (figure 4.3b, c).

4.4.3 Pressure Fields in Cell-Containing Devices

To determine the suitability of this non-invasive technique to measure IFP in microscale tissues, we imaged the distensions of three distinct cell configurations: 1) a packed bed of LLC tumor cells in a blind-ended channel in the gel, 2) a monolayer of MDCK epithelial cells that lined a blind-ended channel, and 3) a monolayer of
Figure 4.3: Application of the method to obtain IFP maps in cell-free collagen gels. (a) Solid gel. (b) Gel with blind-ended channel. (c) Gel with opposing channels that were offset. In each panel, the upper plots of distension refer to the case of \( P_a = P_b = 800 \) Pa. The remaining plots of distension refer to \( P_a = 800 \) Pa and \( P_b = 0 \) Pa [(a)-(c), middle], and to \( P_b = 800 \) Pa and \( P_a = 0 \) Pa [(b) and (c), lower]. Arrows refer to the direction of the resulting flow for cases of non-uniform pressure. The IFP maps were calculated from the distension maps, and line plots of pressure versus axial distance along the PDMS centerline are shown. Contours of constant IFP are shown in (b).
BLMECs that lined an open, perfused channel (figure 4-4). Because the microscope stage that we used did not allow for sample heating, we limited the imaging to three pressure conditions (uniform 0 Pa, uniform 800 Pa, and a pressure difference of 800 Pa) to minimize cooling of the sample and exposure of cells to sub-optimal temperatures. From the distension field that was measured at 800 Pa uniform pressure, we calculated stiffness maps of the PDMS devices. As in the cell-free devices, each combination of stiffness map and distension field yielded a predicted IFP field when the tissue was placed under a pressure gradient.

We note that the predicted IFP fields qualitatively matched what would be expected from the organization and type of cells within each device. For the LLC sample (figure 4-4a), the IFP was intermediate between the two applied pressures, which suggested that the packed bed of cells had a hydraulic permeability comparable to or lower than that of the gel. For the MDCK sample (figure 4-4b), IFP was very close to the pressure applied at the end opposite to the opening of the channel; this result is expected since the epithelium should form an extremely tight hydraulic barrier, which would isolate the gel from the pressure applied to the channel. For the BLMEC sample under flow (figure 4-4c), IFP was closer to the pressure applied at the downstream end; this result is consistent with the endothelium being a resistive barrier that is not as tight as epithelium, and is predicted in previously (Wong et al., 2013; Price et al., 2010).

To quantify these comparisons, we built computational models of these three cell-containing configurations and varied the hydraulic properties of the LLC packed bed and MDCK and BLMEC monolayers. The plots in figure 4 show the root-mean-square error between the computationally obtained and experimentally predicted IFP axial gradients (i.e., $\partial P/\partial x$) as a function of cell hydraulic properties. With a gel hydraulic permeability of $\approx 5 \times 10^{-8}$ cm$^4$/dyn·s,
Figure 4.4: Application of the method to obtain IFP maps in gels that contained cells seeded within a channel. (a) LLC aggregate within a blind-ended channel. (b) MDCK epithelium that lined a blind-ended channel. (c) BLMEC endothelium that lined an open, perfused channel. In each panel, the IFP maps were calculated from distension maps. In (a) and (b), root-mean-square errors between the experimental IFP gradients and those obtained from computational models with a range of cell hydraulic properties are plotted. In (c), a line plot of pressure versus axial distance along the PDMS centerline is shown.
minimization of the errors between these models and the experimental data implied that the hydraulic permeability of the LLC packed bed was less than or equal to $\approx 2 \times 10^{-8}$ cm$^4$/dyn·s, ($K_{LLC}/K_{gel} = 0.4$; figure 4.4b), and that the hydraulic conductivity of the MDCK monolayer was less than or equal to $\approx 2 \times 10^{-9}$ cm$^3$/dyn·s ($L_{MDCK}/K_{gel} = 0.04$ cm$^{-1}$; figure 4.4c). These estimates for the hydraulic properties of tumor cell aggregates and MDCK epithelium agree well with values listed in the literature (Tien et al., 2012; Turner, 1992), which provide further evidence that our method is accurately measuring the pressure field within the gel.

Computational models did not predict pressure fields that agreed well with experiment for BLMEC-lined channels. This result suggests that the hydraulic properties of the endothelium were not uniform along the channel, in contrast to the assumptions of the model. Indeed, the experimental IFP field showed a smaller gradient in the downstream portion of the endothelium (figure 4.4c), which implies that the endothelium was leakier downstream. It has been noted in previous studies that the downstream end is where IFP can exceed the pressure within the channel, which can lead to endothelial instability (Price et al., 2010).

4.5 Conclusions

To our knowledge, this study is the first to describe a non-invasive technique to measure the interstitial fluid pressure field within microscale hydrogels. The non-invasive nature of the method provides advantages in spatial and temporal resolution over existing, invasive methods of measuring IFP. In the current work, the distension reached a steady-state distribution within $\approx 5$ min, which we could image instantaneously across an area of $> 20$ mm$^2$ with $\sim 9$ μm resolution using a 2.5× objective. The fine spatial resolution yielded a two-dimensional pressure map that could be compared with those predicted by computational modeling to elucidate the hydraulic proper-
ties of cells (or, in principle, any hydraulically resistive element) contained within the device. The method performed best when the IFP gradient was below 1 kPa/mm; using a thinner top PDMS wall would allow sharper gradients to be resolved, albeit at the cost of slower response. We emphasize that the method requires only that the microfluidic device deform slightly under pressure, as is true for any PDMS-based device; no specialized elements need to be coupled to the microfluidic channel, and the device can thus operate in its native state.

Strictly speaking, the measured IFP maps are the pressures at the PDMS-gel interface, which may differ from the pressures in the bulk of the gel or the pressures experienced by cells. If the pressure gradient is mainly along the channel axis, then this difference is small. To compute the complete, three-dimensional IFP field from knowledge of IFP at a two-dimensional boundary is possible, but these boundary-value problems require special care (e.g., regularization) to minimize noise. By matching the experimental IFP data to computed pressure fields from computational models, our method allows one to determine the hydraulic properties of the cultured cells. One can use these models to obtain an average hydraulic permeability (as for LLC aggregates and MDCK epithelium), or to locate spatial heterogeneities in cell permeability (as for BLMEC tubes). We note that the method only yielded upper bounds to the hydraulic permeabilities of the tumor cell aggregates and epithelium. To obtain more precise ranges of the cell hydraulic properties from IFP maps, it will be necessary to match the hydraulic resistance of the gel with that of the cells that it contains. For instance, application of this method to gels that are more resistive may allow one to distinguish between different types of epithelia, which have low permeabilities.

In the next chapter, we study fluid-structure interactions at the microscopic scale. We examine the shift in the boundary condition from no-slip to partial-slip in flows over porous superhydrophobic membranes. We begin the chapter with a background
on superhydrophobicity and the slip boundary condition.
Chapter 5

Drag Reduction in Laminar Flows over Superhydrophobic Surfaces

5.1 Background

5.1.1 Superhydrophobicity

When a small amount of water is deposited on a hydrophilic surface, it spreads over the surface making a large contact area and small contact angle. A hydrophobic surface, on the other hand, repels water making a smaller contact area and a larger contact angle. In both cases the system tends to be in a minimum energy state.

Young’s relation relates contact angle and the surface tensions of coexisting states at the contact line. In figure 5.1a drop of water is deposited on a plain flat solid surface. Here $2l$ is the diameter of the disc of water that’s in contact with the solid substrate. Liquid, solid and vapor phases coexist on the periphery of the disc. $\theta$ is the contact angle between the water and the solid substrate. The final shape of the drop depends on the energy balance between the surface tensions of the three interfaces (de Gennes et al., 2004). When the contact line moves by an amount $dx$, figure 5.1b, the change in the surface energy is

$$dE = (\gamma_{SL} - \gamma_{SV})dx + \gamma_{LV}dx \cos(\theta),$$

where $\gamma_{SL}$, $\gamma_{SV}$ and $\gamma_{LV}$ are the solid-liquid, solid-vapor and liquid-vapor surface tensions respectively. At equilibrium, the surface energy minimizes and the contact
Figure 5.1: a) A drop of water placed on a solid surface has a liquid-solid contact radius $l$ and contact angle $\theta$. b) Displacing the contact line by an infinitesimal amount $dx$ modifies the contact energies of all three interfaces.

The angle is given by the Young’s relation,

$$\cos(\theta) = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}}$$  \hspace{1cm} (5.2)

Depending on the solid and liquid surface tensions, it’s possible to switch from complete wetting ($\theta = 0^\circ$) to complete drying ($\theta = 180^\circ$).

The surface is called a superhydrophobic surface if the contact angle exceeds $150^\circ$ (Quéré, 2005). Many natural plants and animals exhibit superhydrophobicity such as the self-cleaning lotus leaf (Barthlott and Neinhuis, 1997), legs of water striders (Gao and Jiang, 2004) and butterfly wings (Zheng et al., 2007). All of these structures share in common a combination of water repelling coatings that make their surfaces hydrophobic and underlying micro or nano textures that alter the wetting properties, making the surfaces superhydrophobic.

There are two widely accepted models explaining the effect of roughness on superhydrophobicity. According to the Wenzel model (Wenzel, 1936), roughness can only enhance the natural tendency of a surface. Roughness increases the actual surface area. When compared to a smooth hydrophilic surface, on a rough one, water spreads more in order to minimize the surface energy. Hence, the solid-liquid contact area increases while the apparent contact angle decreases. Conversely, if a surface
is hydrophobic, an increase in the surface area induced by the roughness will be compensated by a decrease in the contact angle. Water has to make an unfavorable contact with the hydrophobic surface over a larger area if the contact angle stays the same. As the roughness increases or arrays of micro-nano structures are present on the surface, the water cannot penetrate through the gaps between the patterns and sits partially on air. This is known as the Cassie state (Cassie and Baxter, 1944). Flow over a textured surface in Cassie state will be superhydrophobic so long as air remains trapped in between the textures (see figure 5·3a).

5.1.2 Slip Boundary Condition in Flows over Superhydrophobic Surfaces

To completely describe the flow of a viscous fluid past a solid body, one must solve the Navier-Stokes equations inside the fluid subject to boundary conditions on the solid surface (Landau and Lifshitz, 1987). These boundary conditions cannot be obtained from hydrodynamics, but emerge from the microscopic interactions of fluid particles with the surface. Consequently, they are not universal.

One common microscopic boundary condition occurring at a fluid-solid interface is no slip. Contrary to most macroscopic flows where the fluid is expected to “stick” to solid boundaries, in microscopic flows the fluid is sometimes modeled as having some non-zero component of velocity tangent to a solid interface. A general boundary condition that captures the effect of slip is given by Navier (Maxwell, 1879). The velocity component of the fluid in the flow direction at the wall, is proportional to the shear rate at the wall. The slip velocity is

\[ u_s = \frac{\nu}{\kappa} \dot{\gamma} = b \partial_z u_z, \] (5.3)

where \( \dot{\gamma} \) is the shear rate, \( \nu \) is the fluid kinematic viscosity, \( \kappa \) is the interfacial friction and \( b \) is the so called the slip length (Bocquet and Barrat, 2007). The slip length
Figure 5.2: Standard No-slip BC with slip length $b = 0$ and Navier BC with partial slip, $b > 0$. Arrows indicate the fluid velocity in the flow direction.

is not a pure property of the interface, but the ratio of the bulk viscosity $\nu$ to the interfacial friction $\kappa$. It has the units of length and refers to the imaginary distance below the solid surface where the no slip boundary condition applies (figure 5.2).

5.2 Stokes’ 2\textsuperscript{nd} Flow over Porous Superhydrophobic Membranes in the High Frequency Limit

Flow of a viscous liquid along a solid surface experiences drag. Drag friction is directly proportional to the wetted surface area of the solid body in contact with the fluid. Superhydrophobic surfaces can reduce viscous dissipation in the solid-liquid boundary layer by altering the boundary condition at the solid-liquid interface (Rothstein, 2010). On a conventional superhydrophobic surface (de Gennes et al., 2004), hydrophobicity combined with microscopic roughness causes the water surface to remain suspended above the solid tips, with mostly trapped air underneath (Miwa et al., 2000; Bico et al., 2001)(see figure 5.3a). Since the flow is on a composite surface made up of solid and air, one solves the Navier-Stokes equations subject to no-slip on
the solid elements and to slip at the water-air interface (Lauga et al., 2007). Thus, viscous friction force on a superhydrophobic surface is found to be proportional to the wet solid area, $\phi_s$ (Ybert et al., 2007).

Here we study flows over porous superhydrophobic membranes which have intimate contact with both the water reservoir atop and the gas reservoir below. We study oscillatory laminar flows over superhydrophobic porous membranes. We show that flow on these porous structures deviates from the above picture. Oscillatory hydrodynamic response (Ekinci et al., 2008) of the membrane suggests that a stable Knudsen layer of gas percolates on the membrane, changing the boundary condition (see figure 5-3b). This is because the porous superhydrophobic membrane structure enables the surrounding air to move ballistically to the interface with little resistance — in contrast to a conventional superhydrophobic surface, where trapped gas pockets are diffusively connected to a gas reservoir through macroscopic distances.

5.2.1 Experimental System

We have fabricated tension-dominated porous silicon nitride membranes. We made them superhydrophobic by silanization. Figure 5-4 shows a typical system under study. The square membrane has macroscopic dimensions $a = 600\ \mu m$ and thickness $t = 200\ nm$ with square pores of dimension $l_a = 10\ \mu m$. The pitch is given by $l_a + l_s$.
Figure 5-4: (a) Top view of a porous membrane chip \((a \times a = 600 \times 600 \mu m^2)\). (b), (c) Optical micrographs of \(\phi_s = 0.78\) and 0.34 membranes, respectively. (d) A drop of water placed on a larger membrane \((a \times a = 2 \times 2 \text{ mm}^2\) and \(\phi_s = 0.48\)\) showing the superhydrophobicity of the surface. (e) Vacuum mechanical resonances of a \(600 \times 600 \mu m^2\) porous membrane with \(\phi_s = 0.34\). Nearly-degenerate modes \((m, n)\) and \((n, m)\) are observed when \(m \neq n\). Single standard deviations in the data are smaller than the symbols. (f) The first three normal modes of the membrane

where \(l_s\) is the linear dimension of the solid patch between two adjacent pores. This results in a solid fraction \(\phi_s = 1 - \frac{l_s^2}{(l_s + l_a)^2}\). When a drop of water is placed on the porous membrane it is supported by the composite surface and wetting is not favored as shown in figure 5-4d.

We first characterize the intrinsic mechanical properties of the porous membrane. We perform these measurements in vacuum to eliminate any fluidic effect. The normal modes of a tension-dominated membrane are described by the number of nodes and antinodes in that mode. Figure 5-4f shows the first three normal modes of a porous membrane with \(\phi_s = 34\%\). The frequency of a normal mode \((m,n)\) is given by

\[
\frac{\Omega_{mn}}{2\pi} = \sqrt{\frac{\sigma_s}{\phi_s \rho_s}} \left( \frac{m^2 + n^2}{l_s^2 + l_a^2} \right)^{1/2}
\]

(Timoshenko et al., 1974). Here, \(\sigma_s\) is the tension, \(\rho_s\) is the density, and \(m\) and \(n\) are two integers. The in vacuo mode frequencies \(\frac{\Omega_{mn}}{2\pi}\) of a \(\phi_s = 0.34\) membrane are shown in figure 5-4e. The good agreement between the data and theory confirms that the tension dominated membrane approximation holds well and the system can be modeled as a damped harmonic oscillator with effective mass
$M_s = \frac{\sigma_s \phi_s a^2}{4}$ and stiffness $K_s = \frac{\sigma_s \pi^2}{4}(m^2 + n^2)$. Relevant mechanical parameters for the fundamental modes of all our membranes are displayed in Table 5.1.

After the intrinsic mechanical characterization, we turn to under water measurements. The measurements are performed using a fluid cell atop the membrane as shown in figure 5.5a. The membrane does not leak but supports intimate contact with both the water reservoir atop and the gas reservoir below. We have measured the thermal-noise spectra of all the membranes in their fundamental modes. Figure 5.5b shows the noise spectra measured at the center of three membranes with different $\phi_s$. We work with the fundamental mode $[(m, n) = (1, 1)]$ as shown in the inset of figure 5.5b. The top data trace in figure 5.5c shows all the fundamental resonance frequencies in vacuum, $\frac{\Omega_s}{2\pi}$. Thermal spectra with water atop the membranes have provided the resonance frequencies $\frac{\Omega_s}{2\pi}$ and line-widths $\frac{2\pi}{\Omega_s}$ (bottom trace).

### 5.2.2 Theory

We make a one-dimensional damped harmonic oscillator approximation for the fundamental mode of the membrane oscillations under water. In this approximation, the membrane position is $z_s$, velocity $u_s = \dot{z}_s \hat{z}$, mass $M_s$ and stiffness $K_s$. We assume that $u_s$ is nearly sinusoidal because all membrane resonances in water have quality factors $Q_w \geq 20$ and the thermal drive has a white spectrum: $u_s \approx \Re \{U_s e^{i\Omega_w t}\}$.

<table>
<thead>
<tr>
<th>$\phi_s$</th>
<th>$\Omega_s^{11}/2\pi$ (kHz)</th>
<th>$K_s$ (N/m)</th>
<th>$M_s$ (10$^{-12}$ kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>235</td>
<td>134</td>
<td>61.2</td>
</tr>
<tr>
<td>0.96</td>
<td>233</td>
<td>126</td>
<td>58.7</td>
</tr>
<tr>
<td>0.88</td>
<td>223</td>
<td>104.7</td>
<td>53.8</td>
</tr>
<tr>
<td>0.82</td>
<td>208</td>
<td>67.6</td>
<td>50.2</td>
</tr>
<tr>
<td>0.78</td>
<td>142</td>
<td>38.1</td>
<td>47.7</td>
</tr>
<tr>
<td>0.65</td>
<td>159</td>
<td>39.2</td>
<td>39.8</td>
</tr>
<tr>
<td>0.48</td>
<td>106</td>
<td>12.9</td>
<td>29.4</td>
</tr>
<tr>
<td>0.34</td>
<td>134</td>
<td>14.2</td>
<td>21.1</td>
</tr>
</tbody>
</table>

Table 5.1: Mechanical properties for the fundamental mode of the porous membranes.
Figure 5.5: (a) Schematic of the measurement cell in cross-sectional and isometric views. The cell is filled with water and is placed on top of the membrane chip. A heterodyne Michelson interferometer probes the motion from below. (b) Thermal noise spectra of the fundamental mode of three different membranes with water atop. From top to bottom, $\phi_s = 0.48$, 0.6 and 1. The frequency axis is normalized with the respective resonance frequencies in water. The inset shows the shape of the fundamental mode for the $\phi_s = 0.6$ membrane. (c) Fundamental-mode resonance frequencies in vacuum and with water atop, and line-widths $\gamma/2\pi$ with water atop. Error bars represent the associated single standard deviations and are only shown when larger than the symbols.
Given that the dissipation from water dominates the overall dissipation, we write

\[ M_s \dot{u}_s \approx F_e + F_f, \]  

(5.4)

where \( F_e = -K_s z_s \) is the elastic spring force and \( F_f \) is the magnitude of the fluidic force on the membrane in the high frequency limit (Zhang and Stone, 1998):

\[ F_f \approx \Re \left\{ A \frac{\eta a^2}{\delta} u_d e^{i\phi} + M_f \frac{du_s}{dt} \right\}, \]  

(5.5)

with \( \phi \approx \frac{\pi}{4} \) and \( A \sim 20 \). The viscous boundary layer thickness, \( \delta = \sqrt{\frac{2\eta}{\rho U}} \), depends on the dynamic viscosity \( \eta \) and density \( \rho \) of the fluid. \( M_f \) is the so-called added or hydrodynamic mass, well-known from the potential flow theory around an accelerating solid body. In equation (5.5) the first term denotes the viscous energy dissipation due to tangential flow over the membrane and the second term emphasizes the effect of added mass as will be shown below. We plug \( F_e \) and \( F_f \) into equation (5.4) and solve for the complex response function (Paul et al., 2006)

\[ G(\omega) \approx [K_s - (M_s + M_w)\omega^2 + i\alpha \omega]^{-1}. \]  

(5.6)

### 5.2.3 Results and Discussion

The effect of the fluid is embedded into two measurable parameters in equation (5.6). The added mass goes into inertial term and the friction coefficient \( \alpha \) contains all the viscous dissipation terms. The stiffness of the membrane does not change appreciably from vacuum to water, such that, \( M_s \Omega_v^2 \approx M_w \Omega_w^2 \) where \( M_s \ll M_w \) (Maali et al., 2005). Hence, added water mass \( M_w \) can be determined from the frequency shift of the membrane mode when it is loaded with water. Figure 5-6a shows \( M_w \) as a function of \( \phi_s \), calculated using \( M_s \) values in Table 5.1 and frequency values in figure 5-5c. The added mass shows a fast decrease with decreasing \( \phi_s \), eventually by a factor...
The upper slowly increasing purple trace in figure 5.6b is the cycle averaged viscous friction force, \( F_f \), on the membrane predicted by using the plate model of equation (5.5) and accounting for the membrane mode shape

\[
F_f = \frac{A\eta_w a^2 \phi_s U_s}{2\pi^2 \delta}.
\]

Here, \( \eta_w \) is the dynamic viscosity of water; \( \frac{A}{2\pi^2} \sim 1 \), and \( U_s^2 \approx 2\pi^2 \Omega_w^2 \langle z^2 \rangle \), where \( \langle z^2 \rangle^{1/2} \) being the thermal amplitude of the membrane found from the integral of the measured displacement noise spectral density. The lower black trace is the experimental cycle averaged friction force, \( U_s \), extracted from the one dimensional damped harmonic oscillator model. Here, it is important to note that, as \( \phi_s \) gets smaller, \( U_s \) increases, since \( K_s \) decreases. Therefore, in the calculated plate model, the decrease in the wetted area, \( a^2 \phi_s \) appears to be offset by this increase. To better assess the drag reduction on porous membranes, we eliminate the effect of velocity and and plot the friction coefficient \( \alpha \) in figure 5.6c. The plate model predicts that the friction will reduce linearly with \( \phi_s \) while experimentally an anomalous friction reduction is observed for \( \phi_s \leq 0.9 \).

### 5.2.4 Conclusion

We conjecture the observed deviation from the plate model as follows. On a conventional superhydrophobic surface, the flow is over a composite surface with the no-slip BC on solid elements and the slip BC at the air-water interface. Thus, in a first pass analysis, the viscous friction force is proportional to the wetted solid area \( a^2 \phi_s \). In our structures, a stable Knudsen gas layer percolates to the interface changing the BC completely. The porous membrane allows the surrounding air to move ballistically to the interface. This is in contrast to conventional superhydrophobic surfaces where trapped gas pockets are connected diffusively to a gas reservoir through macroscopic
Figure 5.6: (a) Measured $M_w$ as a function of $\phi_s$. The line segment is the hydrodynamic mass of the entire water layer. (b) Average friction force and (c) the normalized friction coefficient. The plate prediction is calculated from equation (5.5) using experimental velocities and frequencies where needed. The normalized friction coefficient for the plate model is $\approx \phi_s$. Error bars represent the associated single standard deviations and are only shown when larger than the symbols.
distances. Assuming a complete Knudsen layer at the interface, we can assess the friction reduction on a porous membrane with small $\phi_s$. The shear stress is a continuous function of the coordinate at the interface ($z \approx 0$), $n_\parallel U_w \sim \rho_g U_s \phi_s u_{th}$. Here, $U_s \phi_s$ and $u_{th}$ are respectively the average hydrodynamic velocity and the thermal velocity of air molecules; $\rho_g$ is the density of air. The $1/6$ factor accounts for the fraction of molecules traveling in the $+z$ direction. Using the parameters available, we derive $\frac{U_s}{U_w} \sim \frac{3}{\phi_s}$. The slip length (Lauga et al., 2007), $b \sim \frac{n_\parallel}{\rho_g u_{th} \phi_s}$, emerges as 6 $\mu$m at $\phi_s = 0.34$.

Our results might be relevant to applications. Unlike air bubbles on a hydrophobic surface (Brenner and Lohse, 2008), the air layer in our system is stable against diffusion into the water because of the resistance-free influx from the air reservoir. Assuming that porous pipes of macroscopic dimensions can be manufactured, significant drag reduction could be achieved. Several puzzling phenomena in bio-fluid-dynamics, including transport through and over bio-membranes, and propulsion over the water surface, may be related to the physics observed here (Bush et al., 2007; Gao and Jiang, 2004).

In next section, we will test the efficacy of our superhydrophobic porous walls in drag reduction in pressure driven channel flows. We present our experimental system, analysis and initial results on drag measurements in channel flows over porous superhydrophobic membranes. We list our suggestions for future directions in the outlook.

5.3 Drag Measurements in Laminar Channel Flows over Porous Superhydrophobic Membranes

Slip has been observed in channel flows over smooth hydrophobic surfaces (Tretheway and Meinhart, 2002). The most accepted interpretation of the slip on hydrophobic
surfaces is due to surface nanobubbles (Tyrrell and Attard, 2001). The fluid flows over these shear free regions and exhibits less contact with the solid wall (Ruckenstein and Rajora, 1983). For flows over superhydrophobic surfaces in the Cassie state, slip lengths on the order of microns have been numerically predicted (Cottin-Bizonne et al., 2003) and experimentally demonstrated (Joseph et al., 2006; Tsai et al., 2009; Choi et al., 2006; Ou et al., 2004). Scaling laws for underlying surface texture length scales are proposed (Ybert et al., 2007). Similar to these studies, we perform drag measurements in laminar channel flows over porous membrane walls. We use both superhydrophobic porous membranes and hydrophilic membranes without holes to test the effects of solid fraction $\phi_s$ on the measured drag.

### 5.3.1 Experimental System

We have fabricated 15.5 mm $\times$ 1.7 mm $\times$ 1 $\mu$m rectangular SiN membranes (figure 5.7a). We etch holes on these membranes to make them porous (figure 5.7b). We study samples with both different hole sizes and hole densities. We follow standard photolithographic techniques for sample preparation. We spin-coat the back side of 1 $\mu$m thick Si$_3$N$_4$ wafers with HMDS primer first, and then, S1818 positive tone photoresist at 2500 rpm for 40 seconds. The resulting resist thickness is measured to be around 2 $\mu$m. After lithographically defining etch windows on the back side of the wafer, we etch the 1 $\mu$m thick nitride layer with RIE to reach the 500 $\mu$m thick Si layer. After dry etching, the resist is stripped and the wafers are dipped into KOH solution (1:2 KOH:H$_2$O by mass) at 80°C for 4-5 hours. KOH etches Si anisotropically until the nitride layer on the other side of the wafer is reached. Rectangular chips with free standing rectangular Si$_3$N$_4$ membranes attached to Si frames are obtained with this process as shown in figure 5.7a. After KOH etching, we go through another lithography and dry etching step to introduce porosity on the membranes individually figure (5.7b). After the fabrication, each membrane is cleaned in piranha solution and
placed in oxygen plasma for half an hour before silanization. In the last stage, the samples are placed in a vacuum desiccator where they are silanized with vapor phase trichloro(1H, 1H, 2H, 2H - perfluoroocetyl) silane.

Figure 5.7 illustrates the custom flow cell with inlet and outlet ports. The membrane chips are clamped in between two plastic plates with thumb screws. During clamping, due to compressive stresses membranes buckle slightly without wrinkling (see section 2.4.1). We connect the inlet port to the Syringe pump (Harvard Apparatus Pump Elite 11 series) with luer type connectors, 16 ga luer stubs and Pt cured silicone tubes (Inner Diameter: 1.58 mm, Wall thickness: 0.81 mm). First, we measure the surface profiles of the membranes with SWLI under hydrostatic loading. Then, we proceed to flow experiments and measure the pressure drop across the inlet and outlet ports with Omega differential pressure transducers (PX409-005DWUV and PX409-001DWUV) while simultaneously collecting the surface profiles of the deformed membrane. The maximum attainable hydrostatic pressure and flow rate before leak depends on the pore size of the membrane. Membranes with bigger holes such as $S_1$ leak at lower pressures and flow rate lower than samples with smaller holes.
Table 5.2: Initial solid fraction, $l_a$ and $l_s$ of the membranes used in this study. All the membranes are rectangular with $l \times w = 15.5 \text{ mm} \times 1.7 \text{ mm}$ with thickness $t = 1 \mu \text{m}$.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\phi_s$</th>
<th>$l_a$ (µm)</th>
<th>$l_s$ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$S_1$</td>
<td>28.6</td>
<td>34</td>
<td>6.2</td>
</tr>
<tr>
<td>$S_2$</td>
<td>56.8</td>
<td>10.3</td>
<td>5.4</td>
</tr>
<tr>
<td>$S_3$</td>
<td>73.4</td>
<td>20.7</td>
<td>19.4</td>
</tr>
<tr>
<td>$S_4$</td>
<td>88</td>
<td>11.2</td>
<td>21.1</td>
</tr>
<tr>
<td>$S_5$</td>
<td>100</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

such as $S_4$. Examples of surface profiles collected around 4 kPa hydrostatic pressure and $Q = 10 \text{ ml/min}$ for sample $S_4$ are shown in figures 5.7d and 5.7e respectively. Table 5.3.1 lists the samples with their relevant parameters used in this study.

5.3.2 Theory

The flow is modeled as a pressure driven incompressible viscous steady flow in a rectangular channel of height $2h_0$ and width $w$. The relation between the total volume flow rate, $Q$, and the pressure drop, $\Delta p$, between two points separated by a distance $l$ along the flow direction can be written as

$$ Q = \frac{\Delta p}{R_{hyd}} + Q_a, $$

(5.8)

where $R_{hyd}$ and $Q_a$ are the total hydraulic resistance in the channel and the added volume flow rate due to slip at the boundary, respectively. $R_{hyd}$ and $Q_a$ can be found by solving the governing equation (3.4) with Navier boundary condition at the slip boundary.

A solution for the local velocity satisfying the boundary conditions $u(y = -b = 2h_0) = 0$ is

$$ u(y) = \frac{1}{2\eta} \frac{dp}{dx} (y + b)(2h_0 - y). $$

(5.9)

Then, the volume flux across a cross section normal to the flow direction, $Q$, can be
written as

\[ Q = \frac{w \ dp}{2\eta \ dx} \int_{0}^{2h_0} (y + b)(2h_0 - y)dy \]

\[ = \frac{w \ dp}{2\eta \ dx} \left( \frac{4h_0^3}{3} + 2bh_0^2 \right). \]  \hspace{1cm} (5.10)

Here, \( b \) can be found from the slip velocity at the boundary, \( u_{\text{slip}} = u(y = 0) = \frac{1}{2\eta \ dx} \Delta p \). Then, plugging \( 2bh_0 = \eta u_{\text{slip}} \frac{dx}{dp} \) in equation (5.10), and integrating over the distance \( l \) between two points, the volumetric flow rate is given by

\[ Q = \frac{2wh_0^3}{3\eta l} \Delta p + 2wh_0u_{\text{slip}}. \]  \hspace{1cm} (5.11)

Here, the first term on the right hand side is the volume flow rate for standard Poiseuille flow with no-slip boundary conditions. The second term is the additional volume flow rate due to slip boundary condition, \( Q_a \). Comparing equations (5.11) and (5.8), we find \( R_{\text{hyd}} = \frac{3\eta l}{2wh_0^3} \), and the additional volume flow rate \( Q_a = 2wh_0u_{\text{slip}} \).

Equation (5.8) can be written in a normalized form:

\[ \frac{QR_{\text{hyd}}}{\Delta p} = 1 + \frac{QaR_{\text{hyd}}}{\Delta p}. \]  \hspace{1cm} (5.12)

Here, \( \frac{QR_{\text{hyd}}}{\Delta p} = 1 \) for no drag reduction. The second term is the normalized pressure drop reduction, \( \Delta p_a \), due to added volume flow rate,

\[ \Delta p_a = \frac{QaR_{\text{hyd}}}{\Delta p} = \frac{QR_{\text{hyd}}}{\Delta p} - 1. \]  \hspace{1cm} (5.13)

A higher value of \( \Delta p_a \) indicates a higher drag reduction.

5.3.3 Results and Discussion

In equation (5.13), \( R_{\text{hyd}} \) is the total hydraulic resistance of the channel. It’s found by integrating local hydraulic resistances, \( r(x) \) in equation (3.11), along the entire
Figure 5.8: (a) \( QR_{\text{hyd}} \) vs. \( \Delta p_e \) plot in test sections for samples \( S_1 - S_5 \). (b) Pressure drop reduction as a function of flow rate for samples \( S_2 - S_5 \) calculated from figure (a). Pressure drop reduction falls sharply for the full membrane as the flow rate increases while for porous membranes, pressure drop reduction increases with flow rate in general.

We plot \( QR_{\text{hyd}} \) vs. \( \Delta p_e \) curves for all samples in figure 5.8a. The solid line is \( \frac{QR}{\Delta p_e} = 1 \) line. The dashed line is for the membrane with no holes (sample \( S_5 \)). Note that a smaller \( QR_{\text{hyd}} \) indicates a lower flow rate. Our first observation is that for a membrane without holes, the data deviates from the solid line indicating a drag reduction. However, drag reduction due to slip is not possible on a membrane without holes. We will discuss later. Next we see that at low flow rates, the data converge to \( \frac{QR}{\Delta p_e} = 1 \) line for all samples. As the flow rate increases, porous membrane curves start to deviate from the solid line around \( \Delta p_e > 2 \text{ kPa} \).

Assuming the reason of this deviation to be drag reduction, we plot normalized
pressure drop reduction, $\Delta p_a$, as a function of flow rate, $Q$, for all samples in figure 5.8b. The solid line is $\Delta p_a$ for a membrane without holes. We see that for the full membrane, $\Delta p_a \to 0$ as the flow rate increases, while for porous membranes, $\Delta p_a$ increases in general. $\Delta p_a = 0$ Pa is expected for the full membrane due to no slip boundary condition. However, we see this behavior only as the flow rate increases. The reason for high $\Delta p_a$ for full membrane at low flow rates can be due to some other mechanism that reduces drag or elastic behavior of the buckled membrane at low flow rates. Large pressure gradients and high curvature of the membrane at the inlet of the test section might have an effect on the flow as well.

We need to refer to the corrected transducer pressures, $\Delta p = \Delta p_t$, to measure the pressure drop across the entire membrane. $\Delta p_t$ is the pressure drop over the entire membrane including the regions of high curvature. $\Delta p_t$ is found by subtracting the calculated pressure drop at the rigid inlet and outlet regions from transducer pressure read out (see section 3.4.2 for details). However, in this flow cell configuration, we can’t use our transducer pressure data. As seen in figure 5.7c, water passes through curved rigid regions before arriving to the membrane. The complex geometry of the inlet and outlet ports makes the calculation of pressure drop impossible analytically and requires numerical approaches.

Instead of using numerical methods, we come up with a simpler channel geometry which allows direct analytical calculation of the pressure drop outside the membrane region and avoid buckling of the membranes. We took out the bottom plate and screws from the flow cell design. We cast a few millimeter thick PDMS slabs with same lateral dimensions as the membrane chip. On these PDMS slabs, we define rectangular grooves of the same lateral dimensions as the membranes and a height $2h_0 \approx 170 \mu$m. First, we punch circular holes at both ends of the rectangular groove. Then, we place the PDMS slab on the membrane chip where the groove on the PDMS
Figure 5-9: $QR_{hyd}$ vs. $\Delta p$ plot for samples $S_2 - S_5$ (a) in test sections and (b) over the entire membrane. Note that full membrane data falls on the $QR_{hyd}/\Delta p = 1$ line after including the effects of highly curved membrane regions.

We observe similar behavior to that observed in figure 5-8a. At low flow rates, all the data converges to solid line. Full membrane data deviates from the solid line and falls on the porous membrane data.

We can extract the corrected transducer pressure due to simple geometry of the...
Figure 5.10: (a) pressure drop reduction as a function of flow rate for samples $S_2 - S_5$ calculated from figure 5.9b. The full membrane shows almost no pressure drop reduction, while porous membranes do, although the data is quite scattered. (b) Average pressure drop reduction as a function of solid fraction for samples $S_2 - S_5$. The results do not show a linear relation between pressure drop reduction and $1 - \phi_s$.

Inlet and outlet regions. In figure 5.9b, we plot $QR_{hyd}$ vs. $\Delta p_t$ curves over the entire membrane for samples $S_2 - S_5$. This time, $R_{hyd}$ is found by integrating local hydraulic resistances, $r(x)$, along the entire membrane including the highly curved regions. Similarly, $\Delta p_t$ is the pressure drop between two ends of entire membrane. The solid line is $\frac{QR}{\Delta p_t} = 1$ line. The dashed line is for the membrane with no holes (sample $S_5$). When the pressure drop across the inlet and outlet regions are accounted, full membrane data falls on the solid line agreeing with the no-slip boundary condition. Porous membrane data deviates from the solid line as before indicating a higher pressure drop reduction than the full membrane.

Similar to figure 5.8b, we plot normalized pressure drop reduction, $\Delta p_a = \frac{Q_a R_{hyd}}{\Delta p_t}$, as a function of flow rate, $Q$, for all samples from our new setup in figure 5.10a. The solid line is the normalized pressure drop reduction for a membrane without holes. Our first observation is that $\Delta p_a \approx 0$ Pa for the full membrane as the solid line fluctuates around 0 Pa. This agrees with our expectations such that as $Q_a \to 0$ ml/min as $\phi_s \to 0$ because $u_s \to 0$ m/s at the boundary. We see that all porous
membranes have a higher $\Delta p_a$ value than the full membrane although the data is quite scattered. We average the normalized pressure drop reduction for each sample and plot it as a function of solid fraction, $\phi_s$, in figure 5.10b. We do not see a linear relationship between the pressure drop reduction and the solid fraction. Instead, pressure drop reduction peaks around 70% solid fraction (sample $S_3$). Sample $S_2$, which has the lowest solid fraction, has the lowest pressure drop reduction among porous membranes. This in contrast to our intuition that pressure drop reduction scales linearly with $1 - \phi_s$.

### 5.3.4 Conclusion and Outlook

We perform drag measurements in channel flows with superhydrophobic porous membrane walls on one side. We analyze one dimensional deformation data over the entire membrane. Higher pressure drop reduction is observed for porous membranes compared to membranes without holes, albeit data is scattered across a large range. Assuming pressure drop reduction to be due to fluid slip at the boundary, we calculate the average pressure drop reduction for each membrane. The results show that pressure drop reduction for different solid fractions does not scale linearly with $1-\phi_s$.

We suspect that one dimensional model employed in data analysis is not sufficient to capture the complete pressure distribution over the entire membrane.

The one dimensional model works well in measuring pressure distributions in slightly varying sections of rectangular channels in Chapter 3. However, at the entrance and end of the channel, one dimensional model may not work. In these highly curved regions, there must be large pressure gradients present. A comparison between membranes with different solid fractions is not possible unless the complete pressure distribution is known.

A complete picture of the pressure distribution is possible by collecting high quality optical data with minimal missing pixels and analyzing them with improved ver-
sions of methods described in section 4.3.3. With high resolution two dimensional data, we can analyze the effects of decreasing contact area on drag reduction. We can look for effects of entrance curvature on streamlining; and see, if there's any, how this streamlining affects the flow (Alben et al., 2002).
Chapter 6

Resonant Hot Beam Pressure Sensor

6.1 Introduction

Thermal Pirani gauges have been widely used in pressure measurements in gaseous environments (Weng and Shie, 1994; Bedo et al., 2000). A typical Pirani gauge consists of a doubly clamped beam whose temperature is higher than the surrounding gas environment. The working principle is based on the increased heat transfer from the hot beam to the surrounding gas with increasing gas thermal conductivity $k_g$ with pressure $p$ (Mastrangelo and Muller, 1991; Lee et al., 2007). The operating pressure range of Pirani sensors can be predicted by considering the mean free path of the gas molecules $\lambda$ and the gap between the beam and the substrate underneath $d$. An empirical relation between $p$, $d$ and $k_g$ has been verified in a number of studies (Doms et al., 2005; Khosraviani and Leung, 2009; Li et al., 2010; Bruschi et al., 2006). In the low pressure limit, the gas density is too low to transfer heat efficiently to the surrounding gas. In the high pressure limit, gas thermal conductivity reaches its continuum limit and the heat transfer rate is independent of pressure. Therefore only in a certain pressure range heat transfer rate is proportional to pressure.

With increasingly miniaturized sensors (Puers et al., 2002; Mailly et al., 2009), non-contact measurement methods become desirable. In this study, we describe how to measure the changes in pressure by optically monitoring the resonance frequency shift of a doubly clamped bilayer beam. For this purpose, we fabricate a Pt-coated SiN fixed-fixed beam (200 nm SiN and 35 nm Platinum). In figure 6.1, we show
Figure 6.1: Cartoon representation of a doubly clamped beam oscillating with amplitude $\delta$ and frequency $f$ at temperature $T$ in a vacuum chamber at pressure $P$ and ambient temperature $T_{\text{amb}}$. The beam is driven by applying an alternating voltage difference between the contact pads. Beam’s displacement is monitored optically with a Michelson interferometer illustrated as incident and reflecting light beams in the drawing.

an illustration of the fixed-fixed beam placed in a vacuum chamber. An AC voltage difference is applied to the ends of the beam to heat and oscillate it. Since thermal expansion coefficients of the two materials are different, upon heating the two layers expand different amounts. This nonuniform expansion creates an axial force on the beam which changes the resonance frequency (Pandey et al., 2010; Bargatin et al., 2007). The beam is a conductor and its temperature is monitored by measuring its electrical resistance with the four-probe method. Our temperature measurements agree well with the empirical laws for pressure-dependent heat transfer from a hot beam with a substrate underneath. We also measure temperature-dependent resonance frequency shifts (Pandey et al., 2010; Zhang et al., 2013) of the beam during pressure sweeps. The oscillation amplitude $\delta$ and frequency $f$ is monitored with a Michelson interferometer using a 632 nm red HeNe laser at constant intensity. By establishing the thermal-mechanical relation between the resonance frequency shift and the temperature-induced axial force, we relate the resonance frequency shifts of the beam to the gas pressure in the chamber.
6.2 Theory

6.2.1 Heat Rate Equation

The total power input to the beam is \( Q_{in} = \dot{Q}_{abs} + \dot{Q}_{el} \) where \( \dot{Q}_{abs} \) is the absorbed laser power and \( \dot{Q}_{el} = V^2/R_{el} \) is the power generation due to joule heating (Incropera and Dewitt, 1985). At equilibrium, the heat flow rate from the hot beam to the surrounding gas is
\[
\dot{Q}_f = \frac{k_g A_s \Delta T}{d},
\]
and to its anchors where it's attached to the substrate is
\[
\dot{Q}_s = \frac{k_s A_c \Delta T}{L}.
\]
Here, \( k_g \) and \( k_s \) are the thermal conductivities (\( \text{W/m·K} \)) of the gas and the beam; \( A_s \) and \( A_c \) are the surface and the cross sectional areas (\( \text{m}^2 \)) of the beam; \( \Delta T = T - T_{amb} \) is the difference between the average temperature (K) of the beam and the temperature of the gas or the anchors (here we take \( T_{gas} = T_{anchors} = T_{amb} \)); \( L \) is the length (m) of the beam and \( d \) is a characteristic length of the system.

We can write the heat rate equation at equilibrium as (Incropera and Dewitt, 1985)
\[
\dot{Q}_{in} = \dot{Q}_{out} = \frac{k_s A_c \Delta T}{L} + \frac{k_g A_s \Delta T}{d}.
\]

Now consider that we place the beam in a vacuum chamber and decrease the pressure of the system. Then in equation (6.1), \( k_g \to k_g(P) \), \( \Delta T \to \Delta T(P) \), \( \dot{Q}_{in} \to \dot{Q}_{in}(P) \) as \( R_{el} \to R_{el}(T(P)) \), and \( k_s(T(P)) \approx \text{constant} \) in the temperature range we are considering, \( 293 \text{ K} < T < 340 \text{ K} \). The pressure dependence of gas thermal conductivity constitutes the basic working principle of a thermal pirani sensor. Heat flow to the gas increases as the gas thermal conductivity increases with pressure in a certain pressure range resulting in lower average temperature of the beam, \( \Delta T(P) \). In order to understand this working principle we must first understand the pressure dependence of the gas thermal conductivity.
Pressure Dependence of Gas Thermal Conductivity

Thermal conductivity of a dilute gas

Consider a closed container filled with a dilute gas (Reif, 2009). The container has dimensions $x$, $y$ and $z$. Imagine a $x - y$ plane at $z = 0$ across which temperature is uniform, but a mean free path, $\lambda$, above and below this plane, there exists a temperature gradient $\partial T / \partial z = \Delta T / 2\lambda$. In figure 6.2, we illustrate the cross section of the container on the $x - z$ plane. The dashed lines are the cross sections of the $x - y$ planes at $z = \pm \lambda$. Thermal conductivity of the gas can be calculated by considering the mean energy transport per unit time per unit area across the $x - y$ plane at $z = 0$. The particles above and below this plane have mean energies $\bar{e}(z + \lambda)$ and $\bar{e}(z - \lambda)$. The total number of particles, $N$, crossing the plane is proportional to the number density of molecules in the container, $n$, and the average thermal velocity of the molecules, $\bar{v}$, $N = n \bar{v}$. The heat flux, $Q_z = -k \frac{\partial T}{\partial z}$, across this plane is given by
Defining the specific heat per molecule $c = \frac{\partial \epsilon}{\partial T}$, we find that

$$k_g \sim n \bar{\nu} c \lambda.$$

The thermal conductivity of a dilute gas depends only on the particle speed, $\bar{v}$, hence temperature, but is independent of the gas pressure. Although both $n$ and $\lambda$ are pressure dependent, this dependence is cancels out as $\lambda \propto n^{-1}$. This result is valid only the range, $Kn = \frac{\lambda}{d} \ll 1$, where the mean free path, $\lambda$ of the gas particles are much smaller than the characteristic length of the system, $d$ (in this case it's the dimensions of the container, $x$, $y$ and $z$). In this region, the particles collide with themselves more frequently than the walls of the container. However, below a certain number density inside the container, the collision rate with the container walls become comparable to the collision rate between particles, $Kn \sim 1$. The energy transport, hence the heat flux, across the imaginary plane at $z = 0$ decreases. In this range, the thermal conductivity, $k_g \rightarrow k_g(n) = \kappa_g(P)$ and Pirani sensors operate in this range. Finally, close to perfect vacuum, $Kn \gg 1$, the net energy transport tends to vanish as $k_g \rightarrow 0$. It's this $Kn$ dependence of the thermal conductivity that's exploited in thermal Pirani sensors.
Estimation of the Working Pressure Range of a Pirani Sensor

An empirical expression for gas thermal conductivity valid for the entire \( Kn \) range is given by (Mastrangelo and Muller, 1991)

\[
k_g(P) = k_g^c \left[ \frac{P/P_0}{1 + (P/P_0)} \right], \tag{6.4}
\]

where \( k_g^c \) is the gas thermal conductivity in the continuum limit (i.e, \( Kn \ll 1 \)) and \( P_0 = P_0(\text{Kn}) \) is the empirical transition pressure below which \( k_g \rightarrow k_g(P) \). This transition takes place around \( Kn \approx 1 \) when the mean free path of the gas molecules are comparable to the characteristic length of the system, \( \lambda \approx d \). The mean free path for an ideal gas at pressure \( P \) and temperature \( T \) is given by the expression (Reif, 2009),

\[
\lambda = \frac{k_B T}{\sqrt{2 \pi \sigma^2 P}}, \tag{6.5}
\]

where \( k_B \) is the Boltzmann constant and \( \sigma \) is the collision cross section. Replacing \( P \) with \( P_0 \) and \( \lambda \) with \( d \), one finds the empirical transition pressure,

\[
P_0 = \frac{k_B T}{\sqrt{2 \pi \sigma^2 d}},
= \frac{C T}{d} \tag{6.6}
\]

where \( C = k_B / \sqrt{2 \pi \sigma^2} \approx 10^{-4} \) is a constant (Brun et al., 2012). Plugging equation (6.6) into equation (6.4), we find an empirical expression for the gas thermal conductivity,

\[
k_g(P) = k_g^c \frac{1}{1 + CT/Pd}, \tag{6.7}
\]

Plugging equation (6.7) into equation (6.1), we obtain an expression for the
overtemperature of the beam as a function of $P$ and $d$,

$$
\Delta T(P) = \dot{Q}_{in}(P) \left( k_s \frac{A_c}{L} + k_g \frac{A_s}{d} + \frac{1}{P d} \right)^{-1} . \tag{6.8}
$$

In the Pirani sensor architecture, the characteristic length refers to the gap distance between the beam and the substrate underneath (see figure 6.6b). Hence, in equation (6.8), the expression $k_g A_s/d$ refers to the thermal conductance of the gas in the continuum limit, $G_g$. This implies that the shorter the gap between the beam and substrate, the more efficient the heat transfer rate. The first term in the parentheses is the lumped thermal conductance of the beam, $G_s = k_s A_c / L$, and can be estimated by considering equation (6.8) in the vacuum limit where $G_s = \dot{Q}_{in}^{vac} / \Delta T^{vac}$. Plugging $G_s$ back into equation (6.8), the final expression for the overtemperature of the beam is given by

$$
\frac{\Delta T(P)}{\Delta T^{vac}} = \frac{\dot{Q}_{in}(P)}{\dot{Q}_{in}^{vac}} \left( 1 + k_g A_s \frac{\Delta T^{vac}}{d \dot{Q}_{in}^{vac}} \frac{1}{1 + \frac{C_T}{P d}} \right)^{-1} . \tag{6.9}
$$

It's obvious from equation (6.9) that the only way for decreasing the beam's temperature is by increasing the pressure. The fact that beam's electrical resistance decreases with decreasing temperature rules out the possibility of $\dot{Q}_{in}(P)$ having any additional effect on beam's heat loss. Hence, by monitoring the temperature of the beam, one can deduce the gas pressure. In section (6.5), we will show our experimental results on the linear relation between the change in beam's temperature and change in gas thermal conductivity, equation (6.7).
6.2.3 Pressure sensing through temperature dependent resonance shift of a bilayer doubly clamped beam

The equation of motion for the transverse displacement, $\zeta$, of a fixed-fixed beam under tension, $\Gamma$, is given by (Timoshenko et al., 1974)

$$\rho_s A_c \frac{\partial^2 \zeta}{\partial t^2} + EI \frac{\partial^4 \zeta}{\partial x^4} - \Gamma \frac{\partial^2 \zeta}{\partial x^2} = 0,$$

(6.10)

$$\zeta|_{x=0=L} = \frac{\partial \zeta}{\partial x}|_{x=0=L} = 0,$$

where $\rho_s$, $A_c$, $E$, $I$ are the density, cross sectional area, Young’s modulus and moment of inertia of the beam. A solution that satisfies the partial differential equation in equation (6.10) for the $n^{th}$ mode of the beam can be written as

$$\zeta_n(x,t) = \zeta_n(x)e^{i\omega_nt}. \quad (6.11)$$

In the absence of the tension term in equation (6.10), the spatial dependence of the transverse displacements could be assumed as $\zeta(x) \sim e^{\beta x}$, and a general solution for $\zeta(x)$ can be written as

$$\zeta_n(x) = a_n \cos(\beta_n x) + b_n \sin(\beta_n x) + c_n \cosh(\beta_n x) + d_n \sinh(\beta_n x). \quad (6.12)$$

Using the boundary conditions given in equation (6.10), it’s found that $a_n = -c_n$ and $b_n = -d_n$ with $\beta_n$ satisfying the condition $\cos(\beta_n L) \cosh(\beta_n L) - 1 = 0$. Then the general expression for the deflection of a fixed-fixed beam at its $n^{th}$ mode becomes (Cleland, 2003)

$$\zeta_n(x) = a_n (\cos(\beta_n x) - \cosh(\beta_n x)) + b_n (\sin(\beta_n x) - \sinh(\beta_n x)), \quad (6.13)$$

$$a_n = \frac{(\cos(\beta_n L) - \cosh(\beta_n L))}{(\sin(\beta_n L) + \sinh(\beta_n L))},$$

$$b_n = \frac{(\cos(\beta_n L) + \cosh(\beta_n L))}{(\sin(\beta_n L) - \sinh(\beta_n L))}.$$
Using equation (6.13) and equation (6.11) in equation (6.10), the natural frequency of the \( n \)th mode is found as

\[
-\omega_n^2\rho_s A_c \zeta_n(x) + EI \beta_n^4 \zeta_n(x) = 0,
\]

\[
\Rightarrow \omega_n = \sqrt{\frac{EI}{\rho_s A_c} \beta_n^2}.
\]

(6.14)

However, in the presence of an axial tension, \( \Gamma \), a solution of the form in equation (6.12) does not satisfy the partial differential equation in equation (6.10). The solution in this case requires perturbative methods. Nevertheless, without getting into hefty calculations, the fundamental resonance frequency can still be approximated with Rayleigh’s method (Timoshenko et al., 1974) where the Rayleigh quotient \( RQ \) is proportional to the ratio of the cycle averaged kinetic and potential energies of the beam, \( RQ = \frac{E_{\text{kinetic}}}{E_{\text{potential}}} \) such that,

\[
E_{\text{kinetic}} = \frac{\omega_n^2 \rho_s A_c}{2} \int_0^L \phi(x)^2 dx,
\]

\[
E_{\text{potential}} = \frac{EI}{2} \int_0^L \left( \frac{\partial^2 \phi}{\partial x^2} \right)^2 dx + \frac{\Gamma}{2} \int_0^L \left( \frac{\partial \phi}{\partial x} \right)^2 dx,
\]

\[
RQ = \frac{EI \int_0^L \left( \frac{\partial^2 \phi}{\partial x^2} \right)^2 dx + \Gamma \int_0^L \left( \frac{\partial \phi}{\partial x} \right)^2 dx}{\omega_n^2 \rho_s A_c \int_0^L \phi(x)^2 dx},
\]

(6.15)

\[
RQ = 1,
\]

\[
\Rightarrow \omega_n^2 = \frac{EI \int_0^L \left( \frac{\partial^2 \phi}{\partial x^2} \right)^2 dx + \Gamma \int_0^L \left( \frac{\partial \phi}{\partial x} \right)^2 dx}{\rho_s A_c \int_0^L \phi(x)^2 dx},
\]

where \( \zeta_n(x) = \phi(x) \) is a guess solution for the deflection of the fundamental mode that satisfies the boundary conditions in equation (6.10). By setting \( RQ = 1 \), one can
readily obtain an approximation for \( \omega_0 \). The first term in equation (6.15) represents the fundamental frequency in the absence of tension given in equation (6.14) at \( n = 0 \). Let's call this term \( \omega_0^2(0) \). The second term represents the frequency change due to stress accumulation on the beam and let's call it \( \omega_0^2(\Gamma) \). Then equation (6.15) can be written as \( \omega_0^2 = \omega_0^2(0) + \omega_0^2(\Gamma) \). Then, \( \omega_0^2 - \omega_0^2(0) = \omega_0^2(\Gamma) \Rightarrow \Delta \omega_0 = 2\omega_0(0) \) in the limit \( \Delta \omega_0/\omega_0(0) \ll 1 \). Finally an expression for the stress induced fundamental frequency shift of the fixed-fixed beam can be written as \( \Delta \omega_0/\omega_0(0) \sim \omega_0^2(\Gamma)/\omega_0^2(0) \). From equation (6.15), \( \omega_0^2(\Gamma) \sim \frac{\Gamma}{\rho_s A_c L^2} \), then, we find the normalized frequency shift to be proportional to the axial stress applied to the beam

\[
\frac{\Delta \omega_0}{\omega_0(0)} \sim \frac{\Gamma}{\rho_s A_c L^2 \omega_0^2(0)}. \quad (6.16)
\]

For now, we limit ourselves to figure out the functional dependence and therefore consider all constants coming from the integration to be 1. In this sense the result of equation (6.16) can be generalized to higher order modes as well. The constant, \((\rho_s A_c L^2 \omega_0^2)^{-1}\) in equation (6.16) can be found by plugging in a guess solution of the form \( \phi(x) = x^2(L - x)^2 \) that satisfies the boundary conditions and solving for the integrals in equation (6.15). Accuracy of the constants can be increased by using an improved Ritz method (Timoshenko et al., 1974). This method is not limited to estimate the fundamental mode only; it can be used for higher modes as well.

Finally, the relation between the induced stress (in units of \( N \)) and temperature is given by (Pandey et al., 2010)

\[
\Gamma \sim E A_c (\alpha_1 - \alpha_2) \Delta T. \quad (6.17)
\]

Plugging equations (6.17) into equation (6.16), we obtain the temperature dependence
of the frequency shift as
\[
\frac{\Delta \omega_0}{\omega_0(0)} \sim \frac{E A_c (\alpha_1 - \alpha_2) \Delta T}{\rho_s A_c L^2 \omega_0^2(0)}.
\] (6.18)

Multiplying and dividing both sides of equation (6.18) with the maximum temperature difference, \(\Delta T_{\text{vac}}\), a non-dimensional relation between the normalized frequency shift and normalized overtemperature change is given by
\[
\frac{\Delta \omega_0}{\omega_0(0)} \sim C_1 \frac{\Delta T}{\Delta T_{\text{vac}}},
\] (6.19)

and plugging equation (6.9) into equation (6.19), we obtain
\[
\frac{\Delta \omega_0}{\omega_0(0)} \sim C_1 \frac{\tilde{Q}_{in}(P)}{\tilde{Q}_{in}^{\text{vac}}} \left(1 + \frac{k^c A_s \Delta T_{\text{vac}}}{\tilde{Q}_{in}^{\text{vac}}} \frac{1}{1 + \frac{c T}{P_d}}\right)^{-1}.
\] (6.20)

Equation (6.20) relates the resonance frequency shift of the beam to the pressure dependent heat transfer from the beam. Therefore, in an indirect way, we relate the frequency shift to the pressure inside the chamber. In our experiments, we approximate \(\tilde{Q}_{in}\) to be constant and equation (6.20) reduces to the relation between the frequency shift and pressure. In section 6.5, we experimentally measure this relation.

### 6.3 Instrumentation

#### 6.3.1 Four-Probe Measurement of Beam’s Electrical Resistance

We used the four-probe method to measure the beam’s electrical resistance in our experiments (Valdes, 1954). In this technique the current carrying and voltage sensing electrodes are separated to avoid the contribution of contact resistances and resistance of the current carrying wires. Figure 6.3 is a schematic which illustrates the four-probe geometry used in our experiment. The beam is connected to four square contact
pads (3 mm × 3 mm) via unsuspended wires with much smaller aspect ratio (3 mm × 500 μm) compared to the beam (28 μm × 2 μm). The reason to keep the contact pads and contact wires with smaller aspect ratio is to enhance the contribution of the beam’s electrical resistance to the measured total resistance. The resistance of a thin film with uniform thickness can be written in terms of sheet resistance, $R_s$ (Smits, 1958). In a regular three dimensional material, $R_{el} = \rho_e \frac{l}{wt}$ where $\rho_e$ is the electrical resistivity, $l$, $w$ and $t$ are the length, width and thickness of the material. In a thin film with uniform thickness resistivity per unit thickness of the sheet is, $R_s = \rho_e/t$. The total resistance can be written as $R_{el} = R_s \frac{l}{w}$. Hence, the maximum contribution to the total resistance of the thin film geometry in figure 6-3 comes from the beam since it has the largest aspect ratio as obvious in figure 6-3.

The contact pads are bonded with thin aluminum wires using silver paint. A
Stanford Research model SR830 DSP Lock-in-Amplifier is used to determine the beam’s electrical resistance. A 30 Hz sinusoidal voltage from (varied in the range $0.001 V - -0.5 V$) is passed over a $100k \Omega$ resistor and converted into current. The current is applied to the outer pads while the voltage drop is measured between the inner pads with the lock-in amplifier. A linear fit to the beam’s $I - V$ curve displayed in the inset of figure 6.7a gives a resistance value of $R_{el} \approx 163 \Omega$ around room temperature.

**Temperature Detection Sensitivity**

The minimum detectable temperature fluctuations, $\delta T$, with voltage measurements across the beam are related to the temperature response and the fluctuations of the measured voltage, $\Delta V$ due to Johnson noise (Cleland, 2002):

$$\delta T \approx R^{-1}_V \Delta V,$$  

(6.21)

where $R_V = \partial V / \partial T$ is the responsivity of the voltage with respect to temperature. 

$\Delta V = \sqrt{4k_B T R_{el} \Delta f}$ from Nyquist theorem (Zwanzig, 2001) where $k_B$ is the Boltzmann constant, $T$ is the temperature of the beam, $R_{el}$ is the beam’s electrical resistance and $\Delta f$ is the measurement bandwidth. In the right hand side of equation (6.21), replacing $V$ with $IR$ in $R^{-1}_V$ and dividing and multiplying by $\sqrt{R_{el}}$ gives the minimum detectable temperature in terms of electrical driving power, $Q_{el} = I^2R$,

$$\delta T \approx R_{el} \frac{\partial T}{\partial R_{el}} \sqrt{\frac{4k_B T \Delta f}{Q_{el}}},$$  

(6.22)

Finally dividing both sides of equation (6.22) by $T$ gives the normalized minimum detectable temperature as

$$\frac{\delta T}{T} \approx \frac{R_{el}}{T} \frac{\partial T}{\partial R_{el}} \sqrt{\frac{4k_B T \Delta f}{Q_{el}}}. $$  

(6.23)
Here, $\partial T/\partial R_{el}$ is inverse of the temperature coefficient of the beam’s electrical resistance (TCR) and is found to be $\approx 5 \, K/\Omega$. In our experiments the minimum detectable temperature change at the highest average temperature of the beam, $T \approx 335 \, K$ is found to be $\delta T \approx 2 \, mK$. In this configuration, $R_{el} \approx 163 \, \Omega$, $Q_{el} \approx 3mW$ and $\Delta f = 300 \, kHz$ is the bandwidth of the input signal amplifier in the lock-in amplifier.

### 6.3.2 Vibration Detection with Path Stabilized Michelson Interferometry

The mathematical expression for the electric field of a monochromatic plane wave of wavelength, $\lambda$, traveling in the $z$ direction with velocity $c$ can be found from the solution of the wave equation and is written as $A = a \exp[i(\omega t - k z)]$ (Saleh and Teich, 2007). Here $a$ is the maximum amplitude, $\omega = 2\pi c/\lambda$ is the angular frequency and $k = 2\pi/\lambda$ is the wavenumber. A monochromatic plane wave reflecting from a vibrating surface experiences a change in its optical path length. If the peak amplitude of the vibrations perpendicular to the incident beam is $\delta$, then the total change in the optical path length is $2\delta$ corresponding to a phase change $2k\delta$. Then the expression for the electric field becomes $A = a \exp[i\omega t - k(z-2\delta)]$ (Wagner, 1990). This small phase change cannot be measured with traditional intensity or power measurements. The basic principle of interferometric measurement is converting the phase change in the optical signal caused by the change in optical path length into intensity variations. In this study vibrations of the doubly clamped beam are detected with path stabilized Michelson Interferometry.

Figure 6.4a illustrates the schematic of a typical Michelson Interferometer. A monochromatic, coherent light beam (such as light emitted from a HeNe laser at 632 nm), is split into two beams with a beam splitter. One beam is directed onto a reference mirror at a distance $z_R/2$ and the other is directed onto the vibrating object at a distance $z_O/2$. Both beams reflect back to the beam splitter where they interfere with each other and are directed onto the photo-detector. The electric field
of the reference and object beams can be written as

\[ A_R = a_R \exp(i (\omega t - k z_R)), \]

\[ A_O = a_O \exp(i (\omega t - k (z_o - 2 \delta))). \]  

(6.24)

A Photo-detector measures incident optical power, \( P \) which is proportional to the light intensity on the photo-detector, \( I \). The total light intensity in an interferometric setup is simply the square of the sum of the electric field amplitudes of the reference and object beams

\[ I = |A_O + A_R|^2 = (A_O + A_R)(A_O^* + A_R^*) \]

\[ = a_O^2 + a_R^2 + 2a_Oa_R \cos[k(z_R - z_O) + 2k \delta] \]

\[ = (a_O^2 + a_R^2) \left\{ 1 + 2 \frac{a_Oa_R}{a_O^2 + a_R^2} \cos[k(z_R - z_O) + 2k \delta] \right\} \]

\[ = (a_O^2 + a_R^2) \left\{ 1 + 2 \frac{a_Oa_R}{a_O^2 + a_R^2} [\cos k(z_R - z_O) \cos 2k \delta - \sin k(z_R - z_O) \sin 2k \delta] \right\}. \]

(6.25)

When the vibrations of the beam are much smaller than the optical wavelength, \( \delta \ll \lambda \), equation (6.25) can be simplified as

\[ I \approx (a_O^2 + a_R^2) + 2a_Oa_R \cos k(z_R - z_O) - 2a_Oa_R 2k \delta \sin k(z_R - z_O). \]  

(6.26)
Here, the first two terms are the constant background intensity (DC signal) and the third term is due to vibrations of the beam, $\delta$ (AC signal). Hence, an interferometer converts phase fluctuations due to displacement fluctuations into intensity fluctuations that can be detected by a photo-detector. When the path difference is an integer multiple of the wavelength, $z_R - z_O = n\lambda$ where $n = 0, 1, 2...$, the optical intensity on the photo-detector is maximum and comes from only the DC background, $I_{DC}^{max} \approx (a_O^2 + a_R^2) + 2a_Oa_R$. Similarly, when $n$ is a half integer $z_R - z_O = n\lambda/2$, the optical intensity on the photo-detector is minimum, $I_{DC}^{min} \approx (a_O^2 + a_R^2) - 2a_Oa_R$. Then, $a_Oa_R \approx (I_{DC}^{max} - I_{DC}^{min})/4$. At $z_R - z_O = n\lambda/4$, the AC intensity signal, $I_{AC}$ due to beam vibrations is maximum and

$$I_{AC} \approx (I_{DC}^{max} - I_{DC}^{min}) \frac{2\pi}{\lambda} \delta.$$ \hspace{1cm} (6.27)

Since the intensity on the photo-detector is proportional to optical power,

$$I_{AC} \propto P_{AC} = \frac{V_{AC}}{R_{AC}R_{pd}},$$

$$I_{DC}^{max} \propto P_{DC}^{max} = \frac{V_{DC}^{max}}{R_{DC}R_{pd}},$$ \hspace{1cm} (6.28)

$$I_{DC}^{min} \propto P_{DC}^{min} = \frac{V_{DC}^{min}}{R_{DC}R_{pd}},$$

where $R_{pd}$ is the responsivity of the photo-detector and has units of $A/W$. $R_{AC}$ and $R_{DC}$ are the AC and DC gains of the photo-detector amplifier. $V_{AC}$, $V_{DC}^{max}$ and $V_{DC}^{min}$ are the AC, maximum DC and minimum DC voltages at the output of the photo-detector, respectively. Then the amplitude of the beam vibration, $\delta$ can be represented in terms of measured voltages and photo-detector gains as

$$\delta = \frac{\lambda}{2\pi} \frac{V_{AC}}{V_{pp}} \frac{R_{DC}}{R_{AC}}.$$ \hspace{1cm} (6.29)
where $V_{pp} = V_{DC}^{max} - V_{DC}^{min}$ is the peak-to-peak DC voltage. Higher the $V_{pp}$, smaller the minimum detectable displacement. The sensitivity of the Michelson interferometer is maximum when the path difference $z_R - z_O \approx n\lambda/4$. This can be seen by plotting equation (6.26) with $a_O = a_R$ as shown in figure 6.4b. The maximum slope and linearity is in the region when the path length difference is between $\lambda/4$ and $\lambda/2$. Here, a small fluctuation in the displacement will cause the maximal intensity fluctuation in the interference signal. Conversely, when the path difference is a multiple of either $\lambda$ or $\lambda/2$, the slope and the intensity fluctuations are zero. At these points, the interferometer is insensitive to vibrations of the beam. Therefore, to operate the interferometer in the most sensitive region, the reference mirror is attached to a piezo actuator and the DC component of the photo-detector signal is fed back to a PID controller which drives the piezo actuator to keep the path difference around $\lambda/4$.

**Interferometric Detection Sensitivity**

The ultimate sensing capability of a path stabilized Michelson interferometer is dependent on how strong the signal power, $P_{sig}$ is compared to noise power, $P_{noise}$. This noise is due to various sources in the system including the light source, detector, processing electronics and the environment. Considering all the noise sources, the minimum detectable signal is determined when signal-to-noise ratio $SNR = \frac{P_{sig}}{P_{noise}} = 1$.

The low frequency noise caused by environmental fluctuations such as air turbulence, room vibrations or drifts in the optical system are mostly taken care of by the path-stabilization described previously. Noise due to thermal fluctuations in the photo-detector and processing electronics is broadband and strongly dependent on temperature and bandwidth. Reducing the temperature and the bandwidth of the processing electronics, significantly reduces the thermal noise (Wagner, 1990). Shot noise is caused by the statistical fluctuations in the number of the photons incident on the photo-detector (Fox, 2006). The current generated by the photo-detector is
proportional to light intensity, hence the number of photons. Fluctuations in photon distribution cause fluctuations in the current generated. Hence, shot noise is proportional to the signal amplitude unlike other noise sources which do not change with light intensity. By increasing the light intensity, shot noise can be increased to be the dominant most dominant noise contributor and the interferometer becomes shot noise-limited.

Shot noise power can be estimated in terms of voltage fluctuations at the output of the photo-detector considering the fluctuations in the number of photons impinging on the photo-detector. Upon arrival of a photon, the photo-detector generates a time varying photocurrent, $i(t)$, which can be decomposed into a time independent average current $< i >$ and a time varying fluctuation $\Delta i(t)$, such that $i(t) = < i > + \Delta i(t)$. The photons obey Poissonian statistics and the fluctuations, $\Delta N$, in the average number of photons impinging on the photo-detector, $< N >$, follow the relation $(\Delta N)^2 \propto < N >$. Since $i(t)$ is related to the number of photoelectrons generated per second, then the variance in the photocurrent, $\Delta i(t)$, will also obey Poissonian statistics, $(\Delta i)^2 \propto < i >$. The exact relation between the average photocurrent and its fluctuating component is given by $(\Delta i)^2 \propto 2e\Delta f < i >$, where $e = 1.602 \times 10^{-19}$ coulombs is the electronic charge and the $\Delta f$ is the frequency bandwidth where the current fluctuations are measured. Then the voltage fluctuations at the output of the photo-detector amplifier, $(\Delta V)^2$, are given by $(\Delta V)^2 = 2e\Delta f(R_{AC})^2 < i >$ where the average photocurrent $< i > = < V_{DC} > / R_{DC}$. The shot noise power $P_{\text{shot}} = (\Delta V)^2 / R_{\text{out}}$ can be written in terms of the average DC voltage and measurement bandwidth as

$$P_{\text{shot}} = 2e\Delta f \left( \frac{R_{AC}}{R_{DC}R_{\text{out}}} \right) < V_{DC} >,$$  \hspace{1cm} (6.30)$$

where $R_{\text{out}}$ is the output impedance of the photo-detector. From equations (6.27) and
(6.28), the AC signal power, $P_{\text{sig}}$, can be written as

$$P_{\text{sig}} = \frac{V_{pp}}{R_{DC}\overline{R}_{pd}} \frac{2\pi}{\lambda \delta}. \tag{6.31}$$

Then the SNR in a shot noise limited interferometer takes the form:

$$SNR = M \frac{4e\Delta f\overline{R}_{pd}R_{\text{out}}}{(R_{AC})^2} \frac{2\pi}{\lambda \delta}, \tag{6.32}$$

where $M = V_{pp}/2 < V_{DC}$ is the modulation depth which takes values between 0 and 1 proportional to strength of the fringe contrast. An interferometer with a high modulation depth combined with a high responsivity photo-detector and a short wavelength laser source results in higher SNR. Another way of increasing the SNR is by increasing the amplitude of the beam vibrations by externally driving the beam. Minimum detectable displacement $\zeta_{\text{min}}$ and SNR of our shot noise-limited interferometer can be calculated using equations (6.29) and (6.32). $< V >$ is proportional to the average laser power incident on the photodiode, $P_{\text{laser}} = \frac{<V>}{R_{DC}\overline{R}_{pd}}$ and the $(\Delta V)^2/\Delta f = \frac{P_{\text{noise}}R_{DC}}{\Delta f}$. In order to measure the spectral density of the voltage fluctuations, $(\Delta V)^2/\Delta f$ as a function of the mean voltage, $< V >$, at the output of the amplifier experimentally, the noise power, $P_{\text{noise}}$, at the output of the amplifier is measured between 2 MHz and 5 MHz with $\Delta f = 100$ kHz as the average laser power, $P_{\text{laser}}$, hence $< V >$ is increased gradually. The noise power per unit bandwidth, $P_{\text{noise}}/\Delta f$ for a number of different incident laser power values are shown in figure 6.5a. From this information we plot in figure 6.5b, the average spectral density of voltage fluctuations as a function of the average voltage. The solid line is calculated using equation (6.30) and plugging $P_{\text{shot}} = (\Delta V)^2/R_{\text{out}}$. Experimental values are in good agreement with the first principles calculation and demonstrate that our interferometric detection scheme is shot noise limited with a minimum detectable voltage fluctuations per unit voltage and bandwidth, $(\Delta V)^2 \approx 5 \times 10^{-14}$ V. Plugging this
back into equation (6.29), under typical operating conditions, we obtain the minimum detectable displacement, $\delta_{min} \approx 50 \text{ fm}$. For a beam displacement of $\sim 1 \text{ nm}$, a typical resonance amplitude in our experiments, SNR $\sim 2 \times 10^4$.

### 6.4 Experimental System

To test these ideas we fabricated a suspended 200 nm thick SiN beam ($L = 28 \text{ \mu m}$, $w = 2 \text{ \mu m}$ and $d = 7 \text{ \mu m}$) coated with a 30 nm e-beam evaporated Pt layer on top. The beam is connected to four contact pads (3 mm x 3 mm) via much wider and longer unsuspended wires (3 mm x 500 \mu m). An SEM image of the pads and contact wires is shown in figure 6.6a and a close up view of the suspended beam is shown in 6.6b. The experiment simply consists of recording the average temperature and resonance frequency of the beam simultaneously while performing a pressure sweep.

The temperature of the beam is measured by monitoring the change in its electrical resistance. The linear relation between the electrical resistance and the temperature
Figure 6.6: Micrograph of the device. (a) A low magnification SEM image showing the contact pads and contact wires with the beam highlighted with a dashed rectangle. (b) A close up of the beam with geometric parameters of a conductor is given by

$$R_{el} = R_{el}^0 (1 + \xi(T - T_{amb})),$$

(6.33)

where $\xi$ is the temperature coefficient of the beam's electrical resistivity (TCR). First, we determined the TCR by placing the chip on a hot plate and monitoring the beam’s electrical resistance with 4-Probe technique while increasing the temperature of the hotplate. A plot of measured $R_{el}$ vs $T$ is shown in figure 6.7a. The inset shows the $I - V$ curve at room temperature, $T_{amb} = 293K$. A linear fit to our data yielded $\xi = 0.19$.

After this initial calibration, we placed the beam into a custom built vacuum chamber with inlet and outlet ports as well as sealed electrical connectors. The chamber is connected to capacitive pressure sensors (MKS instruments) on one end and a vacuum pump on the other. We performed pressure sweeps from 100 mTorr
to 800 Torr while measuring the temperature and resonance frequency of the beam simultaneously. We used a Stanford Research model SIM 921 AC Resistance Bridge to measure the beam's electrical resistance. From our calibration value of \( \xi \), we converted the resistance values into temperature. We show the absolute average temperature of the beam as a function of chamber pressure in figure 6.7b. In order to increase the SNR in our experiments we employed a voltage driven measurement scheme using a SR844 DSP lock-in amplifier. We applied a sinusoidal driving voltage to the ends of the beam which heats the sample and actuates it due to stress induced deformations. We measured the amplitude and frequency of the oscillations of the beam with a custom built path-stabilized Michelson interferometer. Figure 6.8a shows the spectra of the beam's displacement in its fundamental mode in vacuum and near atmospheric pressure as a function of driving frequency. Input driving power, \( Q_{\text{el}} = -5 \) dBm. The frequencies corresponding to the peak displacements are the resonance values and are extracted by fitting a Lorentzian to the experimental data. Similar measurements are performed for the beam's first harmonic mode.

### 6.5 Results and Discussion

In section (6.2), we derived a general expression for the beam's temperature change. Examining equation (6.9), if the input power, \( Q_{\text{in}} \), is constant, the only mechanism that changes the beam's temperature is the heat flow to the gas. In our experiments the input power \( Q_{\text{in}} \) is almost constant increasing only 0.2 % from vacuum to atmospheric pressure. Therefore, the measured temperature change, \( \Delta T \), is directly related to the change in gas thermal conductivity, \( k_g \). In equation (6.9) the temperature change of the beam is defined with respect to the ambient temperature, \( \Delta T(P) = T - T_{\text{amb}} \) and \( \Delta T_{\text{vac}} = T_{\text{vac}} - T_{\text{amb}} \). A consequence of this, as \( P \to 0 \), \( \Delta T(P) \) reaches its maximum while \( k_g \sim 1/[1 + (CT/Pd)] \to 0 \). It's necessary to
Figure 6.7: (a) Calibration of the electrical resistance with temperature. A linear fit to the data yields $\xi = 0.19$. The inset shows the $I - V$ curve at room temperature. (b) Average temperature of the beam during a pressure sweep from 400 mTorr to 800 Torr. The driving power of the lock-in amplifier is -5 dBm. Note that at atmospheric pressure, the temperature of the beam is still higher than room temperature 293 K. (c) Absolute change of the beam’s average temperature with gas thermal conductivity. Both the temperature change and gas thermal conductivity $\rightarrow 0$ at low pressures.

Define the absolute temperature change of the beam in a manner consistent with the change of the thermal conductivity such that in the low pressure limit $\Delta T \rightarrow 0$ as $k_g$ does. Note that beam’s temperature at atmospheric pressure, $T_{atm} = 299$ K, is higher than $T_{amb} = 293$ K (See figure 6.7b). A direct comparison of the beam’s absolute temperature change with the change in gas thermal conductivity valid for the entire pressure range with correct limits can be possible by considering the relation,

$$\left| \frac{T - T_{vac}}{T_{vac} - T_{atm}} \right| \propto \frac{1}{1 + \frac{C_T}{P_d}}.$$  

Here, on the right hand side we used gas thermal conductivity from equation (6.7) with $k_g^c = 1$. We plot equation (6.34) in figure 6.7c. The temperature of the beam changes linearly with the change in gas thermal conductivity agreeing well with empirical predictions of equations (6.7) and (6.9).

Next, we consider the frequency change of the beam due to temperature induced axial stress. In figure 6.8b, we plot the measured resonance frequency of the beam
Figure 6.8: (a) Displacement spectra of the fundamental mode near vacuum and near atmospheric pressure. (b) Resonance frequency of the fundamental mode of the beam during a pressure sweep from 400 mTorr to 800 Torr. The driving power of the lock-in amplifier is -5 dBm. Absolute change of the beam's fundamental resonance frequency with (c) beam's average temperature and (d) gas thermal conductivity.

at its fundamental mode as a function of pressure. We extracted the frequencies by fitting a Lorentzian to the spectra shown in figure 6.8a. Referring to equation (6.19) and redefining the change in frequency similar to that in equation (6.34), we show that the relation,

$$\left| \frac{f - f_{\text{vac}}}{f_{\text{vac}} - f_{\text{atm}}} \right| \propto \left| \frac{T - T_{\text{vac}}}{T_{\text{vac}} - T_{\text{atm}}} \right|,$$

holds in figure 6.8c. Finally, a direct consequence of equations (6.34) and (6.35) is
Figure 6.9: (a) Average temperature (b) resonance frequency of the first mode of the beam during a pressure sweep from 900 mTorr to 800 Torr. The driving power of the lock-in amplifier is 10 dBm. Note that at atmospheric pressure, the temperature of the beam is still higher than room temperature 293 K. (b) (d) Absolute change of the beam's fundamental resonance frequency with (c) beam's average temperature and (d) gas thermal conductivity.

considering the relation between $\Delta f$ vs $1/[1 + (CT/Pd)]$:

$$\left| \frac{f - f_{vac}}{f_{vac} - f_{atm}} \right| \propto \frac{1}{1 + \frac{CT}{Pd}}.$$  

(6.36)

We plot equation (6.36) in figure 6-8d. The linear relationship agrees well with the predictions of equation (6.20). A deviation from linearity is observed in figures (6-8b&c) at higher pressures. With increasing gas density mass loading could be dominating the frequency shift due to heat transfer. In order to induce a heat transfer dependent
frequency shift higher than that which is induced by the mass loading effect, we increased the drive power from -5 dBm to 10 dBm. To avoid high amplitude oscillations of the beam, particularly in the low pressure regime, we monitored frequency shift of the first harmonic mode.

Figure 6-9a shows absolute temperature of the beam as a function pressure. Note that the maximum temperature increased by 20 K. As expected higher input power results in the beam having a higher average temperature. Higher temperature causes a higher mechanical stress inducing a higher frequency shift (figure 6-9b). The frequency shift percentage is roughly 3x higher at higher input power (10 dBm). In Figures (6-9c&d), we plot equations (6.35) and (6.36) for the first harmonic mode. Compared to figures (6-8c&d), the linear relation holds at higher pressures as well, albeit with a small deviation around 800 Torr. Also noticeable from the comparison of figures (6-8d) and (6-9d) is the slope difference. A beam operated at higher driving power has a lower $df/dP$ which means a higher sensitivity. This is reasonable because a higher frequency shift provides a larger dynamic range for the operation of the sensor.

6.6 Conclusion

We monitor the gas pressure in a vacuum chamber through interferometric measurements of the resonance frequency shift of a fixed-fixed bilayer beam. The shift in resonance is induced by applying an axial stress on the beam which is created through different thermal expansions of the layers. This frequency shift is linearly proportional to the stress and the stress is linearly proportional to temperature. Hence by monitoring frequency, we indirectly monitor the temperature which is related to gas pressure. We provided a model that predicts the linear relation between the frequency shift and the applied stress. We showed experimentally that this relation holds for both the fundamental and first harmonic modes of a beam.
Constant miniaturization of MEMS/NEMS packaging requires smaller Pirani sensors and non-contact measurement methods to be employed in Pirani sensor designs. Here, we show a proof-of-concept for non-contact pressure measurement with a hot resonant beam. Combined with laser heating and multiplexed optical measurement of arrays of beams with different gaps, it's possible to cover a wide pressure range without directly interfering with the vacuum system such as wire bonding for temperature readout.
Chapter 7

Conclusion

In this thesis, we study fluid-structure interactions in deformable microchannels. In particular, we develop a technique for measuring pressure distribution in a deformable microchannel by exploiting channel wall deformations under flow. We first measure the membrane distention as a function of uniform hydrostatic pressure. With this initial measurement we generate the “calibration curves” for the deformable channel. By matching the distention profile under flow to the calibration curves, hydrodynamic pressure distribution is obtained. Our measurements in the test sections of deformable microchannels agree well with the analytical approximation of Poiseuille flow in slowly varying channels. At the inlet and outlet regions of the flexible part of our channels we observe significant pressure gradients that present themselves by the high local curvature. The pressure distribution in these regions can be resolved with a two-dimensional analysis including the effect of the axial wall curvature.

In our experiments in Chapter 3, the spatial resolution in a $p(x)$ measurement depends upon the resolution in $\zeta$, the noise in the hydrostatic pressure measurement, and the magnitude of the response of the wall. With our current imaging system, we can detect deflections with $\lesssim 20$ nm precision, and the r.m.s. noise in the hydrostatic pressure transducer is $\sim 10$ Pa. By collecting the constitutive curves in figure 3.3a at smaller pressure intervals, we estimate that we can measure $p(x)$ with $\sim 10$ $\mu$m spatial resolution in this particular system. This method can easily be scaled down to provide sub-micron resolution in a nano-fluidic channel by employing a higher numerical
aperture objective. It may also be possible to extend the method to study time-dependent fluid-structure interactions (Bertram and Tscherry, 2006; Huang, 2001) by collecting surface deformation maps faster (Sampathkumar et al., 2011). By optimizing the averaging time, one should be able to collect high-speed, high-resolution pressure measurements in miniaturized channels. Such advances could open up many other interesting fluid dynamics problems, especially in biological systems.

We show the potential of our method in biological flows with measurements of the two-dimensional IFP field within hydrogels in deformable microchannels. Using this technique, a future study can focus on measurement of the IFP field in microscale tissues. Current studies of interstitial effects on tissue function have focused on interstitial flow, rather than pressure, in part because non-invasive measurement of flow velocity is currently possible (Chary and Jain, 1989). The ability to measure pressure along with flow velocity would enable direct calculation of the hydraulic permeability tensor of porous media, such as tissues and gels. Moreover, the ability to image IFP could yield deeper insights into the role of physical forces in tissue function. For instance, based on indirect evidence, it has been proposed that stable adhesion of endothelium to a scaffold requires a vascular pressure that exceeds interstitial pressure by a critical amount (Wong et al., 2014; Tien, 2014) Similarly, others have hypothesized that excessive IFP may retard the growth of tissues (Nelson and Gleghorn, 2012; DiFiore et al., 1994). The current method would allow a direct test of these and related hypotheses.

This noninvasive method can possibly find applications in characterizing physiological flows. In blood flow in arteries (Ku, 1997) and smaller vessels (Popel and Johnson, 2005), flow-structure interactions are critical in determining functionality (Grotberg and Jensen, 2004; Heil and Jensen, 2003; Heil, 1997). Using our method, for example, one could extract local pressure distribution in an arterial aneurysm,
where the arterial wall degrades and eventually ruptures due to the pressure and shear forces during blood flow (Lasheras, 2007).

In separate experiments in Chapter 2, we study the elastic response of our deformable membrane walls when they are initially in a wrinkled state. We inflate the wrinkled membranes by applying hydrostatic pressure while monitoring pressure and characteristic parameters of the wrinkled membrane, $\zeta$, $A$, $\lambda$ and $\Lambda$. We consider the deformation mechanism during the transition from the wrinkled to the unwrinkled state. Due to high compressive stress on the wrinkled membranes, we assume that the transition happens at constant strain, i.e. without stretching. Geometric scaling relations emerging from this condition agree well with our experiments. Our results indicate that during unwrinkling, the membrane does not stretch and the transformation happens through bending. The most important consequence of membrane expansion through bending is the linear response to pressure fluctuations with an average stiffness, $k$. Elastic membranes whose stiffness are controlled by wrinkles can be used as finely tuned elastic channel walls. By monitoring the wall deflection during flow, it's possible to deduce the average pressure inside the channel instantaneously provided that $k$ is found from separate hydrostatic measurements. With a feedback system, flow rate can be adjusted with respect to the changes in the average pressure inside the channel. Another application could be in frequency and dissipation analysis in oscillatory flows by modeling the membrane-fluid interaction with a damped harmonic oscillator model (Rajauria et al., 2011).

We study fluid-structure interactions at the fluid-solid interface by altering the boundary condition from no-slip to partial slip in flows over porous superhydrophobic membranes. In our experiments on Stokes' second flow over porous superhydrophobic membranes we observe a decrease in fluid-solid friction with decreasing solid fraction. Our results show that this reduction starts when the solid fraction is around 90%
and increases anomalously almost 23 fold at 34% solid fraction. We conjecture the observed anomaly to a stable Knudsen layer of gas percolating through the membrane pores and forming a gas layer between the solid and the liquid. We test the possibility of using porous superhydrophobic membranes in microchannel flows as a drag reducing wall. Our initial measurements and analysis based on our own technique and conventional differential pressure measurement techniques suggest that high pressure gradients at the inlet and outlet of the deformable section may be a significant source of error in drag comparisons. A complete measurement of the drag in these experiments can be possible if the complete pressure distribution on the deformable membrane is known. We suggest that two dimensional analysis including the changes in wall curvature can resolve the pressure distribution in these high pressure gradient regions.

In the final section of this thesis, we study fluid-structure interactions in heat transfer from a hot oscillating beam to the surrounding gas at varying pressures. In particular, we monitor the temperature and resonance frequency of the beam as a function of pressure. Our temperature measurements as a function of pressure agree well with the empirical models. We also observe that resonance frequency of a fixed-fixed bilayer beam shifts with temperature. Through a thermal-mechanical analysis, we relate the changes in temperature of the beam to its resonance frequency shift. Therefore, we relate the changes in pressure to the frequency shift. We propose that this method, with an initial calibration, can be used as an effective non-contact gas pressure sensing mechanism.

In conclusion, we have presented a range of studies on fluid structure interactions. In particular, we focus on characterizing interactions of microscale flows and the confining channel. We have approached the problem from an experimental point of view and characterized fluid-structure interactions in cases both in the large deformation
limit and at the microscopic scale.
References


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