Mechanical Properties of CMOS Thin Films

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ABSTRACT

The development and fabrication of micromachined microsensors and microactuators using IC technology requires reliable mechanical property data of the constituent thin film materials. With thicknesses of about one micrometer, these thin films cannot be easily characterized using standard techniques.

This experimental and theoretical thesis reports the development of three interrelated powerful methods for the extraction of mechanical properties from thin films under weakly tensile to strongly compressive residual stress. Mechanical properties such as residual stress and strain, Young's modulus, Poisson's ratio, and the coefficient of thermal expansion of CMOS thin films were measured. Thin film stresses and plane strain moduli were extracted from the load-deflection of long micromachined thin film membranes under plane strain deformations. Poisson's ratio was determined from the ripple transition, a mechanical instability transition of long, pressurized and compressively prestressed thin film membranes. Thin film strains and the coefficient of thermal expansion were extracted from the temperature dependent post-buckling deflection of square membranes.

Analytical and numerical models for the load-deflection and buckling behavior of such micromachined structures were developed. An analytical plate model for the plane strain deflection of long micromachined membranes was established. The latter model takes into account the flexural rigidity of the structures and accurately describes their mechanical response to uniform differential pressure in the range from strongly tensile to strongly compressive residual stress. Plane strain buckling is modeled accurately. The ripple transition of long membranes was numerically investigated using the Ritz method. This instability phenomenon was analyzed in-depth as a function of geometry and mechanical properties. A procedure to extract Poisson's ratio from experimentally determined critical points is formulated and discussed. The post-buckling of square micromachined membranes was numerically investigated using nonlinear finite element simulations and the Ritz method. Two residual strain regimes were identified in which the buckling profiles show different symmetries. Buckling profiles were calculated.
for a wide range of residual strains and were compared with experimental profiles. Experimental results correlated excellently with the models. The numerical results were condensed into an easy-to-use formula to compute the residual thin film strain from measured center deflections and geometries of buckled square membranes.

Three different PECVD silicon nitride thin films were characterized. These films were deposited on silicon substrates, with contributions of 50%, 55%, and 60% low frequency (LF) power to the total deposition power. Membranes were fabricated using silicon bulk micromachining. The plane strain modulus of these films is between 134 GPa and 142 GPa and increases slightly with increasing percentage of LF power. The stress changes from slightly tensile (25 MPa) at 50% LF power, to compressive stress (-63.2 MPa) at 60% LF power. Poisson's ratio of the nitride with 60% LF power is 0.254±0.022. The coefficient of thermal expansion of the nitride deposited with 55% LF power was found to be \( \alpha_{\text{SiN}}(T) = \alpha_0 + \alpha_1 (T-T_0) \), with \( T_0 = 25^\circ\text{C} \), \( \alpha_0 = (1.803\pm0.006)\times10^{-6} \text{K}^{-1} \) and \( \alpha_1 = (7.5\pm0.5)\times10^{-9} \text{K}^{-2} \).
Die Entwicklung und Herstellung mikromechanischer Sensoren und Aktuatoren mittels IC-Technologie ist undenkbar ohne die Kenntnis der mechanische Materialparameter der verwendeten dünnen Schichten. Da jedoch die Dicke dieser Schichten etwa ein Mikrometer beträgt, können sie nicht ohne weiteres mit den üblichen Messmethoden charakterisiert werden.


Drei verschiedene PECVD Siliziumnitride wurden charakterisiert. Diese Schichten wurden mit Anteilen von 50%, 55%, bzw. 60% niederfrequenter (LF) Energie zur gesamten Depositionsenergie abgeschieden. Mikromechanische Platten wurden mittels Siliziumstrukturierung hergestellt. Das effektive Elastizitätsmodul für ebene Verzerrungszustände dieser Schichten liegt zwischen 134 GPa und 142 GPa und nimmt leicht mit dem Anteil an LF Depositionsenergie zu. Die intrinsische Spannung beträgt 25 MPa bei 50% LF Energie und hängt stark vom Anteil der LF Energie ab. Bei 60% LF Energie hat die Nitritdschicht eine intrinsische Druckspannung von -63.2 MPa. Die Querkontraktionszahl beträgt 0.254±0.022 bei 60% LF Energie. Der thermische Ausdehnungskoeffizient des Nitrides, welches mit 55% LF Energie abgeschieden wurde, ist \( \alpha_{SiN}(T) = \alpha_0 + \alpha_1 (T-T_0) \) mit \( T_0 = 25^\circ C, \alpha_0 = (1.803\pm0.006)\times10^{-6} K^{-1} \) und \( \alpha_1 = (7.5\pm0.5)\times10^{-9} K^{-2} \).
1 INTRODUCTION

1.1 Integrated Micro Electro Mechanical Systems

The progress of silicon integrated circuit (IC) technology has enabled the reliable and cost-effective batch fabrication of highly complex ICs with structures in the submicrometer range. In the seventies, it was demonstrated that silicon wafer material can also be used to produce µm-sized mechanical components [1,2]. The successful combination of electrical devices with mechanical microstructures has led to the rapidly growing field of micro electro mechanical systems (MEMS).

An elegant and cost-efficient approach to the fabrication of MEMS is the application of established IC processes such as complementary metal oxide semiconductor (CMOS) technology [3-5]. This approach, however, limits the range of available materials and the freedom of the process sequence. On the other hand, it allows the integration of transducers and circuitry on the same chip. This results in integrated micro electro mechanical systems (IMEMS) with improved performance and decreased system size. Today, a broad variety of CMOS integrated microsensors and microactuators have been demonstrated. Among these are angular rate sensors [6], gas flow sensors [7], radiation sensors [8], pressure sensors [9], micromirror arrays [10], chemical sensors [11-12], and force sensors [13].

Numerous IMEMS comprise mechanical sensing or actuation components. Typically, such mechanical components are thin film plate and beam structures, fabricated using silicon bulk micromachining or surface micromachining [7-11,13]. The mechanical behavior of these structures is determined, apart from their geometry, by the mechanical properties of the thin films involved. For example, elastic properties, such as Young's modulus and Poisson's ratio, and residual film stresses determine the static and dynamic mechanical behavior of the structures [1]. In addition, the thermomechanical behavior is influenced by the coefficients of thermal expansion of the materials [14]. Thus, knowledge of these mechanical properties is essential for the design, development, and optimization of IMEMS [15,16].
It is well known that thin films materials used in IC technology can have properties different to their bulk counterparts [17]. Moreover, in many cases the materials have a microscopic structure that cannot be obtained in bulk form. For these reasons, thin film properties cannot be inferred from bulk data. This makes the measurement of mechanical thin film properties a necessity. However, due to the small thicknesses of these films, standard mechanical testing techniques are not easily applied and new measurement methods had to be developed. In the following section some of these methods are summarized.

### 1.2 State of the Art of Mechanical Thin Film Characterization

Several methods to determine mechanical thin film properties are available. Among these are the:

- wafer curvature technique,
- beam buckling technique,
- membrane load-deflection method,
- microtensile test,
- nanoindentation,
- resonant frequency analysis,
- pull-in voltage measurement.

In the **wafer curvature technique**, thin film stresses are measured using the substrate curvature caused by the film stress [18]. For a sufficiently thin film, the substrate curvature is independent of the elastic properties of the film. This leads to a simple stress-curvature relationship, known as the Stoney formula [18]. The method is fast and easy to apply. However, it provides only average stress values across the substrate. Temperature dependent film stresses have been investigated using heated substrates [19]. Using different substrates, the coefficient of thermal expansion can be determined [20], but the use of substrates other than silicon in IC fabrication lines is often out of the question.

Various microstructures to determine local film strains were developed [21-24]. The **beam buckling technique** uses the fact that beams undergo Euler buckling, under a well known Euler stress, depending on the beam size [24]. By fabricating arrays of beams with different lengths, the critical beam length at which the
beams start to buckle is determined. This enables the film strain to be obtained. The method is very sensitive to compressive film stresses. However, it has the disadvantage that a large number of differently sized beams is necessary to obtain high measurement accuracy. Recently, Zou et al. determined film strains using the postbuckling deflection of doubly clamped beams [25].

One of the first methods to measure elastic thin film properties was the membrane load-deflection method, also referred to as bulge test [26,27]. A circumferentially supported thin film membrane is loaded by a differential pressure while the resulting deflection is detected. Using a mechanical model, the residual stress and a combination of Young’s modulus and Poisson’s ratio can be extracted from the linear and nonlinear membrane responses, respectively. Since the nonlinear response varies with the fourth power of the lateral membrane size, precise sample fabrication and accurate size determination are crucial. The bulge test is only sensitive to mechanical properties in the plane of the film. This makes the interpretation of the experimental data more straightforward than in other methods such as nanoindentation. A further advantage is that precise bulge test samples can be fabricated by selective removal of the silicon substrate underneath the film using silicon micromachining techniques [28]. This avoids the delicate handling of free-standing thin films. Thus, even films with submicrometer thicknesses can be characterized.

The microtensile test is a miniaturized version of the conventional tensile test. The main advantage of this method is that the stress state of the sample is nearly uniaxial, making the interpretation of the experimental data straightforward [29]. However, sample fabrication, handling, and alignment in the testing apparatus are critical [30]. If the sample is not properly aligned, the applied force is not uniformly distributed over the film and film wrinkling may occur. These problems are partly solved by new sample designs and preparation techniques based on silicon micromachining [29-32]. Recently, Poisson’s ratio of a thin film was determined by measuring the lateral contraction of the sample during microtensile testing [33].

Nanoindentation is a thin film characterization technique similar to the hardness tests commonly used for bulk material [34-36]. In this method, a diamond indenter is pressed into the film. The indentation depth as a function of the indenter load is recorded. Plastic and elastic deformations occur during the load-
ing step. For this reason, the elastic properties are extracted from the elastic unloading curve of the indentation process. The method is fast and sample preparation is simple. The thin film has neither to be removed from the substrate nor to be structured. Sample areas of a few square micrometers are sufficient. However, the stress state underneath the indenter tip is highly nonuniform. Thus, the elastic response of the film during unloading is a complicated function of the possibly anisotropic material properties of the sample. Moreover, depending on indentation depth and elastic properties of substrate and thin film, the substrate also contributes to the elastic response curve [37]. This makes the extraction of reliable elastic coefficients of thin films difficult [34].

A further approach to measure Young’s modulus and the residual stress of thin films is based on the resonant frequency analysis of beam or plate structures [38-43]. The structures are excited into vibration. Variation of the excitation frequency enables the resonant frequencies to be determined. The accuracy of the extracted properties can be improved by including several resonant modes into the analysis [39,43]. For the evaluation of the mechanical properties, the mass density of the material in question has to be measured independently. The determined Young’s modulus depends strongly on sample geometry. Therefore the geometrical characterization of the sample is crucial.

Recently, surface micromachined structures were developed for wafer level measurement and process monitoring of mechanical properties [25,44-47]. These structures are bridges, cantilevers, or plates separated from the substrate by a small gap. Application of a voltage between structure and substrate generates an electrostatic force pulling the structure to the substrate. At a critical voltage, known as the pull-in voltage, the structure collapses. The material properties are extracted from such pull-in voltage measurements, the dimensions of the structure, and the size of the air gap. The advantages of this technique are that the force is applied electrically and that the pull-in voltage can be measured by detecting the shortage between structure and substrate upon collapse. However, the extracted mechanical properties depend strongly on the gap size and structure dimensions, which may cause large errors [44].
1.3 Scope and Organization of this Thesis

This thesis focuses on the determination of mechanical and thermomechanical material properties of CMOS thin films under weakly tensile and compressive residual stress. Properties of interest are stress, strain, Young's modulus, Poisson's ratio, and the coefficient of thermal expansion. These properties were extracted from the load-deflection and postbuckling of micromachined membranes. For stress measurements, the wafer curvature method was used in addition to verify the results. The membrane deflection method was chosen because it appeared to have a sufficient number of degrees of freedom – such as sample geometry, dimensions, loading method and temperature – to make the determination of all parameters of interest possible. As the results show, this expectation was well-founded.

Previously, the bulge test was applied to the characterization of sufficiently thin tensile layers. Theoretical models with only very limited range of application were available. For this reason, more general models for the buckling, postbuckling, and load-deflection behavior of micromachined membranes had to be developed.

In Chapter 2, the mechanical properties of interest are defined and the method used to measure them are introduced. Chapter 3 deals with the theory of thin plates. In Chapter 4 an analytical plane strain model for the load-deflection behavior of long clamped plates is developed. Using this model mechanical properties are extracted from micromachined membranes with either significant flexural rigidity or highly compressive stress. In Chapter 5, a mechanical instability transition of long plates is investigated theoretically and experimentally. Using this transition Poisson’s ratio of thin film membranes is determined with high accuracy. Chapter 6 deals with strongly buckled square plates. The postbuckling deflection is investigated using two numerical methods. The results are utilized to measure the residual strain and the coefficient of thermal expansion of thin films with compressive residual stress.
1.4 Major Results

Plane Strain Load-Deflection of Long Membranes

An analytical plane-strain model for the load-deflection of long micromachined membranes clamped to a rigid support was developed. The model takes into account the flexural rigidity of the structures and accurately describes the response to uniform differential pressure over the range of strongly tensile to strongly compressive residual stress. Plane strain buckling is also modeled accurately. Using this model we successfully extracted, for the first time, reliable mechanical properties from the load-deflection behavior of initially buckled membranes.

Ripple Transition of Long Membranes

The ripple transition, which is a mechanical instability transition of compressively stressed long membranes, was investigated numerically and experimentally. It was found to depend strongly on Poisson’s ratio of the membrane material and thus provides a novel measurement principle to determined this mechanical parameter with high accuracy. The critical deflections which determine the instability points were calculated for a wide range of residual stresses and membrane dimensions and for different Poisson’s ratios.
1.4 Major Results

Postbuckling of Square Membranes

The buckling and postbuckling of square micromachined membranes were experimentally investigated and numerically analyzed. Two strain regimes were found in which the postbuckling profiles show distinct symmetries: either mirror and rotational symmetries or only rotational symmetries. The bifurcation points and postbuckling profiles were calculated as a function of residual strain, Poisson’s ratio, and membrane dimensions using two numerical methods. We found excellent agreement between computed and experimental buckling profiles. Based on the theoretical results we developed a novel method to measure thin film residual strain and the coefficient of thermal expansion with high accuracy.

Extracted Material Properties

Mechanical material properties of thin films were extracted from the response of micromachined membranes to uniform pressure and in-plane residual stress. The plane strain modulus of PECVD silicon nitride was found to increase slightly with increasing ratio of low frequency (LF) power to the total deposition power. The residual stress changes from tensile to compressive values when the percentage of LF power is increased from 50% to 60%. Poisson’s ratio of the nitride deposited with 60% LF power is $v = 0.254 \pm 0.022$ and Young’s modulus is $E = 133 \pm 3$ GPa. The coefficient of thermal expansion of the nitride deposited with 60% LF power is $\alpha_{SiN}(T) = \alpha_0 + \alpha_1 (T-T_0)$ where $T_0 = 25^\circ C$, $\alpha_0 = (1.803 \pm 0.006) \times 10^{-6}$ K$^{-1}$ and $\alpha_1 = (7.5 \pm 0.5) \times 10^{-9}$ K$^{-2}$. 
2 MEASUREMENT OF MATERIAL PROPERTIES

In this chapter basic notions and methods are presented. In Section 2.1 the mechanical and thermomechanical properties of interest are introduced. Measurement methods are explained in Section 2.2. Sample fabrication and the mechanical characterization setup are described in Sections 2.3 and 2.4, respectively.

2.1 Mechanical and Thermomechanical Properties

The thin films investigated in this thesis are made of brittle material. Such material shows a reversible strain-stress relationship up to the fracture strength of the material. For stresses below the fracture strength, no plastic deformation is observed. The mechanical behavior of these films is therefore well described by the theory of elasticity, the basic equations of which are summarized in this section. For a more detailed description the reader is referred to Refs. [48] and [49].

Elastic Properties

We consider a body subject to external forces. Due to the forces each point \( r = \{x_1, x_2, x_3\} \) of the body is displaced by a vector \( u(r) = \{u_1(r), u_2(r), u_3(r)\} \). The field \( u(r) \) is termed the displacement field of the body.

Unless \( u(r) \) is constant, the displacement changes the distance between two nearby points \( r \) and \( r+dr \). Before the deformation, their distance \( dl \) is

\[
dl^2 = dx_i dx_i,
\]

(2.1)

After the deformation the new distance is
2 Measurement of Material Properties

\[ dl'^2 = \left( dx_i + \frac{\partial u_i}{\partial x_j} dx_j \right)^2. \quad (2.2) \]

Expanding \( dl' \) with respect to the derivatives \( \partial u_i/\partial x_j \) leads to

\[ dl' = \sqrt{dl'^2 + 2\varepsilon_{ij} dx_i dx_j} = dl + \varepsilon_{ij} dx_i dx_j, \quad (2.3) \]

where \( \varepsilon_{ij} \) are the components of the strain tensor \( \varepsilon \). They are defined by

\[ \varepsilon_{ij} = \frac{1}{2} \left( \frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i} + \frac{\partial u_i}{\partial x_i} \right). \quad (2.4) \]

The diagonal elements of the strain tensor are the normal strains describing the local relative length change of the body in the \( x, y, \) and \( z \) directions. Positive strains describe elongations, while negative strains correspond to compressions. The off-diagonal elements are the shear strains. In most situations, the derivatives \( \partial u_i/\partial x_j \) are much smaller than unity and thus it is sufficient to consider only the first two terms of \( \varepsilon_{ij} \) linear in \( \partial u_i/\partial x_j \). However, as shown in Chapter 3, this is not the case for the plates considered in this thesis.

Under deformation, stresses develop in the interior of the body. These stresses are usually expressed by the stress tensor \( \sigma \) whose components \( \sigma_{ij} \) describe the normal and shear stresses that arise in the body. Hooke’s law states that for sufficiently small displacements strains and stresses are related linearly by

\[ \sigma_{ij} = c_{ijkl} e_{kl}. \quad (2.5) \]

where \( c_{ijkl} \) denotes the components of the stiffness tensor \( c \) of the material. Considering symmetry, it can be shown that a maximum of 21 of the 81 components \( c_{ijkl} \) are independent. Crystal symmetries in real materials further restrict the degree of independence [50]. As an example, monocrystalline silicon with its cubic crystal symmetry has three independent stiffness components. In the case of isotropic materials, \( c \) can be expressed in terms of two elastic parameters. Frequently, Young’s modulus \( E \), and Poisson’s ratio \( \nu \), are used to parameterize the
components \( c_{ijkl} \) in the isotropic case. Both \( E \) and \( v \) have a straightforward physical meaning. An unconstrained bar experiences a relative length change \( \sigma/E \), if the force per unit area \( \sigma \) pulls on two opposite faces. Simultaneously, the bar contracts laterally and its relative width change is \(-v\sigma/E\). In terms of \( E \) and \( v \) the stress-strain relation Eq. 2.5 explicitly reads

\[
\sigma_{ii} = \frac{E}{(1 + v)(1 - 2v)}(\nu\varepsilon_{kk} + (1 - 2v)\varepsilon_{ii})
\]

for the normal stresses and

\[
\sigma_{ij} = \frac{E}{(1 + v)}\varepsilon_{ij}
\]

for the shear stresses.

In some cases, the full, three dimensional equations can be reduced to a lower dimension. The plane stress situation arises when stresses occur only in one plane and all stress components involving the direction perpendicular to this plane vanish. If the coordinate system is chosen so that this plane coincides with the \( x-y \) plane, Eqs. 2.6 and 2.7 read

\[
\sigma_{xx} = \frac{E}{1 - v^2}(\varepsilon_{xx} + \nu\varepsilon_{yy})
\]

\[
\sigma_{yy} = \frac{E}{1 - v^2}(\varepsilon_{yy} + \nu\varepsilon_{xx}).
\]

\[
\sigma_{xy} = \frac{E}{1 + v}\varepsilon_{xy}
\]

A common example of plane stress is a thin film deposited on a flat substrate. The residual biaxial stress \( \sigma_0 \) in the film acts only in a plane parallel to the film surface. The corresponding homogeneous in-plane strain is denoted \( \varepsilon_0 \) and according to Eqs. 2.8 is related to \( \sigma_0 \) by

\[
\sigma_0 = \frac{E}{1 - v}\varepsilon_0.
\]
The factor $E/(1-\nu)$ is termed the biaxial modulus and is denoted in the following as $E_p$. Note that in a plane stress situation, the perpendicular strain does not necessarily vanish. However, as shown in Section 3.1, this strain component does not contribute to the elastic energy of the film and therefore does not affect its mechanical equilibrium.

A second special case is the plane strain situation. It arises when a body is constrained to deform in parallel planes only. Such a situation occurs, for example, in the middle section of a long rectangular plate that is circumferentially clamped to a rigid support as described in Chapter 4. If the long sides of the plate are parallel to the y-axis and the short sides are parallel to the x-axis, strains develop only in the x-z plane. Since no stresses perpendicular to the surface of the plate can develop the plate is also in a state of plane stress. A strain increase $\Delta \varepsilon_{xx}$ in the plate is then associated to a stress $\Delta \sigma_{xx}$ which is, according to Eqs. 2.6 and 2.7, given by

$$\Delta \sigma_{xx} = \frac{E}{1-\nu^2} \Delta \varepsilon_{xx}. \quad (2.10)$$

The factor $E/(1-\nu^2)$ is the so-called plane strain modulus. As a consequence of Eq. 2.8, a stress in the y-direction also develops, which is given by

$$\Delta \sigma_{yy} = \nu \Delta \sigma_{xx}. \quad (2.11)$$

**Thermal Expansion**

A stress-free body at a temperature $T_0$ experiences a deformation even in the absence of external forces, if its temperature is increased by $\Delta T$. This deformation is described by a second rank tensor, the tensor of thermal expansion [51]. It can be shown that for isotropic materials and materials with cubic crystal symmetry the thermal expansion is isotropic and can thus be described by a single parameter, the coefficient of thermal expansion commonly denoted as $\alpha$ [51]. It describes the relative elongation per temperature change of a stress-free body. For instance, a cube of size $l$ expands to the new size $l'$ given by

$$l' = l(1 + \alpha \Delta T). \quad (2.12)$$
2.1 Mechanical and Thermomechanical Properties

In thin film technology a film adheres to a substrate. Since the film is usually a few hundred times thinner than the substrate, it is constrained to expand with the substrate. If the coefficient of thermal expansion \( \alpha_f \) of the thin film differs from that of the substrate, \( \alpha_s \), a temperature change \( \Delta T \) causes both a deformation and an additional stress in the film. To calculate the thermal stress change in a thin film on a rigid substrate, it is helpful to imagine the four step procedure outlined in Fig. 2.1. We start with a film deposited at a temperature \( T_0 \) with residual stress \( \sigma_0 < 0 \) and residual strain \( \varepsilon_0 = E_b^{-1} \sigma_0 \). Subsequently, the film is removed from the substrate and expands by \(-\varepsilon_0\) to its nondeformed state. Since the film is now unconstrained a temperature increase \( \Delta T \) causes the film and the substrate to expand according to their respective coefficients of thermal expansion. Finally, to attach the film again to the substrate in its original position, the film must be shrinked by the amount

\[
\varepsilon_0(T_0 + \Delta T) = \varepsilon_0(T_0) - [\alpha_f(T_0) - \alpha_s(T_0)]\Delta T. \tag{2.13}
\]
Hence, the stress in the film at the final temperature is given by
\[ \sigma_0(T_0 + \Delta T) = E_b(T_0 + \Delta T)\varepsilon_0(T_0 + \Delta T). \]
If terms of second order in \( \Delta T \) are neglected this results in
\[
\sigma_0(T_0 + \Delta T) = \sigma_0(T_0) - E_b(T_0)[\alpha_f(T_0) - \alpha_s(T_0)]\Delta T
+ \frac{\partial E_b}{\partial T}(T_0)\varepsilon_0(T_0) \Delta T
\]
(2.14)

where the third term accounts for the fact that the material may change its stiffness at a higher temperature. From Eq. 2.14 it can be inferred that the coefficient of thermal stress \( \beta_f \mid_{\varepsilon_0} = d\sigma_0/dT \mid_{\varepsilon_0} \) at a constant biaxial strain \( \varepsilon_0 \) is given by
\[
\beta_f \mid_{\varepsilon_0} = \beta_f + \frac{\partial E_b}{\partial T}\varepsilon_0
\]
(2.15)

where \( \beta_f = -E_b\alpha_f \) denotes the coefficient of thermal stress at zero strain.

It is noteworthy that for many materials common in thin film technology the temperature dependence of the elastic constants at room temperature is weak. The factor \( dE_b/dT \) is often of the order of \( E_b\times10^{-4} \text{ K}^{-1} \) [52]. Since \( \alpha \) is usually of the order of \( 10^{-6} \text{ K}^{-1} \), the second term of Eq. 2.15 is small if \( \varepsilon_0 \) is less than \( 10^{-3} \).

Using Eqs. 2.13 and 2.14 the thermal stress change of a thin film with residual strain \( \varepsilon_0 \) can be written as
\[
\frac{d\sigma_0}{dT} = \beta_f \mid_{\varepsilon_0} + \alpha_s E_b.
\]
(2.16)

2.2 Measurement Methods

We now describe methods used in this thesis to measure the material properties defined above. The bulge test was utilized to measure the residual stress and the plane strain modulus. Poisson’s ratio, the residual strain and the coefficient of thermal expansion were extracted from the buckling of square and rectangular...
membranes. The results were compared with stress measurements using the wafer curvature method.

**Bulge Testing**

The bulge test was one of the first methods to measure elastic thin film properties. It was introduced by J. W. Beams in 1959 [26]. In its original form, a thin film is clamped over an orifice such that a circumferentially supported membrane is built. A hydrostatic differential pressure is then applied, causing the membrane to deflect. From the measurement of the center deflection as a function of the applied pressure, the stress-strain relation of the film material is finally extracted. However, in the original technique the handling of free standing films and the fabrication of initially flat membranes is difficult.

The development of micromachining techniques, in particular the anisotropic etching of silicon, has greatly simplified the fabrication of samples with accurately controlled dimensions for bulge testing. Fabrication techniques are explained in Section 2.3. Using these techniques, thin film membranes are fabricated without ever removing the film from the substrate. Thus, not only the elastic in-plane properties, but also the residual film stress can be extracted from a bulge test. Fig. 2.2 shows schematically a sample under test.

![Micromachined rectangular membrane under bulge test](image-url)
2 Measurement of Material Properties

During the last two decades a large number of thin film materials have been investigated using the bulge test. Single films and multilayers including silicon [53,54], silicon nitride [15,27,55-64] silicon oxide [15,65], polysilicon [15,56,57,62,66-68], metal [26,28,55,68-73], zinc oxide [59,74], titanium nitride [75], diamond [76-78], silicon carbide [79], and polymer [72,80-84] thin films have been characterized. For the parameter evaluation in these investigations, models based on variational approximations and finite element analysis have been used.

For tensile samples \((\sigma_0 > 0)\) with negligible flexural rigidity, the most reliable model for the load deflection relationship is given by

\[
P(w_0) = c_1 \frac{\sigma_0 h}{a^2} w_0 + c_3(v) \frac{E h}{a^4 (1 - v)} w_0^3,
\]

where \(P\) denotes the differential pressure, \(a\) the membrane width, \(h\) the film thickness and \(w_0\) the center deflection as shown in Fig. 2.2. The coefficients \(c_1\) and \(c_3(v)\) as given by Ref. [85] for the commonly used sample geometries are listed in Table 2.1. They have been determined using finite element simulations and are in good agreement with analytical solutions [86]. If the size and thickness of the membrane is known, the stress and a combination of Young's modulus and Poisson's ratio of the film material can be extracted from the linear and cubic responses of the membrane to the pressure load. Since the \(v\)-dependence of the coefficient \(c_3\) changes with geometry, it is possible to determine Poisson's ratio of a film by using different sample geometries [15,27,61,67]. However, the extracted \(v\) values often show a large uncertainty. This is due to the weak dependence of \(c_3(v)\) on geometry.

Bulge test samples frequently have lateral dimensions of the order of 1 mm at a film thickness of the order of 1 \(\mu\)m. Thus, the samples are only able to bear very small compressive in-plane stresses. At higher compressive stresses, membrane buckling (see next subsection) makes the load deflection data of such samples difficult to interpret. Large uncertainties in the deduced material properties may arise if a model of the form of Eq. 2.17 is used [87]. Unfortunately, up to now no quantitative models are available that fully account for the buckling effect in compressive stressed samples. Similar difficulties arise with initially flat samples.
2.2 Measurement Methods

Table 2.1 Coefficients $c_1$ and $c_3(v)$ of load deflection model Eq. 2.17

<table>
<thead>
<tr>
<th>Geometry</th>
<th>$c_1$</th>
<th>$c_3(v)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Square</td>
<td>13.64</td>
<td>31.70-9.360v</td>
</tr>
<tr>
<td>Long, rectangular</td>
<td>8</td>
<td>$64 / 3(1+v)$</td>
</tr>
</tbody>
</table>

whose load deflection is significantly affected by the flexural rigidity of the film, since Eq. 2.17 does not take into account bending stresses. This situation arises at residual stresses close to the critical buckling stress. Attempts were made to correct Eq. 2.17 to include these buckling or bending effects [87,88]. However, these solutions are not generally valid.

Buckling

Buckling translates small in-plane strains into comparatively large displacements and thus provides a useful principle to measure thin film strains [23,24,89-95]. As a mechanical instability phenomenon, buckling results from the competition between two effects. The first is the tendency of a thin flat structure under in-plane compressive stress to release the stress by an out-of-plane displacement. This forces the structure to bend. The second effect is the restoring force due to the flexural rigidity of the structure which tends to drive it back to its flat position.

Another interpretation of buckling is that of symmetry breaking. A weakly pre-strained structure is in equilibrium in the most symmetric position. A membrane for instance rests in the plane, i.e., in a position symmetric with respect to reflection at its plane. As the prestress is increased to sufficiently compressive values, this reflection symmetry is broken in a way reminiscent of second order phase transitions, as described, for example, by the Landau-Ginzburg model [96].

For perfect structures, i.e., structures whose symmetries are not disturbed by material inhomogeneities or geometrical imperfections, the onset of the buckling occurs at a sharply defined compressive critical strain $\varepsilon_{cr} < 0$. In the case of doubly supported beams or circumferentially supported plates, the center deflection increases as $w_0 \sim (\varepsilon_{cr} - \varepsilon_0)^{1/2}$ for prestrains $\varepsilon_0 \leq \varepsilon_{cr}$ close to $\varepsilon_{cr}$ [64,89,97]. However, in real structures small imperfections can lead to considerable smoothing of the buckling transition. In the case of beams and plates, such smoothed
transitions have been termed quasibuckling. However, with increasingly negative strain the influence of these imperfections decreases strongly. Thus, sufficiently far away from the transition the structure behavior can be well approximated by assuming an ideal structure [98,99].

As is shown in Chapter 3, the buckling of single layer microstructures caused by their residual strain does not depend on Young’s modulus. It is only a function of the structure size, the residual strain and Poisson’s ratio. In most cases the influence of $v$ is weak. Thus, structural buckling has been widely used to detect and measure thin film strains. In MEMS, micromachined beams undergoing Euler buckling have been successfully used for this purpose. Residual strains of polysilicon [24,90] and silicon oxide [91] have been measured. Structures with more complex buckling have been used to determine residual strains of diamond-like carbon [92], silicon oxide [23], and polysilicon thin films [93]. Tensile materials have been characterized by fabricating ring structures translating the overall tensile stress into the compression of a central beam [94,95].

In this thesis, the residual strain $\varepsilon_0$ is extracted from the center deflection $w_0$ of buckled membranes. The relationship between buckling height and residual strain is established in Chapter 6 for square membranes. A measurement of $w_0$ versus temperature similar to the $u_0$ versus $P$ measurement in the bulge test enables $\varepsilon_0(T)$ to be determined. According to Eq. 2.13 the derivative of $\varepsilon_0$ with respect to $T$ is directly related to the coefficient of thermal expansion of the film, that is,

$$\frac{d\varepsilon_0}{dT} = \alpha_f - \alpha_s. \tag{2.18}$$

To extract the coefficient of thermal expansion using Eq. 2.18, the coefficient of thermal expansion of the substrate must be known. In this thesis, silicon wafers served as the substrate. The coefficient of thermal expansion of single-crystal silicon has been investigated by many researchers. Fig. 2.3 shows reliable experimental data for temperatures between -24°C and 227°C [100-104]. These data are well represented by the following third order polynomial fit
Fig. 2.3 Coefficient of thermal expansion of single-crystal silicon measured by Roberts [100], Norton [101], Bergamin et al. [102], Okaji [103], Lyon et al. [104]. The solid line shows the smoothed data given by Eq. 2.19.

\[
\alpha_{Si}(T) = (2.364 \times 10^{-6} + 1.001 \times 10^{-8}T - 2.809 \times 10^{-11}T^2 + 3.626 \times 10^{-14}T^3) \text{°C}^{-1},
\]  

(2.19)

where \(T\) is in °C.

**Wafer Curvature**

A common technique to measure thin film stresses is the wafer curvature method [18]. It exploits the fact that stressed films cause their substrate to bend. In this method the substrate curvature is measured before and after film deposition. The thin film stress \(\sigma_0\) is related to the curvature difference \(\Delta \kappa\) by Stoney’s formula [105]

\[
\sigma_0 = \Delta \kappa \frac{E_{b.s} h_s^2}{6h_f}.
\]

(2.20)
where the indices "s" and "f" refer to the substrate and the thin film, respectively, and \( h_s \) and \( h_f \) denote the thicknesses. Eq. 2.20 only holds if \( h_s \gg h_f \) which is the case for most IC-layers with film thicknesses of the order of 1 \( \mu \text{m} \) and substrate thicknesses around 500 \( \mu \text{m} \). For (100) silicon wafers the biaxial modulus is constant in the substrate plane and is 180.5 GPa [34,106].

Usually, the substrate curvatures are measured in terms of the radii of curvature. If \( R_1 \) and \( R_2 \) denote the radii before and after film deposition, respectively, the curvature difference is given by

\[
\Delta \kappa = \frac{1}{R_2} - \frac{1}{R_1}.
\]

If the stress-induced curvature change is measured as a function of temperature, and \( \alpha_s \) as well as \( E_{b,f} \) is known, the coefficient of thermal stress of the thin film can be determined. According to Eqs. 2.16 and 2.20 the coefficient of thermal stress at the residual strain present in the film is given by

\[
\beta_f \mid_{e_0} = \frac{h_s^2}{6h_f} \frac{d}{dT}(\Delta \kappa E_{b,s}) - \alpha_s E_{b,f}.
\]

In most situations the temperature dependence of the biaxial moduli of film and substrate is negligible and Eq. 2.22 simplifies to

\[
\alpha_f = -\frac{h_s^2}{6h_f E_{b,f}} \frac{d}{dT} \Delta \kappa + \alpha_s,
\]

allowing the coefficient of thermal expansion to be determined from a measurement of substrate curvature versus temperature.

To measure the thermal expansion without knowledge of \( E_{b,f} \), the film can be deposited on two substrates with different coefficients of thermal expansion. The two resulting equations of the form of Eq. 2.23 can than be solved for both the coefficient of thermal expansion and the biaxial modulus of the film. However, the use of substrates other than silicon in a semiconductor fabrication line is often out of the question.
2.3 Membrane Fabrication

Four types of samples for bulge testing and buckling measurements were fabricated. Substrate preparation and film deposition were performed by EM Microelectronic-Marin SA, Marin, Switzerland. All further fabrication steps were carried out at the Physical Electronics Laboratory. Table 2.2 lists the sample types and their characteristics. The samples were fabricated as follows: 675 μm thick single-polished 6 in. 10-20 Ωcm p-Si wafers were used as substrate material. Roughly 75 μm of silicon were removed from the wafer rear by chemical polishing. This eliminated superficial defects and ensured reliable rear mask adhesion. Silicon nitride layers were deposited from SiH₄ and NH₃ on the wafer front using a plasma deposition system, Concept One, from Novellus. Three different layer types were deposited. For each layer type the residual stress σ₀ was adjusted to the desired level by appropriate choice of the low-frequency power contributions to the total deposition power [107]. Residual stresses of several wafers of each system were determined from the wafer curvature. Results are shown in Table 2.2. Film thicknesses of each wafer were determined with a Leica MPV-SP spectrometer. The thickness of the thicker films (type A and B) was measured at three locations. The thickness of the other films (type C and D) was determined at five locations. Table 2.2 lists the averages and their standard deviations.

Table 2.2 Characteristics of PECVD silicon nitride films for the different sample types. The stress given in column 4 was routinely measured at EM Microelectronic-Marin SA, Marin, Switzerland and has to be considered as an approximative value.

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>Ratio of LF-power</th>
<th>Film Thickness [μm]</th>
<th>Stress [MPa]</th>
<th>Reflection Layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>50 %</td>
<td>3.549±0.015</td>
<td>26</td>
<td>15 nm Ti</td>
</tr>
<tr>
<td>B</td>
<td>55 %</td>
<td>3.531±0.008</td>
<td>-13</td>
<td>15 nm Ti</td>
</tr>
<tr>
<td>C</td>
<td>60 %</td>
<td>0.703±0.003</td>
<td>-75</td>
<td>no</td>
</tr>
<tr>
<td>D</td>
<td>60 %</td>
<td>1.008±0.003</td>
<td>-75</td>
<td>3 nm Au</td>
</tr>
</tbody>
</table>
A silicon nitride mask was PECVD deposited on the wafer back. After photolithography, it was structured using SF$_6$-based reactive ion etching. The wafers were anisotropically etched in 6 M KOH at 95 °C, with etch stop on the front nitride. For enhanced reflection very thin Ti- or Au-films were sputtered onto the silicon nitride films. In view of the small thicknesses of these layers (see Table 2.2), they were assumed to have the same mechanical properties as the respective nitride layers. The overall thicknesses of the sample layers were therefore the sum of nitride and metal film thicknesses. The error introduced by this simplification is discussed in Chapters 4 and 6. For temperature dependent buckling experiments a structured wafer was cut into small chips which fit into the temperature chamber described in the next section.

2.4 Experimental Setup

Bulge Test

Fig. 2.4 schematically shows the experimental setup for bulge testing. The wafer containing the membranes to be tested is attached onto the holder using a double face adhesive tape from Scotch®. The holder has 2×4 openings in its center with a pitch of 6 mm to supply the differential pressure to different membranes without need to position the wafer anew. Each of these openings can be closed and opened independently. The differential pressure was applied using a differential pressure controller DPI 520 from Druck Limited. Two models with ranges of -90 to 400 kPa and -99 to 99 kPa of differential pressure were used with respective accuracies of ±40 Pa and ±10 Pa in the experimentally used pressure range. A bottle of liquid air and a vacuum pump, to which a 10 liter reservoir was connected, served as the pressure source. The reservoir allowed the vacuum pump to be shut down during measurements to minimize vibrations.

The deflection of the membranes was measured using a scanning and autofocusing optical profilometer from UBM. Two measurement heads were used: the Microfocus® for bulge testing at room temperature and the Telefocus® with a larger working distance for temperature dependent measurements. The instrument lateral resolution is limited to 1 μm by the spot diameter of the laser beam. The vertical resolution is about 10 nm. The instrument was calibrated before measurement using a calibration standard from Carl Zeiss Jena GmbH containing
2.4 Experimental Setup

Fig. 2.4 Schematic of bulge test setup.

seven steps of different high. The absolute accuracy for vertical distances of the order of 10 μm was found to be 0.15 μm. The membranes were aligned to the axes of the profilometer using a rotary table mounted to the x-y table of the profilometer. The setup is controlled using a PC. A LabView program directly controls the DPI 520 and communicates via Digital Data Exchange provided by Windows 95 with the profiler software to synchronize the action of both subsystems.

Before bulge testing, each membrane was optically checked under a microscope to determine whether its edges were straight and parallel. Only those with apparently faultless geometry were then characterized. After mounting on the holder the impermeability of the seals was checked. Then the membrane was aligned to the axes of the profilometer within 0.2° and the width of the membrane was determined using the profilometer at three different locations. Subsequently, the offset of the differential pressure sensor at atmospheric pressure was measured and if necessary adjusted. Next, pressure was applied to the sample and increased in discrete steps. At each stabilized pressure level a deflection profile was recorded. This provided the deflection shape and the center deflection w0 as a function of load.
2 Measurement of Material Properties

In the case of temperature dependent measurements the wafer holder shown in Fig. 2.4 was exchanged with the temperature chamber described in the following subsection.

**Temperature Chamber**

For a temperature dependent buckling measurement, as described in Section 6.5, a silicon chip containing a membrane was thermalized in a small temperature chamber specially developed for this purpose. The temperature chamber is capable of controlling the chip temperature between room temperature and 180 °C. A schematic cross-section is shown in Fig. 2.5. The heated part of the temperature chamber is made of copper, ensuring a homogeneous temperature distribution. Two Pt-100 temperature sensors measure the temperature close to the membrane and heater, respectively. The copper block is heated using a 0.18 mm thin drilled Isotan wire which is housed in a glass spiral. The glass spiral is located in the bottom part of the cylindrical copper block. The wire resistor is powered and controlled by an LTC-11 Temperature Controller from Neocera, which also receives the signals of the temperature sensors. Data are collected by a LabView program. For better control performance the copper block is simultaneously cooled by compressed air that flows through several cavities in the copper block close to the heater. During heating the chamber can be purged with dry nitrogen to avoid oxidation of the sample. A small hole in the center of the chamber allows the membrane to be pressurized.

A double-pane glass window made of BK-7 glass from Schott, located above the membrane, enables the measurement of the membranes buckling profile at a stabilized temperature level. The profiles were recorded using the laser profilometer from UBM with a Telefocus® measurement head. Its optics are corrected for the aberration of the window. To minimize drift and noise in the measured profiles caused by the thermal expansion of the temperature chamber, the copper block is mounted on a quartz cylinder with a small coefficient of thermal expansion. The cylinder is polished at both its ends. Like the copper block, it rests on three steel balls to avoid slight tilting caused by the lateral expansion of the material in combination with the roughness of the supports. The vertical position of the chip in the copper block with respect to the support of the copper block was chosen so that the thermal expansion of the copper block compensates for the expansion of the cylinder. Thus, the position of the sample remains constant during heating. To
2.4 Experimental Setup

Fig. 2.5 Schematic cross-section of temperature chamber for micromachined membranes used for the temperature dependent buckling measurements. The sample chamber diameter is 21 mm and the height is 2 mm.

calculate the optimal chip position it was assumed that the temperature in the quartz cylinder changes linearly from chip temperature at the upper end, to room temperature at the bottom. The copper block and the quartz cylinder are isolated with glass wool, mounted on an aluminum plate and covered by a stainless steel sheet.

To avoid heating of the temperature sensitive measurement head during measurement a water cooled brass ring is attached to the lower end of the measurement head. This ring also serves as support for a thin circular aluminum plate, as shown in Fig. 2.5. This plate prevents air turbulence between the temperature chamber and measurement head at elevated chamber temperatures and remarkably improved the quality of the recorded profiles.
2 Measurement of Material Properties

Wafer Curvature Measurement

Wafer curvature measurements at EM Microelectronic-Marin SA, Marin, Switzerland were performed using a Tencor P2. The film stress was calculated using a wafer thickness of 675 μm for each wafer.

The wafer curvature measurements at the Physical Electronics Laboratory were performed using the thin film stress measurement system Tencor FLX-2320. It measures the wafer curvature optically by detecting the reflection angle of a narrow laser beam shining on the wafer. The curvature measurement uncertainty is stated to be 10^{-4} \text{ m}^{-1} [108]. The wafers were preprocessed at EM Microelectronic-Marin SA, Marin, Switzerland, including both the front side nitride and the mask nitride on the wafer back. The latter was removed before curvature measurement using SF₆-based reactive ion etching. During etching the front side nitride was protected by photoresist. After measurement, the front nitride was also etched and the curvature again determined. The substrate thickness was measured by SEZ AG, Austria, at nine locations across the wafer with an uncertainty of about 1 μm. The average thickness was used as input for Eq. 2.18 to calculate the film stress.
This chapter is a brief introduction to the theory of thin plates with large deflections. These are deflections much larger than the thickness of the plate but still small compared to its lateral dimensions. Hence, the slope of the deflection profile is significantly smaller than one.

The fundamental assumptions of thin plate theory that lead to very satisfactory approximations of the deflection of thin plates under lateral and transverse loads are the following [109,110]:

- Points on the plate lying initially on a line perpendicular to the middle plane remain on the line perpendicular to the middle plane after deflection.
- All stresses corresponding to the direction perpendicular to the plate can be neglected. In other words, a plane stress situation is assumed.

The latter assumption is based on the fact that in-plane stresses caused by a deflection of the plate are generally much larger than surface loads such as a hydrostatic pressure. The plates considered in this thesis are thin enough for these components be small also in the interior of the plates. From the first assumption it follows that stress varies linearly across the thickness of the plate. This variation is due to bending of the plate. The fundamental difference between thin plate theory and the theory of membranes, i.e., of extremely thin plates, is that the theory of membranes does not take into account the flexural rigidity. The stresses are therefore considered to be constant throughout the thickness of the plate.

It should be noted that the MEMS community uses the term "membrane" for the type of micromachined plate structures discussed in this thesis. This term is even applied for structures with significant flexural rigidity. Therefore, if we refer to these objects as microstructures, we will use the term "membrane", irrespective of their flexural rigidity.

In Section 3.1, the energy equations for loaded and prestressed thin plates are derived and the equilibrium conditions formulated. These equations are rewritten in terms of dimensionless parameters in Section 3.2. For a detailed treatment of
3 Thin Plate Theory

thin plate theory the reader may refer to standard textbooks, such as Refs. [110] and [111].

3.1 Energy and Equilibrium Equations

In the following, we consider a rectangular plate with width \( a \), length \( b \), thickness \( h \), uniform residual stress \( \sigma_0 \), Poisson's ratio \( v \), and Young's modulus \( E \), as shown in Fig. 3.1. The coordinate system is centered on the plate with the edges of the plate parallel to the \( x \)- and \( y \)-axes. The deflection of the plate is described by the three dimensional displacement field \( \{u_x(x,y), u_y(x,y), w(x,y)\} \) of its middle plane. The components \( u_x \) and \( u_y \) are the in-plane displacements and \( w \) denotes the out-of-plane displacement. Before we calculate the elastic energy of the deflected plate as a function of the displacement field, the boundary conditions of micromachined plates are briefly discussed.

Fig. 3.1 Schematic view of a micromachined plate deflected by a pressure \( P \). Only half of the structure is shown. The deflection of the membrane is strongly exaggerated. The deflection profile is described by the displacement \( \{u_x(x,y), u_y(x,y), w(x,y)\} \) of the middle plane of the plate.
3.1 Energy and Equilibrium Equations

Boundary Conditions

The micromachined membranes commonly used in bulge testing have large width to thickness ratios and tensile residual stresses. The load-deflection behavior of such samples is dominated by stretching stresses at the middle plane of the structure [64]. To simplify the load-deflection models, bending effects are usually neglected and simply supported boundary conditions are assumed [27,86].

However, the bulge test and buckling samples investigated in this thesis show significant bending effects at their edges, as shown in Figs. 4.7 and 6.9. The membrane profiles shown in these figures have a vanishing slope at the boundaries. We therefore adopt clamped boundary conditions for the micromachined membranes investigated in this thesis. These are

\[ u_x = u_y = w = \frac{dw}{dn} = 0, \quad (3.1) \]

at the edges of the membrane, where \( \frac{dw}{dn} \) denotes the derivative perpendicular to these edges.

It should be noted that the boundary conditions Eq. 3.1 are an idealization of the real situation, since the supporting substrate is not perfectly rigid. Boman et al. [112] studied the influence of the elasticity of the support on the linear load-deflection behavior of micromachined silicon membranes by means of finite element simulations. The influence of the support increases with decreasing slenderness \( a/h \) of the membranes. For ratios \( a/h \) larger than 300, the center deflection increases by less than 1% if the elasticity of the support is taken into account. The slenderness of the silicon nitride membranes investigated in this thesis are between 107 and 1400. Most of the membranes have a slenderness much larger than 300. Since the stiffness of the silicon nitride is lower than the stiffness of silicon, the influence of the support in the case of the silicon nitride membranes may be even smaller than in the situation studied in Ref. [112]. For a slenderness of 107, Boman et al. found an increase in the center deflection of 3.9% due to the inclusion of the elasticity of the support.
3 Thin Plate Theory

Elastic Energy

According to basic mechanics, the elastic energy $U_{el}$ of a plate is given by [113]

$$ U_{el} = \frac{1}{2} \int_{V} \sigma_{ij} \varepsilon_{ij} dV, \quad (3.2) $$

where the integration runs over the entire volume $V$ of the plate. With use of Hooke's law, Eq. 2.8, the elastic energy reads for the specified geometry

$$ U_{el} = \frac{Eh}{2(1 - \nu^2)} \int_{V} \{ \varepsilon_{xx}^2 + \varepsilon_{yy}^2 + 2\nu \varepsilon_{xx} \varepsilon_{yy} + 2(1 - \nu) \varepsilon_{xy}^2 \} dz dy dx, \quad (3.3) $$

where $z_0$ is the position of the middle plane of the plate.

To perform the $z$-integration it is useful to write the components of the strain tensor as a sum of three contributions, namely the prestrain $\varepsilon_0$, the strain due to the elongation of the middle plane $\varepsilon_{ij}^{(m)}$, and the $z$-dependent bending strain $\varepsilon_{ij}^{(b)}(z)$, i.e.,

$$ \varepsilon_{ij}(z) = \delta_{ij} \varepsilon_0 + \varepsilon_{ij}^{(m)} + \varepsilon_{ij}^{(b)}(z). \quad (3.4) $$

According to Eq. 2.4, $\varepsilon_{ij}^{(m)}$ is given by

$$ \varepsilon_{ij}^{(m)} = \frac{1}{2} \left( \frac{\partial u_i}{\partial j} + \frac{\partial u_j}{\partial i} \right) + \frac{1}{2} \frac{\partial w}{\partial i} \frac{\partial w}{\partial j}, \quad (3.5) $$

with $i, j \in \{x, y\}$. Note that in Eq. 3.5 all terms quadratic in the in-plane displacements are neglected. The bending strain vanishes at the middle plane, increases linearly with the distance to the middle plane and is proportional to the local curvature of the plate. Thus, it is well approximated by [114]

$$ \varepsilon_{ij}^{(b)}(z) = (z - z_0) \frac{\partial}{\partial i} \frac{\partial}{\partial j} w. \quad (3.6) $$

34
Substitution of Eq. 3.4 into Eq. 3.3 and integration over \( z \) results in two separate contributions \( U_0 \) and \( U_b \) to the elastic energy. The first contribution is due only to stretching of the middle plane and is given by

\[
U_0 = \frac{Eh}{2(1 - \nu^2)} \int_{-a/2}^{a/2} \int_{-b/2}^{b/2} \left\{ \varepsilon_{xx}(z_0) + \varepsilon_{yy}(z_0) + 2\nu \varepsilon_{xx}(z_0) \varepsilon_{yy}(z_0) \right\} dydx. \tag{3.7}
\]

The second contribution \( U_b \) is the bending energy and is given by

\[
U_b = \frac{D}{2} \int_{-a/2}^{a/2} \int_{-b/2}^{b/2} \left[ \left( \frac{\partial^2 w}{\partial x^2} + \frac{\partial^2 w}{\partial y^2} \right)^2 - 2(1 - \nu) \left( \frac{\partial^2 w}{\partial x^2} \frac{\partial^2 w}{\partial y^2} - \left( \frac{\partial w}{\partial x} \frac{\partial w}{\partial y} \right)^2 \right) \right] dydx, \tag{3.8}
\]

where \( D \) is the flexural rigidity of the plate defined by

\[
D = \frac{Eh^3}{12(1 - \nu^2)}. \tag{3.9}
\]

According to Eqs. 3.7 and 3.8, the total elastic energy is expressed only in terms of properties of the middle plane, namely deflection and strain. Hence, all quantities such as displacements, strains, and stresses are in the following referred to this plane unless otherwise stated.

To obtain the energy \( U_0 \) explicitly as a function of the displacement field, Eq. 3.5 must be substituted into Eq. 3.7. Since the resulting equation is quite cumbersome, it is not given here but later in this chapter in terms of dimensionless quantities introduced in Section 3.2.

**Equilibrium Equations**

The equilibrium displacement field minimizes the energy of the plate. It can be obtained by the principle of virtual work

\[
\delta U_0 + \delta U_b + \delta U_p = 0. \tag{3.10}
\]
where $\delta U_p = P\delta w$ denotes the virtual pressure work [110]. This condition is equivalent to the following system of partial differential equations [110]

$$D \Delta^2 w + h \frac{\partial}{\partial j} \left( \sigma_{ij} \frac{\partial w}{\partial i} \right) = P$$

$$\frac{\partial}{\partial j} \sigma_{ij} = 0$$

(3.11)

where $i$ and $j$ assume the values $x$ and $y$ and $\Delta$ denotes the two dimensional Laplace operator $\partial^2/\partial x^2 + \partial^2/\partial y^2$. It is obvious that these equations can be solved only in a few ideal situations. In Chapter 4 such a situation is analyzed. However, in most cases one has to rely on numerical methods to find the energy minimum of the plate. Chapters 5 and 6 deal with such cases.

Non-Homogeneous Residual Strain

So far we have assumed a residual strain independent of $z$. However, many thin films exhibit strains that vary throughout their thickness [115,116]. In the following, we show that except for constants, which are irrelevant, the energies $U_p$ and $U_0$ in Eqs. 3.6 and 3.7 are the same even in this more general case. This is a consequence of the boundary conditions Eq. 3.1. In this case, $\varepsilon_0$ has to be replaced in the energy equations with the $z$-averaged residual strain $\varepsilon_{0ave}$.

We consider a thin film with $z$-dependent residual strain

$$\varepsilon_0(z) = \varepsilon_{0ave} + \Delta \varepsilon_0(z),$$

(3.12)

where

$$\frac{h}{2}$$

$$\int_{-h/2}^{h/2} \Delta \varepsilon_0(z) dz = 0.$$  

(3.13)

It is sufficient to show that if the term $\Delta \varepsilon_0(z)$ is added to the strain tensor in Eq. 3.4 this only results in additional energy terms in Eq. 3.7 which are independent of the displacement field. As an example, the first integrand of Eq. 3.3 is
3.2 Reduced Parameters

considered. The other integrands exhibit similar behavior. Using Eq. 3.4, we obtain

$$
\varepsilon_{xx}^2(z) = (\varepsilon_{0,ave} + \Delta \varepsilon_0(z) + \varepsilon_{x}^{(m)} + \varepsilon_{xx}^{(b)}(z))^2.
$$

(3.14)

Collecting all terms of the right hand side containing $\Delta \varepsilon_0(z)$ yields

$$
\varepsilon_{0,ave} \Delta \varepsilon_0(z) + \Delta \varepsilon_0(z)^2 + \Delta \varepsilon_0(z) \varepsilon_{x}^{(m)} + \Delta \varepsilon_0(z) \varepsilon_{xx}^{(b)}(z).
$$

(3.15)

The first and the third term vanish upon integration with respect to $z$. The second term is independent of the displacement field and thus adds only a constant to $U_{el}$. The latter term vanishes upon integration over $x$ since

$$
\int_{-a/2}^{a/2} \Delta \varepsilon_0(z) \varepsilon_{xx}^{(b)}(z) dx = \Delta \varepsilon_0(z)(z - z_0) \left. \frac{\partial w}{\partial x} \right|_{-a/2}^{a/2} = 0.
$$

(3.16)

Note that the second equality in Eq. 3.16 only holds for built-in boundary conditions, which are assumed for the micromachined membranes investigated in this thesis.

3.2 Reduced Parameters

For a numerical analysis of the mechanical behavior a plate it is advantageous to introduce the reduced dimensionless parameters defined in Table 3.1. The energy $U_b$ in terms of the reduced quantities reads

$$
\bar{U}_b = \frac{1}{24} \int_{-b/2}^{b/2} \int_{-1/2}^{1/2} \left[ \left( \frac{\partial^2 \bar{w}}{\partial \bar{x}^2} + \frac{\partial^2 \bar{w}}{\partial \bar{y}^2} \right)^2 + 2(1 - \nu) \left( \frac{\partial^2 \bar{w}}{\partial \bar{x}^2 \partial \bar{y}} - \left( \frac{\partial^2 \bar{w}}{\partial \bar{y} \partial \bar{y}} \right)^2 \right) \right] d\bar{y} d\bar{x}.
$$

(3.17)

Using the reduced strain tensor $\bar{\varepsilon}_{ij}$ at the neutral plane
### Table 3.1 Physical and the corresponding dimensionless parameters of thin plate theory.

<table>
<thead>
<tr>
<th>Physical quantity</th>
<th>Symbol</th>
<th>Reduced parameter</th>
<th>Symbol</th>
</tr>
</thead>
<tbody>
<tr>
<td>x-coordinate</td>
<td>$x$</td>
<td>$x/a$</td>
<td>$\tilde{x}$</td>
</tr>
<tr>
<td>y-coordinate</td>
<td>$y$</td>
<td>$y/a$</td>
<td>$\tilde{y}$</td>
</tr>
<tr>
<td>Pressure</td>
<td>$P$</td>
<td>$\frac{P(1-v^2)(a/h)^4}{E}$</td>
<td>$\tilde{P}$</td>
</tr>
<tr>
<td>In-plane displacement</td>
<td>$u_x$</td>
<td>$u_xa/h^2$</td>
<td>$\tilde{u}$</td>
</tr>
<tr>
<td>In-plane displacement</td>
<td>$u_y$</td>
<td>$u_ya/h^2$</td>
<td>$\tilde{v}$</td>
</tr>
<tr>
<td>Out-of-plane displacement</td>
<td>$w$</td>
<td>$w/h$</td>
<td>$\tilde{w}$</td>
</tr>
<tr>
<td>Energy</td>
<td>$U$</td>
<td>$\frac{U(1-v^2)a^2}{Eh^5}$</td>
<td>$\tilde{U}$</td>
</tr>
<tr>
<td>Strain tensor</td>
<td>$\varepsilon_{ij}$</td>
<td>$\varepsilon_{ij}\left(\frac{a}{h}\right)^2$</td>
<td>$\tilde{\varepsilon}_{ij}$</td>
</tr>
<tr>
<td>Residual strain</td>
<td>$\varepsilon_0$</td>
<td>$\varepsilon_0\left(\frac{a}{h}\right)^2$</td>
<td>$\tilde{\varepsilon}_0$</td>
</tr>
<tr>
<td>Stress tensor</td>
<td>$\sigma_{ij}$</td>
<td>$\sigma_{ij}\left(\frac{a}{h}\right)^2\frac{(1-v^2)}{E}$</td>
<td>$\tilde{\sigma}_{ij}$</td>
</tr>
<tr>
<td>Residual stress</td>
<td>$\sigma_0$</td>
<td>$\sigma_0\left(\frac{a}{h}\right)^2\frac{(1-v^2)}{E}$</td>
<td>$\tilde{\sigma}_0$</td>
</tr>
<tr>
<td>Plane strain stress $\sigma_{xx}$</td>
<td>$s$</td>
<td>$s\left(\frac{a}{h}\right)^2\frac{(1-v^2)}{E}$</td>
<td>$\tilde{s}$</td>
</tr>
</tbody>
</table>
3.2 Reduced Parameters

\[ \varepsilon_{ij} = \frac{1}{2} \left( \frac{\partial \tilde{u}_i}{\partial j} + \frac{\partial \tilde{u}_j}{\partial i} \right) + \frac{1}{2} \frac{\partial \tilde{w}}{\partial j} \frac{\partial \tilde{w}}{\partial i} \] (3.18)

with \( i, j \in \{x, y\} \), we similarly obtain the energy \( \overline{U}_0 \) as an explicit function of the displacement field, namely

\[
\overline{U}_0 = \frac{1}{2} \int_{-b/2}^{b/2} \int_{-1/2}^{1/2} \left[ 2(1 + \nu) \varepsilon_0 \left( \frac{\partial \tilde{u}_x}{\partial x} + \frac{\partial \tilde{u}_y}{\partial y} \right) + \left( \frac{\partial \tilde{u}_x}{\partial x} \right)^2 + \left( \frac{\partial \tilde{u}_y}{\partial y} \right)^2 
\right. \\
+ \frac{1}{2} (1 - \nu) \left[ \left( \frac{\partial \tilde{u}_x}{\partial y} \right)^2 + \left( \frac{\partial \tilde{u}_y}{\partial x} \right)^2 \right] + 2 \nu \frac{\partial \tilde{u}_x}{\partial x} \frac{\partial \tilde{u}_y}{\partial y} + (1 - \nu) \frac{\partial \tilde{u}_x}{\partial x} \frac{\partial \tilde{u}_y}{\partial y} \\
+ \frac{\partial \tilde{u}_x}{\partial x} \left( \frac{\partial \tilde{w}}{\partial x} \right)^2 + \frac{\partial \tilde{u}_y}{\partial y} \left( \frac{\partial \tilde{w}}{\partial y} \right)^2 + \nu \left[ \frac{\partial \tilde{u}_x}{\partial x} \left( \frac{\partial \tilde{w}}{\partial x} \right)^2 + \frac{\partial \tilde{u}_y}{\partial y} \left( \frac{\partial \tilde{w}}{\partial y} \right)^2 \right] \\
+ (1 - \nu) \frac{\partial \tilde{w}}{\partial y} \frac{\partial \tilde{w}}{\partial y} \left( \frac{\partial \tilde{u}_x}{\partial x} + \frac{\partial \tilde{u}_y}{\partial y} \right) + \varepsilon_0 (1 + \nu) \left[ \left( \frac{\partial \tilde{w}}{\partial x} \right)^2 + \left( \frac{\partial \tilde{w}}{\partial y} \right)^2 \right] \\
+ \frac{1}{4} \left( \frac{\partial \tilde{w}}{\partial x} \right)^4 + \frac{1}{4} \left( \frac{\partial \tilde{w}}{\partial y} \right)^4 + \frac{1}{2} \left( \frac{\partial \tilde{w}}{\partial x} \right)^2 \left( \frac{\partial \tilde{w}}{\partial y} \right)^2 \right] d\tilde{y} d\tilde{x}.
\] (3.19)

For plates with the boundary conditions defined in Eq. 3.1, the first term of the integrand can be disregarded since \( \varepsilon_0 \frac{\partial \tilde{u}_x}{\partial \tilde{x}} \) and \( \varepsilon_0 \frac{\partial \tilde{u}_y}{\partial \tilde{x}} \) vanish upon integration and the term containing \( \varepsilon_0^2 \) is independent of the displacement field and only adds a constant.

In view of Eqs. 3.17 and 3.19, the reduced equilibrium deflection \( \tilde{w} \), which can be determined using Eq. 3.10 in the reduced notation, is only a function of three dimensionless parameters: \( \tilde{\varepsilon}_0 \), \( \nu \), and \( \tilde{P} \). In the case of a plane strain situation, as discussed in Chapter 4, the number of independent parameters determining \( \tilde{w} \) reduces to two. In Chapter 4 the plate deflection is discussed as a function of the parameters \( \tilde{\sigma}_0 \) and \( \tilde{P} \).
4 Long Rectangular Plates – Plane Strain Deflection

A plane strain load-deflection model for long plates clamped to a rigid support is developed in this chapter. The analytical model describes the nonlinear deflection of plates with compressive or tensile residual stress and finite flexural rigidity under uniform load. It allows for the extraction of the residual stress and plane strain modulus of single-layered thin films. Properties of compressively and weakly prestressed materials are determined with an accuracy achieved previously only with tensile samples. Two approximations of the exact model are derived. The first reduces the plates to membranes by neglecting their flexural rigidity. Considerable errors result from this simplification. The second approximation provides exact expressions for the linear and cubic plate responses. Using the model, mechanical properties were extracted from three PECVD silicon nitride films with vanishing, weakly tensile, and compressive prestress, respectively.

Analytical solutions of von Kármán’s equation for uniformly loaded plates either deal with membranes, i.e., thin plates of negligible flexural rigidity, or provide implicit expressions. The linear responses of circular [36,55,82,87] rectangular [117], and infinitely long [36] membranes with tensile residual stress are known. The nonlinear response of square and rectangular plates can be calculated with arbitrary precision in terms of the Fourier expansion of the deformation [117]. However, this method requires the solution of a system of cubic equations. Usually only the first Fourier term is retained [15,56,59,82,84]. The linear response of unstressed plates is described by Navier’s solution [118]. An analytical nonlinear theory describes the plane strain response of initially unstressed, infinitely long plates [119].

In less ideal situations, one has to rely on approximations, based on the energy method or numerical simulations. These make it possible to determine residual
stresses and elastic coefficients with respective accuracies of a few MPa and GPa. In many cases this is sufficient. In more delicate tasks, however, such as the extraction of Poisson’s ratio from the load-deflection of differently shaped samples [15,27,61,67], or the extraction of single film properties from multilayers [15,68,83], errors propagate and grow to considerable size. The formulation of other exact models covering a broad range of plate parameters is thus highly desirable.

The equilibrium equation for such structures is formulated in Section 4.1. Its solution is developed in Section 4.2. Approximations are derived and their accuracy is analyzed in Section 4.3. Finally, in Section 4.4, mechanical properties are extracted using the model from PECVD silicon nitride films with low and compressive residual stresses, respectively.

4.1 Analytical Model

A long plate as defined in Section 3.1 is considered. If the aspect ratio \( b:a > 4 \) [27], a middle section of the structure responds with a plane strain deformation, with negligible dependence on the longitudinal coordinate as shown in Fig. 4.1. This part behaves like a section of an infinitely long plate. In view of Eq. 3.11 the plane strain deformation is governed by the out-of-plane equilibrium condition [119]

\[
D \frac{d^4}{dx^4} w(x) - \frac{d}{dx} \left( h s(x) \frac{d}{dx} w(x) \right) = P, \tag{4.1}
\]

where \( s(x) = \sigma_{xx}(x, 0) \). The product \( h s(x) \) is the in-plane distributed force acting on the plate in the \( x \)-direction. In-plane equilibrium causes \( s(x) \) to be independent of \( x \). In the undeflected plate, \( s \) is equal to \( \sigma_0 \). Deflected, the plate experiences a relative length change excellently approximated by [119]

\[
\frac{1}{2a} \int_{-a/2}^{a/2} \left( \frac{dw}{dx} \right)^2 dx. \tag{4.2}
\]
4.1 Analytical Model

Fig. 4.1 Deflection profile of a 7630 μm long, 1370 μm wide PECVD silicon nitride thin film under differential pressure. The vertical axis is not to scale. The influence of the shorter supports relaxes over a distance less than 2a.

The plane strain modulus $E/(1-\nu^2)$ translates this into an additional stress contribution. Consequently $s$ is given by

$$s = \sigma_0 + \frac{E}{1-\nu^2} \frac{1}{2a} \int_{-a/2}^{a/2} \left( \frac{dw}{dx} \right)^2 dx. \quad (4.3)$$

In terms of the reduced parameters defined in Section 3.2, the plate equations read

$$\frac{1}{12d\bar{x}^4} \frac{d^4}{d\bar{x}^4} \bar{w}(\bar{x}) - \frac{1}{2} \frac{d^2}{d\bar{x}^2} \bar{w}(\bar{x}) = \bar{P} \quad (4.4)$$

and

$$\bar{s} = \bar{\sigma}_0 + \frac{1}{2} \int_{-1/2}^{1/2} \left( \frac{d}{d\bar{x}} \bar{w}(\bar{x}) \right)^2 d\bar{x}, \quad (4.5)$$

where $\bar{s} = \bar{\sigma}_{xx}(\bar{x},0)$. Equations 4.1 and 4.3 or equivalently 4.4 and 4.5 bring out the nonlinearity of the situation. The load response of the plate depends on the effective in-plane stress. This, in turn, depends quadratically on the deflection profile. The deflection shape of the plate is therefore expected to fundamentally change as a function of the load.
4 Long Rectangular Plates – Plane Strain Deflection

4.2 Load-Deflection Response

For a given reduced effective stress \( \tilde{s} \), the solution of the out-of-plane equilibrium equation, subject to clamped boundary conditions specified in Eq. 3.1, is [119]

\[
\bar{w}(\bar{x}) = \bar{P} G(\tilde{s}, \bar{x}),
\]

with

\[
G(\tilde{s}, \bar{x}) = \frac{1}{2\tilde{s}} \left( \frac{1}{4} - \bar{x}^2 \right) - \frac{\cosh(\sqrt{3\tilde{s}}) - \cosh(2\sqrt{3\tilde{s}}\bar{x})}{4\sqrt{3\tilde{s}}^{3/2} \sinh(\sqrt{3\tilde{s}})}.
\]

Apart from the vertical scaling factor \( \bar{P} \), Eq. 4.6 describes a one-parameter family of deflection shapes parameterized by the effective stress \( \tilde{s} \). The plate center deflection \( \bar{w}_0 \equiv \bar{w}(0) \) is

\[
\bar{w}_0 = \bar{P} G(\tilde{s}, 0).
\]

Note that \( G \) can be extended into the range of compressive stresses (\( \tilde{s} < 0 \)) using the identities \( \sinh(ik) = i \sin(k) \) and \( \cosh(ik) = \cos(k) \), where \( i = \sqrt{-1} \). The integral in Eq. 4.5 is carried out using the deflection profile \( \bar{w}(\bar{x}) \) of Eqs. 4.6 and 4.7, and \( \bar{P} \) is eliminated using Eq. 4.8. This provides an implicit definition of the effective stress \( \tilde{s} \) in terms of residual stress \( \bar{\sigma}_0 \) and center deflection \( \bar{w}_0 \), i.e.,

\[
\tilde{s} = \bar{\sigma}_0 + \bar{w}_0^2 H(\tilde{s})/2,
\]

where

\[
H(\tilde{s}) = \frac{4((8 + 4\tilde{s}) \sinh(\sqrt{3\tilde{s}})^2 - 6\tilde{s} - 3\sqrt{3\tilde{s}} \sinh(2\sqrt{3\tilde{s}}))}{\left( \sqrt{3\tilde{s}} \sinh(\sqrt{3\tilde{s}}) - 4 \sinh(\sqrt{3\tilde{s}}/2)^3 \right)^2}.
\]

The function \( H(\tilde{s}) \) varies smoothly, with values between 4.88 and 5.34 for \(-\pi^2/3 < \tilde{s} < \infty \). It has the values \( H(-\pi^2/3) = \pi^2/2, \ H(0) = 2^9/105, \) and \( H(\infty) = 16/3 \). The quadratic dependence of \( \tilde{s} \) on the center deflection is weakly
modulated by the deformation profile, through $H(s)$. The effective stress $\tilde{s}$ in Eq. 4.9 is implicitly defined. This is similar to the result of [119] where, however, $\bar{\sigma}_0 = 0$. For given values of $\bar{\sigma}_0$ and $\tilde{w}_0$, the evaluation of $\tilde{s}$ requires the solution $\tilde{s}(\tilde{w}_0, \bar{\sigma}_0)$ of Eq. 4.9, which can be obtained numerically with arbitrary precision. With this, Eq. 4.9 is rewritten as

$$\tilde{P} = \tilde{w}_0/G(\tilde{s}(\tilde{w}_0, \bar{\sigma}_0), 0).$$  \hspace{1cm} (4.11)

This is the load-deflection law of a prestressed long clamped plate. The pressure $P$ required to deflect a plate with prestress $\sigma_0$ to a center deflection $w_0 \equiv w(x = 0)$ is thus obtained by calculating consecutively: the reduced parameters $\tilde{w}_0$ and $\bar{\sigma}_0$, the reduced effective stress $\tilde{s}(\tilde{w}_0, \bar{\sigma}_0)$, the reduced pressure $\tilde{w}_0/G(\tilde{s}(\tilde{w}_0, \bar{\sigma}_0), 0)$, and finally the physical pressure $P$. Algorithm to solve Eqs. 4.9 and 4.11 numerically are proposed in Appendix A.1.

An overview of the load-deflection phenomenon described by Eq. 4.11 is given in Fig. 4.2. For different pressures $\tilde{P}$, $\tilde{w}_0$ is shown as a function of $\bar{\sigma}_0$. The

![Fig. 4.2 Reduced deflection $\tilde{w}_0$ of long plates as a function of reduced residual stress $\bar{\sigma}_0$ and pressure $\tilde{P}$. Unloaded plates undergo Euler buckling at $\bar{\sigma}_0 = -\pi^2/3$. Vertical cuts provide load-deflection responses for given $\bar{\sigma}_0$.](image)

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unloaded case, \( P = 0 \), is particularly noteworthy. A stretched or compressed plate with \( \bar{\sigma}_0 > -\pi^2/3 \) is stable in the flat position. At the critical point \( \bar{\sigma}_{cr} = -\pi^2/3 \) the response curve bifurcates. The plate undergoes Euler buckling [120]. This results from the singularity of \( G \) at \( \bar{s} = -\pi^2/3 \), since \( \sinh(i\pi) = 0 \). The two bifurcation branches \( (\bar{\sigma}_0 < \bar{\sigma}_{cr}) \) describe two stable buckling deflections of the plate: upward and downward. On both buckling branches, the effective reduced stress is \(-\pi^2/3\), irrespective of \( \bar{\sigma}_0 \). Plates with \( \bar{\sigma}_0 < \bar{\sigma}_{cr} \) settle into a buckling profile that relaxes their residual stress to \( \bar{\sigma}_{cr} \). In terms of nonreduced parameters, the critical stress is given by \( \sigma_{cr} = -4\pi^2D/ha^2 \). For comparison, in square plates \( \sigma_{cr} \approx -16\pi^2D/3ha^2 \) [54].

On the buckling branches the center deflection \( \bar{w}_0 \) grows according to

\[
\bar{w}_0 = \pm 2\pi^{-1} (\bar{\sigma}_0 - \pi^2/3)^{1/2} \quad (4.12)
\]

In dimensional notation this is

\[
w_0 = \pm \frac{2h}{\sqrt[3]{3}} \left( \frac{\bar{\sigma}_0}{\sigma_{cr}} - 1 \right)^{1/2}
\quad (4.13)
\]

and the deflection profile is \( w(x) = w_0 \{1 + \cos(2\pi x/a)\}/2 \).

Under a differential pressure \( \bar{P} \neq 0 \) the sharp Euler transition is smoothed, as shown in Fig. 4.2. The same phenomenon has been analyzed in the context of clamped-clamped beams [98] and was termed quasibuckling. A transverse load breaks the symmetry of the structure and regularizes its singular instability transition.

Figures 4.3(a) and (b) show the load-deflection response of plates with various residual stresses \( \bar{\sigma}_0 \). For comparison, the response of plates without flexural rigidity (cf. Section 4.3) is also shown. As the deflection increases and the effective stress becomes strongly tensile, bending becomes less important and the responses converge.

The transition from plate to membrane behavior is shown in Fig. 4.4. Deflection profiles normalized to the center deflection, \( w(x)/w_0 \), are shown for a plate with \( a = 500 \mu m, h = 2 \mu m, \sigma_0 = -6.4 \text{ MPa, and } E/(1-v^2) = 135 \text{ GPa. Despite its nega-} \)
4.2 Load-Deflection Response

Fig. 4.3 Load-deflection (solid lines) of prestressed long plates with different reduced residual stress $\bar{\sigma}_0$: (a) Low residual stress, (b) large residual stress. Corresponding responses of membranes (dashed lines) with identical reduced prestress are obtained by neglecting the bending energy.
Fig. 4.4 Normalized deflection shapes of a long plate with $\bar{\sigma}_0 = -2.97$, loaded to different center deflections $w_0$. For plate parameters and required pressures, see main text. The dashed line shows the membrane deflection shape.

tive residual stress, the unloaded plate is flat, since $\bar{\sigma}_0 = -2.97 > -\pi^2/3$. Deflections of $w_0 = 0.1, 2.5, 5, 10,$ and $20\ \mu m$ require pressures of $0.006, 1.73, 12.9, 99.4,$ and $771$ kPa, respectively. Resulting neutral plane stresses $\sigma_{xx}(x,0)$ are $-6.38, 1.83, 27.2, 132, and 559$ MPa. Corresponding $\tilde{s}$ values are shown in Fig. 4.4. As required by the boundary conditions, the edges of the structure merge horizontally into the supports. Regions of positive curvature near the supports accommodate the plate stiffness. As the load and therefore $\bar{s}$ is increased, the inflection points move towards the edges. The contribution of bending to the total elastic energy becomes increasingly unimportant.

4.3 Approximations

For the extraction of mechanical properties from measured load-deflection curves, explicit load-deflection laws may be preferred over Eq. 4.11, which builds on the implicitly defined effective stress in Eq. 4.9. Two explicit approxi-
4.3 Approximations

Approximations of the exact load-deflection model are proposed and discussed in the following.

**Membrane approximation**

A first approximation neglects the bending term in Eq. 4.1. The deflection profile is then

$$w(x) = \frac{P}{2hs} \left( \frac{a^2}{4} - x^2 \right)$$

(4.14)

and the center deflection is given by $w_0 = \frac{Pa^2}{8hs}$. According to Eq. 4.3,

$$s = \sigma_0 + \frac{E}{(1-v^2)} \frac{8}{3a^2} w_0^2$$

(4.15)

using which the load-deflection law of long membranes [36]

$$P = \frac{8\sigma_0 h}{a^2} w_0 + \frac{64Eh}{3(1-v^2)a^4} w_0^3$$

(4.16)

is obtained. In reduced notation this is

$$\overline{P}_{\text{mem}} = 8\bar{\sigma}_0 \bar{w}_0 + \frac{64}{3} \bar{w}_0^3.$$  

(4.17)

Relative errors $\Delta_{\text{mem}} = (\overline{P}_{\text{plate}} - \overline{P}_{\text{mem}})/\overline{P}_{\text{plate}}$ between approximate and exact pressures $\overline{P}_{\text{mem}}$ and $\overline{P}_{\text{plate}}$ calculated using Eqs. 4.17 and 4.11, are shown in Fig. 4.5(a) as a function of $\bar{\sigma}_0$ and $\bar{w}_0$. At small residual stresses, neglecting bending effects results in large errors. In particular, the membrane model is unable to account for the stability of compressively prestressed plates with $-\pi^2/3 < \bar{\sigma}_0 < 0$. At strongly tensile residual stresses, the membrane model progressively becomes a reliable approximation. Along the $\bar{\sigma}_0$-axis of Fig. 4.5(a), $\Delta_{\text{mem}}$ gives the relative error, with which $\sigma_0$ is determined from experimental data using Eq. 4.17. For $\bar{\sigma}_0 > 8$, $\Delta_{\text{mem}}$ is well approximated by $2/(3\bar{\sigma}_0)^{1/2}$. 

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Fig. 4.5  Relative errors $\Delta_{\text{mem}}$ and $\Delta_{\text{cub}}$ introduced by the (a) membrane approximation and (b) the cubic Taylor approximation of the exact plate model.
4.4 Comparison with Experiment

Cubic approximation

The load deflection law, Eq. 4.11, can be expanded into a Taylor series in terms of \( \bar{w}_0 \) with expansion coefficients \( c_n(\bar{\sigma}_0) \) as given in Appendix A.2. The expansion up to third order in \( \bar{w}_0 \) provides a second, more accurate, cubic approximation of the plate response. In reduced notations, this is

\[
\bar{P}_{cub} = c_1(\bar{\sigma}_0)\bar{w}_0 + c_3(\bar{\sigma}_0)\bar{w}_0^3,
\]

(4.18)

with

\[
c_1(\bar{\sigma}_0) = G(\bar{\sigma}_0, 0)^{-1}
\]

(4.19)

and

\[
c_3(\bar{\sigma}_0) = \frac{1}{2} \frac{H(\bar{\sigma}_0)G'(\bar{\sigma}_0, 0)}{G(\bar{\sigma}_0, 0)^2},
\]

(4.20)

where \( G' \) denotes the partial derivative of \( G \) with respect to \( \bar{s} \). Fig. 4.6 shows the coefficients \( c_1 \) and \( c_3 \) as a function of \( \bar{\sigma}_0 \). The accuracy of this approximation is evaluated by the relative error \( \Delta_{cub} = (\bar{P}_{plate} - \bar{P}_{cub})/\bar{P}_{plate} \) based on reduced pressures calculated using Eqs. 4.18 and 4.11, respectively. Errors are shown in Fig. 4.5(b) for a range of residual stresses and deflections. The approximation is most accurate at small deflections. The linear and cubic responses are modeled correctly over the entire range \( \bar{\sigma}_0 > -\pi^2/3 \). At large tensile residual stresses, a satisfactory approximation is obtained over an increasing range of deflections.

4.4 Comparison with Experiment

The plate model developed in the previous sections is now applied to the characterization of silicon nitride thin films. Measured profiles and numerical fits using Eq. 4.6 are shown in Fig. 4.7. The agreement between experimental data and numerical results is excellent. Note that the deflection profile enters the support with vanishing slope. This is strong evidence for the boundary conditions Eq. 3.1 adopted in this thesis for micromachined membranes.
Stress-Free Thin Films

Thirteen type B structures (see Table 2.2) on the same wafer were characterized. They were located in an area of 35 mm by 42 mm close to the wafer center. Widths $a$ are between 374 μm and 1375 μm. Dimensions of the different structures are listed in Table 4.1.

Load-deflection responses of all samples were measured and fitted with the plate model. Five representative results are shown in Fig. 4.8. Standard deviations between data and fits varied from sample to sample between 0.01 μm and 0.17 μm. The residual stresses $\sigma_0$ and plane strain modulus $E/(1-v^2)$ extracted from each sample are listed in Table 4.1. Four samples were compressively pre-stressed. Their residual stress was, however, too small for Euler buckling to occur. The average residual stress of all samples was $1.3 \pm 3.75$ MPa. The plane strain moduli have a standard deviation of ±3.9 GPa around a mean of 134.4 GPa. When measured repeatedly on the same sample, $\sigma_0$ and $E/(1-v^2)$ were reproduced within ±1 MPa and ±1 GPa, respectively.

As shown in Table 4.1, the reduced residual stress $\bar{\sigma}_0$ of the different samples ranges from -2.23, slightly less compressive than the critical value $-\pi^2/3$, to 6. In this range, the membrane model is unsuited for the description of the experimental results (Section 4.3). Residual stress and plane strain moduli calculated from the experimental data using the membrane model, Eq. 4.16, are shown for com-
4.4 Comparison with Experiment

Fig. 4.7 Measured deflection profiles (dots) of sample No. 1, Table 4.1, under different pressures. The solid lines are fits of the data using Eq. 4.6.

Thus, if bending was ignored, the following erroneous conclusions would be drawn: (i) the residual stress has a large scatter (±18.1 MPa) around a significantly tensile average of 20.1 MPa, (ii) the plane strain modulus is 142.7 GPa with a variance of 5.8 GPa. Undeniably, the plate model provides more consistent results.

Weakly Tensile Thin Film

Fifteen type A samples, as described in Table 2.2, on the same wafer were characterized. The shorter of their lateral dimensions ranged from 370 μm to 1369 μm, as listed in Table 4.2. The aspect ratio was between 3.6 and 7.3. An average plane strain modulus of 136±4.5 GPa was measured. The average stress was found to be 25.5 MPa. However, the standard deviation of ±10 MPa is significantly larger than for type B structures. This is due to the larger area of 84 mm by 60 mm over which the samples are spread. The measured stress values show a clear dependence on the sample location, as shown in Fig. 4.9. A stress gradient
Fig. 4.8 Load-deflection response of long clamped PECVD silicon nitride plates of different widths $a$. The solid lines are fits to the experimental data (markers) using Eqs. 4.8 and 4.9.

of 0.38 MPa/mm was found in a direction inclined at -137° with respect to the wafer flat.

The mechanical parameters evaluated using the membrane model show a similar behavior as the type B structures. The plane strain modulus of 143±5.4 GPa is about 5% higher than that extracted using the plate model and the average residual stress shows a higher standard variation of ±14.2 MPa around an average of 42.7 MPa which is 67% larger than the value obtained with the plate model. Moreover, the stress gradient found using the plate model does not show up as clearly when the membrane model is used.

**Compressive Thin Films**

Extraction of mechanical parameters from buckled samples has been judged unreliable [87]. However, the new plate model provides a sound basis for the analysis of load-deflection data of such structures. Type C samples (see Table 2.2) provide an ideal test system. At a thickness of 0.704 μm, and an
4.4 Comparison with Experiment

Table 4.1 Dimensions and mechanical parameters of almost stress-free thin film samples from the same wafer: width \( a \), length \( b \), residual stress \( \sigma_0 \), plane strain modulus \( E/(1-\nu^2) \), and reduced residual stress \( \bar{\sigma}_0 \) calculated using the plate model, and residual stress \( \sigma_{0\text{mem}} \) and plane strain modulus \( E/(1-\nu^2)_{\text{mem}} \) calculated using the membrane approximation.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>( a \times b ) [( \mu m \times \mu m )]</th>
<th>( \sigma_0 ) [MPa]</th>
<th>( E/(1-\nu^2) ) [GPa]</th>
<th>( \bar{\sigma}_0 )</th>
<th>( \sigma_{0\text{mem}} ) [MPa]</th>
<th>( E/(1-\nu^2)_{\text{mem}} ) [GPa]</th>
</tr>
</thead>
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<td>1</td>
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<td>133.2</td>
<td>5.97</td>
<td>10.9</td>
<td>137.6</td>
</tr>
<tr>
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<td>-2.05</td>
<td>130.0</td>
<td>-2.28</td>
<td>2.78</td>
<td>134.6</td>
</tr>
<tr>
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<tr>
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<td>9.24</td>
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</tr>
<tr>
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<td>132.6</td>
<td>1.34</td>
<td>12.7</td>
<td>140.4</td>
</tr>
<tr>
<td>6</td>
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<td>-3.27</td>
<td>134.4</td>
<td>-1.46</td>
<td>5.73</td>
<td>142.9</td>
</tr>
<tr>
<td>7</td>
<td>879 ( \times ) 4653</td>
<td>-0.96</td>
<td>134.4</td>
<td>-0.43</td>
<td>8.32</td>
<td>142.6</td>
</tr>
<tr>
<td>8</td>
<td>879 ( \times ) 4653</td>
<td>-4.98</td>
<td>135.7</td>
<td>-2.23</td>
<td>3.75</td>
<td>144.3</td>
</tr>
<tr>
<td>9</td>
<td>626 ( \times ) 4155</td>
<td>5.33</td>
<td>134.5</td>
<td>1.23</td>
<td>23.8</td>
<td>143.9</td>
</tr>
<tr>
<td>10</td>
<td>627 ( \times ) 3155</td>
<td>3.39</td>
<td>142.5</td>
<td>0.73</td>
<td>22.5</td>
<td>152.7</td>
</tr>
<tr>
<td>11</td>
<td>378 ( \times ) 1628</td>
<td>-2.98</td>
<td>136.5</td>
<td>-0.25</td>
<td>44.3</td>
<td>147.9</td>
</tr>
<tr>
<td>12</td>
<td>375 ( \times ) 2148</td>
<td>0.94</td>
<td>141.6</td>
<td>0.08</td>
<td>51.6</td>
<td>153.3</td>
</tr>
<tr>
<td>13</td>
<td>374 ( \times ) 1633</td>
<td>5.79</td>
<td>129.5</td>
<td>0.49</td>
<td>53.3</td>
<td>139.8</td>
</tr>
</tbody>
</table>

Average ± standard deviation | 1.30 ± 3.75 | 134.4 ± 3.9 | 20.1 ± 18.1 | 142.7 ± 5.8

Expected residual stress and plane strain modulus of -75 MPa and 135 GPa, respectively, type C membranes with \( a > 54 \mu m \) are expected to buckle \((\bar{\sigma}_0 < -\pi^2/3)\). To be unambiguously in the buckling regime samples with widths between 535 \( \mu m \) and 841 \( \mu m \), and expected \( \bar{\sigma}_0 \) between -320 and -800 were fabricated. In the experiments, positive and negative differential pressures were applied. The side length ratio \( b:a \) of all samples was larger than 10:1.
Table 4.2 Dimensions and mechanical parameters of weakly tensile thin film samples from the same wafer. Notation according to Table 4.1.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>$a \times b$ [μm x μm]</th>
<th>$\sigma_0$ [MPa]</th>
<th>$E/(1-\nu^2)$ [GPa]</th>
<th>$\bar{\sigma}_0$</th>
<th>$\sigma_{0\text{mem}}$ [MPa]</th>
<th>$E/(1-\nu^2)_{\text{mem}}$ [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1127 x 8144</td>
<td>23.9</td>
<td>136.5</td>
<td>17.5</td>
<td>33.8</td>
<td>142.4</td>
</tr>
<tr>
<td>2</td>
<td>391 x 2126</td>
<td>15.2</td>
<td>129.5</td>
<td>1.4</td>
<td>61.5</td>
<td>140.7</td>
</tr>
<tr>
<td>3</td>
<td>1122 x 8146</td>
<td>10.9</td>
<td>135.9</td>
<td>7.9</td>
<td>19.0</td>
<td>142.3</td>
</tr>
<tr>
<td>4</td>
<td>870 x 3130</td>
<td>40.4</td>
<td>135.1</td>
<td>17.8</td>
<td>55.8</td>
<td>142.0</td>
</tr>
<tr>
<td>5</td>
<td>871 x 4635</td>
<td>38.3</td>
<td>132.2</td>
<td>17.3</td>
<td>52.6</td>
<td>139.2</td>
</tr>
<tr>
<td>6</td>
<td>1363 x 7660</td>
<td>34.0</td>
<td>132.7</td>
<td>37.5</td>
<td>42.2</td>
<td>137.4</td>
</tr>
<tr>
<td>7</td>
<td>1369 x 7647</td>
<td>33.2</td>
<td>133.8</td>
<td>36.6</td>
<td>41.3</td>
<td>138.2</td>
</tr>
<tr>
<td>8</td>
<td>1117 x 8134</td>
<td>33.2</td>
<td>132.0</td>
<td>24.7</td>
<td>43.6</td>
<td>137.4</td>
</tr>
<tr>
<td>9</td>
<td>1118 x 4134</td>
<td>34.8</td>
<td>140.0</td>
<td>24.5</td>
<td>45.6</td>
<td>145.8</td>
</tr>
<tr>
<td>10</td>
<td>868 x 6158</td>
<td>30.6</td>
<td>135.1</td>
<td>13.4</td>
<td>45.0</td>
<td>142.2</td>
</tr>
<tr>
<td>11</td>
<td>883 x 6144</td>
<td>23.4</td>
<td>135.6</td>
<td>10.6</td>
<td>36.4</td>
<td>143.4</td>
</tr>
<tr>
<td>12</td>
<td>618 x 4142</td>
<td>15.9</td>
<td>137.2</td>
<td>3.5</td>
<td>37.2</td>
<td>146.9</td>
</tr>
<tr>
<td>13</td>
<td>1373 x 7645</td>
<td>21.3</td>
<td>134.1</td>
<td>23.6</td>
<td>28.5</td>
<td>138.9</td>
</tr>
<tr>
<td>14</td>
<td>865 x 6137</td>
<td>12.7</td>
<td>142.7</td>
<td>5.2</td>
<td>24.5</td>
<td>152.0</td>
</tr>
<tr>
<td>15</td>
<td>370 x 2124</td>
<td>15.1</td>
<td>147.2</td>
<td>1.1</td>
<td>73.2</td>
<td>156.4</td>
</tr>
</tbody>
</table>

| Average ± standard deviation | 25.5 ± 10 | 136.0 ± 4.5 | 42.7 ± 14.2 | 143.0 ± 5.4 |

After micromachining, the membranes showed complicated buckling shapes. At such high $\bar{\sigma}_0$ values, the plane strain Euler deformation is not the shape of minimum elastic energy, as shown in Chapter 5. But, at pressures $P$ larger than a critical value $P_{cr}$, the deflection profiles are stretched and become again translationally invariant in their middle section. They are then properly described by the plane strain model. Load-deflection data were acquired in the plane strain regime $|P| > P_{cr}$.
4.4 Comparison with Experiment

Fig. 4.9 Extracted residual stress versus position of the membranes along the stress gradient. The parameter $d$ is the distance of each membrane to the straight line through the center of the wafer and perpendicular to the stress gradient.

Fig. 4.10 Load-deflection response of buckled long clamped PECVD silicon nitride plates of different widths $a$. 
Table 4.3 Dimensions and mechanical parameters of compressively prestressed thin film samples. Notation according to Table 4.1.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>(a) [(\mu m)]</th>
<th>(\sigma_0) [MPa]</th>
<th>(E/(1-\nu^2)) [GPa]</th>
<th>(\overline{\sigma_0})</th>
<th>(\sigma_{\text{mem}}) [MPa]</th>
<th>(E/(1-\nu^2)_{\text{mem}}) [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>841</td>
<td>-57.8</td>
<td>141.9</td>
<td>-582</td>
<td>-56.7</td>
<td>143.3</td>
</tr>
<tr>
<td>2</td>
<td>841</td>
<td>-81.4</td>
<td>143.9</td>
<td>-808</td>
<td>-80.4</td>
<td>145.4</td>
</tr>
<tr>
<td>3</td>
<td>839</td>
<td>-60.4</td>
<td>142.9</td>
<td>-600</td>
<td>-59.6</td>
<td>144.7</td>
</tr>
<tr>
<td>4</td>
<td>639</td>
<td>-63.3</td>
<td>143.9</td>
<td>-363</td>
<td>-62.4</td>
<td>146.0</td>
</tr>
<tr>
<td>5</td>
<td>637</td>
<td>-65.4</td>
<td>144.9</td>
<td>-369</td>
<td>-64.5</td>
<td>147.0</td>
</tr>
<tr>
<td>6</td>
<td>632</td>
<td>-60.3</td>
<td>142.0</td>
<td>-343</td>
<td>-59.3</td>
<td>144.1</td>
</tr>
<tr>
<td>7</td>
<td>535</td>
<td>-82.3</td>
<td>139.0</td>
<td>-342</td>
<td>-82.3</td>
<td>141.7</td>
</tr>
<tr>
<td>8</td>
<td>644</td>
<td>-42.6</td>
<td>142.7</td>
<td>-250</td>
<td>-41.3</td>
<td>144.7</td>
</tr>
<tr>
<td>9</td>
<td>541</td>
<td>-55.1</td>
<td>136.8</td>
<td>-238</td>
<td>-53.9</td>
<td>139.1</td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td>-63.2</td>
<td>142.0</td>
<td>-62.3</td>
<td>144.0</td>
<td></td>
</tr>
<tr>
<td>± standard deviation</td>
<td></td>
<td>±12.4</td>
<td>±2.6</td>
<td>±12.8</td>
<td>±2.4</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 4.10 shows experimental results of three samples with fits based on the plate model. Residual stress values, plane strain moduli, and reduced residual stresses extracted from nine structures are listed in Table 4.3, including dimensions. The residual stress of all samples is well below the critical buckling stress (\(\sigma_0 = -\pi^2/3\)). Residual stresses and plane strain moduli extracted using the membrane approximation are provided in Table 4.3 for comparison. The discrepancies between plate and membrane results are small. Agreement between the two models is better than in the case of type A and B samples. The reason for this is simple: at the pressures required to drive the buckled samples into a plane strain deformation, their effective line stress \(\bar{s}\) was larger than 400. In this range, the structures are well approximated by the membrane model. The results were obtained from data lying well in the nonlinear regime, where the influence of \(\sigma_0\) on the response...
is weak. As a result, the achieved $\sigma_0$ accuracy for such compressive layers is lower than for unbuckled films.

The results obtained with the three sample types are summarized in Fig. 4.11. It shows the average stress and the plane strain modulus as a function of the ratio of LF power to the total deposition power (see Section 2.3). The error bars indicate the standard deviation of the measured values. In the case of the stress values, the uncertainty might be larger than indicated by the error bars due to the presence of a stress gradient across the wafer. The stress clearly varies with the ratio of LF power to the total power. The slope of stress versus LF power is in good agreement with a previously measured value of roughly 10 MPa/(% LF power) using the wafer curvature method [68]. In contrast, the plane strain modulus increases only slightly with the ratio of LF power. Maeda et. al. [20] investigated the dependence of the biaxial modulus of PECVD deposited silicon nitride on the ratio of low frequency to high frequency power. They found an increase in the biaxial modulus with the percentage of LF power of 86 GPa at 0 % to 130 GPa at 44 % LF power. This increase might be explained by the higher density of the nitride films deposited with higher ratio of LF power. Maeda et. al. measured a mass density of the PECVD silicon nitride of about 1.84 g/cm$^3$ at 0% LF power and 2.52 g/cm$^3$ at 44% LF power [20].
The properties of CVD silicon nitride layers depend on fabrication conditions such as energy density [20,107,122,123], pressure [58, 107], temperature [59, 63], gas composition [55, 58], thermal history [57, 59, 62], and ion implantation dose [57]. Reported stress values for PECVD SiNₓ range between -1.6 GPa [58] and 680 MPa [63]. Elastic moduli range from 86 GPa [20] to 220 GPa [58]. The PECVD values reported here are within these extremes. Residual stresses of LPCVD silicon nitrides have been reported in the range from -63.6 MPa to 1.29 GPa. With values between 190 GPa [55] and 366 GPa [57], Young's modulus of LPCVD silicon nitride is usually higher than that of PECVD layers.
5 LONG RECTANGULAR PLATES – RIPPLE TRANSITION

In the previous chapter it was assumed that the long plates considered are invariant with respect to longitudinal translations. As a consequence, the displacement field of such plates is independent of the longitudinal coordinate. However, at a sufficiently strong compressive stress a mechanical instability transition occurs, referred to in the following as ripple transition, through which the translational symmetry is then lost. The critical stress value strongly depends on Poisson’s ratio of the plate. Thus, the instability transition provides a useful measurement principle to determine \( v \). This chapter analyses the ripple transition in detail.

In Section 5.1, the ripple transition is described and the basic underlying mechanism is identified. Based on this, an analytical theory for the ripple transition of infinitely long plates is developed in Section 5.2. A procedure how to extract \( v \) from a ripple transition is proposed in Section 5.3. Numerical results for the ripple transition are presented in Section 5.4. Load-deflection experiments on long micromachined membranes are described in Section 5.5 from which Poisson’s ratio of a PECVD silicon nitride is determined. The chapter closes with a discussion of how well the idealized model of the ripple transition describes the behavior of real samples.

5.1 Phenomenological Description

We consider a long rectangular membrane with length \( b \), and width \( a \ll b \). The coordinate system is that defined in Fig. 3.1 with the \( y \)-axes parallel to the long side of the membrane. The membrane is under high compressive stress causing the membrane to buckle. Fig. 5.1(a) shows a micrograph of such a micromachined membrane at zero differential pressure. In contrast to plane strain buck-
Fig. 5.1 Optical micrographs of a longitudinally buckled membrane (a) at zero pressure, (b) 15 kPa, and (c) 35 kPa. Membrane size is 650×4500 μm².

A differential pressure applied to the sample tends to straighten out the strongly folded profile. Fig. 5.1(b) shows the membrane under a pressure of 15 kPa. It is deflected due to the pressure and still shows a periodic deflection, which is, however, simpler than that of Fig. 5.1(a). This periodic profile is referred to as ripple. If the differential pressure is further increased the ripple disappears. Fig. 5.1(c) shows the deflection profile at 35 kPa.

The pressure dependence of this ripple is shown in Fig. 5.2. Membrane size is 839×8000 μm². The ripple has a sinusoidal form with an amplitude that decreases with increasing pressure. At a critical pressure of $P_{cr} = 20.6$ kPa, causing a critical center deflection of $w_{cr0} = 18.9$ μm, the ripple disappears. This is the ripple transition. At pressures higher than $P_{cr}$, the middle section of the membrane is in a state of plane strain and can thus be described with the plane strain model developed in the previous chapter.
5.1 Phenomenological Description

The ripple transition can be understood as follows. Under the load $P$, the stress components $\sigma_{xx}$ and $\sigma_{yy}$ are both increased above the prestress $\sigma_0$ due to the elongation of the structure in the $x$-direction. In the plane strain region $|P| > P_{cr}$, the stress amounts $\sigma_{xx} - \sigma_0$ and $\sigma_{yy} - \sigma_0$ caused by the deflection of the plate are, in view of Eq. 2.11, related by

$$\sigma_{yy}(P) - \sigma_0 = v(\sigma_{xx}(P) - \sigma_0).$$  (5.1)

At high pressures, $\sigma_{xx}(P)$ is sufficiently large for $\sigma_{yy}(P)$ to become tensile. With decreasing pressure, $\sigma_{yy}(P)$ is reduced and changes from tensile to compressive. At $P_{cr}$, $\sigma_{yy}$ is sufficiently compressive and the plate becomes unstable in the longitudinal direction. At $|P| < P_{cr}$ the ripple profile appears. According to Eq. 5.1 the ripple transition depends on $v$. Therefore, measurement of $P_{cr}$ or $w_{cr0}$ for a
long plate with known dimensions, $a$ and $h$, and material parameters, $\sigma_0$ and $D$, allows the determination of Poisson’s ratio $\nu$.

Fig. 5.3 gives a schematic overview of the load deflection of infinitely long plates. It shows the center deflection as a function of the residual stress and the differential pressure for a fixed $\nu$. The gray area delineates the regime in which the plate is in a state of plane strain. In this area, the load deflection behavior is governed by the plane strain model presented in Chapter 4. The plane strain regime is limited to the left by the Euler buckling and the ripple transition line. However, irrespective of the residual stress, at sufficiently high pressure, the plate is in a state of plane strain. Fig. 5.3 also shows the states of two membranes under bulge testing represented by the dashed lines. In Section 5.4 detailed quantitative results of the ripple transition are presented.
5.2 Analytical Theory

In this section a mathematical model of the ripple transition is developed. Since it is difficult to take into account the effects caused by the shorter edges, an infinitely long plate is discussed. Effects caused by the finite size of real samples are studied in Section 5.6.

To develop a mathematical description of the ripple transition we take advantage of the plane strain model of Chapter 4. Close to the critical point the reduced three dimensional deflection of the membrane can be written as the sum of a plane strain deflection \( \{u_{x,ps}(x,y), 0, w_{ps}(x,y)\} \) and a periodic ripple displacement \( \{u_{r,x}(x,y), u_{r,y}(x,y), w_{r}(x,y)\} \) with dimensionless period \( \lambda = \lambda/a \) and

\[
\int_{-1/2}^{1/2} \int_{0}^{1/2} \bar{w}_r(\bar{x}, \bar{y}) d\bar{x} d\bar{y} = 0. \tag{5.2}
\]

Thus, the full displacement field is

\[
\begin{bmatrix}
\bar{u}_x(\bar{x}, \bar{y}) \\
\bar{u}_y(\bar{x}, \bar{y}) \\
\bar{w}(\bar{x}, \bar{y})
\end{bmatrix} = \begin{bmatrix}
\bar{u}_{x,ps}(\bar{x}) + \bar{u}_{r,x}(\bar{x}, \bar{y}) \\
\bar{u}_{r,y}(\bar{x}, \bar{y}) \\
\bar{w}_{ps}(\bar{x}) + \bar{w}_r(\bar{x}, \bar{y})
\end{bmatrix}. \tag{5.3}
\]

The out-of-plane deflection \( \bar{w}_{ps}(\bar{x}) \) is given by Eq. 4.6. The in-plane displacement \( \bar{u}_{x,ps}(\bar{x}) \) can be calculated using Eq. 3.18, i.e.,

\[
\frac{\partial}{\partial \bar{x}} \bar{u}_{x,ps} = (\bar{\varepsilon}_{xx,ps} - \bar{\varepsilon}_0) - \frac{1}{2} \left( \frac{\partial}{\partial \bar{x}} \bar{w}_{ps}(\bar{x}) \right)^2. \tag{5.4}
\]

In the plane strain situation the strain of the deflected plate \( \bar{\varepsilon}_{xx,ps} \) is independent of \( \bar{x} \) and is given in terms of the parameters \( \bar{s} \) and \( \bar{\sigma}_0 \) (see Section 4.1) by

\[
\bar{\varepsilon}_{xx,ps} = \bar{s} - \frac{\nu}{1+\nu} \bar{\sigma}_0. \tag{5.5}
\]

The reduced residual strain \( \bar{\varepsilon}_0 \) is related to the reduced residual stress \( \bar{\sigma}_0 \) by
The ripple profile has the following symmetry property: if the origin of the coordinate system is chosen so that the plane \( \bar{y} = 0 \) is a mirror plane, the symmetries of the ripple displacement with respect to reflections in the planes \( \bar{x} = 0 \) and \( \bar{y} = 0 \) are

\[
\left[ \begin{array}{c}
\bar{u}_{x,r}(\bar{x}, \bar{y}) \\
\bar{u}_{y,r}(\bar{x}, \bar{y}) \\
\bar{w}_{r}(\bar{x}, \bar{y})
\end{array} \right] = \left[ \begin{array}{c}
-\bar{u}_{x,r}(\bar{x}, \bar{y}) \\
\bar{u}_{y,r}(\bar{x}, \bar{y}) \\
\bar{w}_{r}(\bar{x}, \bar{y})
\end{array} \right]
\] (5.7)

and

\[
\left[ \begin{array}{c}
\bar{u}_{x,r}(\bar{x}, \bar{y}) \\
\bar{u}_{y,r}(\bar{x}, \bar{y}) \\
\bar{w}_{r}(\bar{x}, \bar{y})
\end{array} \right] = \left[ \begin{array}{c}
\bar{u}_{x,r}(\bar{x}, -\bar{y}) \\
-\bar{u}_{x,r}(\bar{x}, -\bar{y}) \\
\bar{w}_{r}(\bar{x}, -\bar{y})
\end{array} \right],
\] (5.8)

respectively. To calculate the critical point, the ripple displacements \( \bar{u}_{x,r} \) and \( \bar{w}_{r} \) are expanded in truncated series of appropriate trial functions, which fulfill Eqs. 5.8 to 5.7 and have built-in boundary conditions along the long sides of the membrane (Eq. 3.1). These are

\[
\bar{u}_{x,r}(\bar{x}, \bar{y}) = \sum_{p=1}^{P_{\text{max}}} u_{p} \sin(2p\pi\bar{x}) \cos(k\bar{y})
\] (5.9)

and

\[
\bar{w}_{r}(\bar{x}, \bar{y}) = \sum_{m=1}^{M_{\text{max}}} w_{m} g_{m}(\bar{x}) \cos(k\bar{y})
\] (5.10)
5.2 Analytical Theory

where \( g_m(\bar{x}) = (\cos(2\pi m \bar{x}) - (-1)^m)/2 \) and \( \bar{k} = 2\pi/\bar{\kappa} \). Later in this section it will become clear that it is not necessary to expand the longitudinal displacement \( \bar{u}_{x, r}(\bar{x}, \bar{y}) \) explicitly.

At pressures \( [P] > P_{cr} \), the membrane is in a state of plane strain and the ripple displacement equals zero, i.e., \( w(\bar{x}, \bar{y}) = \bar{w}_{ps}(\bar{x}) \). At the critical pressure, the membrane becomes unstable with respect to the ripple displacement, i.e., the second order change \( \delta^2 U \) of the total energy \( U \) due to the addition of a small ripple deformation is zero. In view of Eq. 5.2 the work performed by the ripple deformation against \( \bar{P} \) is zero. Therefore, only the elastic energy \( \bar{U}_{el} = \bar{U}_0 + \bar{U}_b \) per unit length of the plate has to be considered. The energy \( \bar{U}_0 \) per unit length in terms of reduced quantities is according to Eq. 3.7 given by

\[
\bar{U}_0 = \frac{1}{2\bar{\kappa}} \int_0^1 \int_0^{1/2} (\bar{\epsilon}_{xx}^2 + \bar{\epsilon}_{yy}^2 + 2\nu \bar{\epsilon}_{xx} \bar{\epsilon}_{yy} + 2(1 - \nu) \bar{\epsilon}_{xy}^2) \, d\bar{x} \, d\bar{y}. \tag{5.11}
\]

Using Eqs. 5.4 to 5.6 and the definition of the strain tensor Eq. 3.18, the components of the strain tensor are

\[
\bar{\epsilon}_{xx} = \bar{\epsilon}_0 + \frac{\partial}{\partial \bar{x}} \bar{u}_{x, ps} + \frac{\partial}{\partial \bar{x}} \bar{u}_{x, r} + \frac{1}{2} \left( \frac{\partial}{\partial \bar{x}}(\bar{w}_{ps} + \bar{w}_r) \right)^2, \tag{5.12}
\]

\[
\bar{\epsilon}_{yy} = \frac{\bar{\sigma}_0}{1 + \nu} + \frac{\partial}{\partial \bar{y}} \bar{u}_{y, r} + \frac{1}{2} \left( \frac{\partial}{\partial \bar{y}} \bar{w}_r \right)^2, \tag{5.13}
\]

and

\[
\bar{\epsilon}_{xy} = \frac{1}{2} \left( \frac{\partial}{\partial \bar{y}} \bar{u}_{x, r} + \frac{\partial}{\partial \bar{y}} \bar{u}_{y, r} + \frac{\partial}{\partial \bar{x}} \bar{w}_{ps} \frac{\partial}{\partial \bar{y}} \bar{w}_r + \frac{\partial}{\partial \bar{x}} \bar{w}_r \frac{\partial}{\partial \bar{y}} \bar{w}_r \right). \tag{5.14}
\]

To find the instability point, at which the elastic energy of the plate can be lowered by adding the ripple, it is sufficient to consider those terms of the elastic
energy that are of second order in the ripple amplitude \( A \equiv \bar{w}_r(0,0) \). From a simple symmetry consideration, as shown in Fig. 5.4, it can be inferred how the in-plane displacements grow with the ripple amplitude. Figures 5.4(a) and (b) show the plane strain profile of membrane along the \( x \)- and \( y \)-axes, respectively. The addition of a small ripple \( \bar{w}_r \) to the profile causes in-plane displacements \( \bar{u}_{x,r} \) and \( \bar{u}_{y,r} \). As indicated in Fig. 5.4(a), \( \bar{u}_{x,r} \) changes its sign if \( \bar{w}_r \) is negated to \(-\bar{w}_r\). However, \( \bar{u}_{y,r} \) is independent of the sign of \( \bar{w}_r \), as shown in Fig. 5.4(b). Therefore, \( \bar{u}_{x,r} \) is, to the lowest order, linear in the ripple amplitude \( A \), while \( \bar{u}_{y,r} \) is quadratic in \( A \). As a consequence only terms of the elastic energy which are quadratic in \( \bar{u}_{x,r} \) or \( \bar{w}_r \) have to be considered. The reason is that terms of \( \bar{U}_0 \) which are linear in \( \bar{u}_{x,r} \) or \( \bar{u}_{y,r} \) and do not contain \( \bar{w}_r \) vanish through integration over \( y \) because of the periodicity of \( \bar{u}_{x,r} \) and \( \bar{u}_{y,r} \). Therefore, none of the terms of \( \bar{U}_0 \) quadratic in \( A \) contain the displacement \( \bar{u}_{y,r} \).

The energy \( \bar{U}_0 \) expanded to the second order in \( A \) and without terms independent of the ripple displacements is
\[
\bar{U}_0 = \frac{1}{2\tilde{k}^{1/2}} \int_0^{\tilde{k}} \int_0^{1/2} \left[ \left( \frac{\partial}{\partial x} \tilde{u}_{x,r} \right)^2 + \left( \frac{\partial}{\partial x} \tilde{w}_{ps} \right)^2 \left( \frac{\partial}{\partial x} \tilde{w}_r \right)^2 + \frac{1}{2} \left( \frac{\partial}{\partial x} \tilde{w}_r \right)^2 \right. \\
\left. + 2 \frac{\partial}{\partial x} \tilde{u}_{x,r} \frac{\partial}{\partial x} \tilde{w}_{ps} \frac{\partial}{\partial x} \tilde{w}_r + (\bar{\sigma}_0 + \nu(\bar{s} - \bar{\sigma}_0)) \left( \frac{\partial}{\partial y} \tilde{w}_r \right)^2 \right] + \frac{1 - \nu}{2} \left( \frac{\partial}{\partial y} \tilde{u}_{x,r} \right)^2 (5.15)
\]

\[
+ \frac{1 - \nu}{2} \left( \frac{\partial}{\partial y} \tilde{w}_{ps} \right)^2 \left( \frac{\partial}{\partial y} \tilde{w}_r \right)^2 + (1 - \nu) \frac{\partial}{\partial y} \tilde{u}_{x,r} \frac{\partial}{\partial y} \tilde{w}_{ps} \frac{\partial}{\partial y} \tilde{w}_r \right] d\tilde{x} d\tilde{y}.
\]

The bending energy Eq. 3.17 expanded to the second order of \( A \) contains no other displacements than \( \tilde{w}_r \). \( \bar{U}_b \) can be simplified by integration by parts with use of the boundary conditions and the periodicity of the ripple displacement. It reads

\[
\bar{U}_b = \frac{1}{2\tilde{k}^{1/2}} \int_0^{\tilde{k}} \int_0^{1/2} \left[ \left( \frac{\partial}{\partial x} \tilde{w}_r \right)^2 + 2 \frac{\partial}{\partial x} \tilde{w}_r \frac{\partial}{\partial x} \tilde{w}_r + \left( \frac{\partial}{\partial y} \tilde{w}_r \right)^2 \right] d\tilde{x} d\tilde{y}. (5.16)
\]

Table 5.1 lists the resulting integrals of the individual terms in Eqs. 5.15 and 5.16. The elastic energy \( \bar{U}_{el} \) is thus a function of the variational parameter \( \tilde{w}_m \) and \( u_p \), but also of the material parameters \( \bar{\sigma}_0, \bar{s} \) and \( \nu \), and the wavevector \( \tilde{k} \). Note that later in this chapter we use \( \bar{w}_0 \) instead of \( \bar{s} \) as a parameter. Both are related by Eq. 4.9.

The equilibrium condition \( d\bar{U}_{el}/du_p = 0 \) leads to

\[
u_p = \sum_m w_m \frac{\vartheta_{pm}(\bar{\sigma}_0, \bar{s}, \tilde{k}, \nu)}{\kappa_p(\tilde{k}, \nu)}, (5.17)
\]

where

\[
\vartheta_{pm}(\bar{\sigma}_0, \bar{s}, \tilde{k}, \nu) = \pi^2 pm \phi_{pm}(\bar{\sigma}_0, \bar{s}) - \frac{1 - \nu}{4} \kappa_p \psi_{pm}(\bar{\sigma}_0, \bar{s}) (5.18)
\]

and
Table 5.1 Individual terms of Eqns. 5.16 and 5.17 and their integrals. The integration runs over $\tilde{x}$ and $\tilde{y}$ with the boundaries as specified in those equations. The factor $1/2k$ is included. The functions $\xi_{m_j}(\sigma, s)$, $\eta_{m_j}(\sigma, s)$, $\psi_{p_m}(\sigma, s)$, and $\phi_{p_m}(\sigma, s)$ are defined in Table 5.2.

<table>
<thead>
<tr>
<th>Integrand</th>
<th>Integral</th>
</tr>
</thead>
<tbody>
<tr>
<td>$(\frac{\partial}{\partial x} \tilde{u}_{x,r})^2$</td>
<td>$\frac{1}{2} \pi^2 \sum_{p,q} \mu_p \mu_q^2$</td>
</tr>
<tr>
<td>$(\frac{\partial}{\partial x} \tilde{w}_{ps})^2 (\frac{\partial}{\partial x} \tilde{w}_r)^2$</td>
<td>$\frac{1}{4} \pi^2 \sum_{m,j} w_m w_j m^2 \xi_{m_j}(\tilde{s})$</td>
</tr>
<tr>
<td>$(\frac{\partial}{\partial x} \tilde{w}_r)^2$</td>
<td>$\frac{1}{8} \pi^2 \sum_{m} (w_m)^2 m^2$</td>
</tr>
<tr>
<td>$\frac{\partial}{\partial x} \tilde{u}<em>{x,r} \frac{\partial}{\partial x} \tilde{w}</em>{ps} \frac{\partial}{\partial x} \tilde{w}_r$</td>
<td>$-\pi^2 \sum_{p,m} u_p w_m p m \phi_{p_m}(\tilde{s})$</td>
</tr>
<tr>
<td>$(\frac{\partial}{\partial y} \tilde{w}_r)^2$</td>
<td>$\frac{1}{8} k^2 \sum_{m,j} w_m w_j (-1)^m+j \left(1 + \frac{1}{2}\delta_{mj}\right)$</td>
</tr>
<tr>
<td>$(\frac{\partial}{\partial y} \tilde{u}_{x,r})^2$</td>
<td>$\frac{1}{8} k^2 \sum_{p} u_p^2$</td>
</tr>
<tr>
<td>$(\frac{\partial}{\partial y} \tilde{w}_{ps})^2 (\frac{\partial}{\partial y} \tilde{w}_r)^2$</td>
<td>$\frac{1}{4} k^2 \sum_{m,j} w_m w_j \eta_{m_j}(\tilde{s})$</td>
</tr>
<tr>
<td>$\frac{\partial}{\partial y} \tilde{u}<em>{x,r} \frac{\partial}{\partial y} \tilde{w}</em>{ps} \frac{\partial}{\partial y} \tilde{w}_r$</td>
<td>$\frac{1}{4} k^2 \sum_{p,m} u_p w_m \psi_{p_m}(\tilde{s})$</td>
</tr>
<tr>
<td>$(\frac{\partial^2}{\partial x^2} \tilde{w}_r)^2$</td>
<td>$\frac{1}{2} \pi^4 \sum_{m} w_m^4$</td>
</tr>
<tr>
<td>$(\frac{\partial^2}{\partial y^2} \tilde{w}_r)^2$</td>
<td>$\frac{1}{16} k^4 \sum_{m,j} w_m w_j (-1)^m+j \left(1 + \frac{1}{2}\delta_{mj}\right)$</td>
</tr>
<tr>
<td>$\frac{\partial^2}{\partial x^2} \tilde{w}_r \frac{\partial^2}{\partial y^2} \tilde{w}_r$</td>
<td>$\frac{1}{8} \pi^2 k^2 \sum_{m} w_m^2 m^2$</td>
</tr>
</tbody>
</table>
5.2 Analytical Theory

Table 5.2 Definition of \( \zeta_{mj}(\tilde{\sigma}_0, \tilde{s}) \), \( \xi_{mj}(\tilde{\sigma}_0, \tilde{s}) \), \( \psi_{pm}(\tilde{\sigma}_0, \tilde{s}) \), and \( \phi_{pm}(\tilde{\sigma}_0, \tilde{s}) \).

\[
\zeta_{mj}(\tilde{\sigma}_0, \tilde{s}) = \int_{-1/2}^{1/2} \left( \frac{\partial}{\partial \tilde{x}} \tilde{w}_{ps}(\tilde{x}) \right)^2 \sin(2\pi m \tilde{x}) \sin(2\pi j \tilde{x}) d\tilde{x}
\]

\[
\xi_{mj}(\tilde{\sigma}_0, \tilde{s}) = \int_{-1/2}^{1/2} \left( \frac{\partial}{\partial \tilde{x}} \tilde{w}_{ps}(\tilde{x}) \right)^2 g_m(\tilde{x}) g_j(\tilde{x}) d\tilde{x}
\]

\[
\phi_{pm}(\tilde{\sigma}_0, \tilde{s}) = \int_{-1/2}^{1/2} \frac{\partial}{\partial \tilde{x}} \tilde{w}_{ps}(\tilde{x}) \cos(2\pi p \tilde{x}) \sin(2\pi m \tilde{x}) d\tilde{x}
\]

\[
\psi_{pm}(\tilde{\sigma}_0, \tilde{s}) = \int_{-1/2}^{1/2} \frac{\partial}{\partial \tilde{x}} \tilde{w}_{ps}(\tilde{x}) \sin(2\pi p \tilde{x}) g_m(\tilde{x}) d\tilde{x}
\]

\[
\kappa_p(\tilde{v}, \tilde{v}) = p^2 \pi^2 + \frac{1 - \tilde{v}^2}{8 \tilde{k}^2} \quad (5.19)
\]

With Eq. 5.17 the variational parameters \( u_p \) of the in-plane displacement \( u_{x,y}(\tilde{x},\tilde{y}) \) can be eliminated from \( \overline{U}_{el} \). Thus, the coefficients \( w_m \) are the only variational parameters. \( \overline{U}_{el} \) is then a quadratic function of the variational parameters \( w_m \) and therefore its gradient with respect to the \( w_m \) at \( w_m = 0 \) equals zero. Hence, the critical point \( \{ \tilde{w}_0, \tilde{\sigma}_0, \tilde{v}, \tilde{k} \} \), at which \( \overline{U}_{el} \) can be lowered by forming the ripple, is the point at which the curvature of \( \overline{U}_{el} \) at \( w_m = 0 \) becomes negative in a direction of the parameter space \( \{ w_m \} \). To find this point, we form the Hessian \( H \) of \( \overline{U}_{el} \) with respect to the coefficients \( w_m \). Its components \( H_{nm} \) are
\[
\frac{\partial}{\partial w_m} \frac{\partial}{\partial w_j} U_{el} = - \sum_p \frac{\partial_{p_m}(\tilde{\sigma}_0, \tilde{s}, \tilde{k}, \nu) \partial_{p_j}(\tilde{\sigma}_0, \tilde{s}, \tilde{k}, \nu)}{\kappa_p(\tilde{k}, \nu)} + \frac{1}{2} \pi^2 m j \zeta_{mj}(\tilde{\sigma}_0, \tilde{s}) \\
+ \left( \frac{k^2}{8} (\tilde{\sigma}_0 + \nu(\tilde{s} - \tilde{\sigma}_0)) + \frac{k^4}{96} \right)(-1)^m + j \left( 1 + \frac{1}{2} \delta_{mj} \right) \\
+ \frac{1 - \nu}{4} k^2 \zeta_{mj}(\tilde{\sigma}_0, \tilde{s}) + \delta_{mj} \frac{1}{4} \left( \frac{\pi^2 m^2}{3} + \frac{\pi^4 m^4}{4} + \frac{k^2}{6} \pi^2 m^2 \right).
\]

\( H \) is a symmetric matrix and can be brought into diagonal form. The eigenvalues of \( H \) describe the curvature of the surface \( \overline{U}_{el}(w_m) \) at \( w_m = 0 \) along the corresponding eigenvectors.

The instability equations can now be formulated. Consider a membrane with material properties \( \tilde{\sigma}_0 \) and \( \nu \). At high pressures causing a large \( \tilde{w}_0 \), the plane strain profile is stable. Therefore the eigenvalues of \( H \) are positive for all \( \tilde{k} \). At the critical center deflection \( \overline{w}_{0cr} \), one eigenvalue of \( H \) becomes zero, i.e., changes its sign, at one particular wavevector \( \tilde{k} \). However, for other \( \tilde{k} \) the eigenvalues of \( H \) are still positive. From the first condition it follows for \( \overline{w}_0 = \overline{w}_{0cr} \) that

\[
\det(H) = 0 \quad (5.21)
\]

and from the second that

\[
\frac{\partial}{\partial \tilde{k}} \det(H) = 0. \quad (5.22)
\]

It is noteworthy that Eqs. 5.21 and 5.22 are not equivalent to the above mentioned critical conditions. There may be more than one solution \( \overline{w}_0 \) to these equations, as shown in Fig. 5.5. In this figure, \( \det(H) \) is plotted versus \( \tilde{k} \) for different values of \( \overline{w}_0 \). Poisson’s ratio is 0.25 and \( \tilde{\sigma}_0 = -500 \). With decreasing \( \overline{w}_0 \), \( \det(H) \) also decreases. At \( \overline{w}_0 = 23.71 \), its minimum becomes zero for the first time at \( \tilde{k} = 27.3 \). This is the critical point. However, there is a second point at a smaller center deflection that also fulfills Eqs. 5.21 and 5.22. This point is \( \overline{w}_0 = 21.45 \) and \( \tilde{k} = 32.5 \). Among all the solution of Eqs. 5.21 and 5.22, that with the largest \( \overline{w}_0 \) value has to be taken.
5.3 Measurement Procedure

Four parameters characterize the critical point: $\bar{\sigma}_0$, $\nu$, $\bar{k}$, and $\bar{w}_0$. The pressure is not listed as a critical parameter since $P$ is defined by $\bar{\sigma}_0$ and $\bar{w}_0$ through the plane strain equations 4.8 and 4.9. The critical equations 5.21 and 5.22 constitute two conditions for the critical parameters $\bar{\sigma}_0$, $\nu$, $\bar{k}$, and $\bar{w}_0$. Thus, Poisson’s ratio can in principle be extracted from a measurement of one of the following parameter pairs: {$\bar{\sigma}_0$, $\bar{k}$}, {$\bar{w}_0$, $\bar{k}$}, and {$\bar{\sigma}_0$, $\bar{w}_0$}. In our experiments best results in terms of a low standard variation were obtained using the parameter pair {$\bar{\sigma}_0$, $\bar{w}_0$}. The other parameter pairs contain the wavevector $\bar{k}$. Although $\bar{k}$ can be measured with good accuracy, it is not a good input parameter to Eqs. 5.21 and 5.22 to calculate $\nu$. The reason is that $\bar{k}$ might be influenced by the finite length of the sample as discussed in Section 5.6. This can lead to a considerable error in $\nu$.

In the following it is explained how $\nu$ is determined in our experiments. The procedure is outlined in Fig. 5.6. Firstly, a bulge test experiment is performed with $|P| \geq |P_{cr}|$. Using the measured film thickness $h$ the experimental center deflections $\bar{w}_{0,i}$ are reduced to $\bar{w}_{0,i}$. The plane strain model is then fitted to the data {$P$,
\begin{align*}
\sum_i (\overline{w}_{0,i} - c P_i G(\overline{\dot{s}}_i, 0))^2 &= \min \\
\overline{\dot{s}}_i &= \overline{\sigma}_0 + \frac{1}{2} \overline{\dot{w}}_{0,i}^2 H(\overline{\dot{s}}_i)
\end{align*}

\begin{align*}
\det(H(\overline{\sigma}_0, \overline{w}_{0cr}, \overline{k}, \nu)) &= 0 \\
\frac{\partial}{\partial \overline{k}} \det(H(\overline{\sigma}_0, \overline{w}_{0cr}, \overline{k}, \nu)) &= 0 \\
\nu \quad \overline{k} &\rightarrow \overline{k} a \approx k_{\text{exp}}^2
\end{align*}

\textbf{Fig. 5.6} Diagram illustrating how to extract the material properties } E, \sigma_0, \nu \text{ from bulge test and ripple transition.}

\(\overline{w}_{0,i}\) with fit coefficients } \overline{\sigma}_0 \text{ and } c, \text{ as shown in Fig. 5.6. The latter parameter reduces } P \text{ to } \overline{P}, \text{i.e., } \overline{P} = cP, \text{ and does not affect the evaluated } \nu.
5.4 Numerical Results

Secondly, the critical center deflection \( w_{0cr} \) and the experimental wavevector \( k_{exp} \) at the critical point are measured. Again, \( h \) is used to reduce \( w_{0cr} \) to \( \bar{w}_{0cr} \). The input parameters for the critical equations 5.21 and 5.22 are \( \sigma_0 \) and \( \bar{w}_{0cr} \), from which finally \( \nu \) and \( \bar{k} \) are computed. If the width \( a \) of the membrane is known, the computed wavevector \( \bar{k} \) and the measured \( k_{exp} \) can be compared, since \( \bar{k}/a = k_{exp} \) should hold. This provides an independent consistency check.

It is noteworthy that the extracted \( \nu \) is independent of the membrane width \( a \) and depends only weakly on the film thickness. In the case of the membranes analyzed in Section 5.5, \( \nu \) varied by 0.33% if \( h \) is changed by 1%. The weak dependence of \( \nu \) on \( h \) has the following reason. An error in \( h \) results in an error in \( \bar{w}_{cr0} \) and in the center deflections \( \bar{w}_{0,i} \) measured during bulge testing. Since \( \sigma_0 \) is dependent on \( \bar{w}_{0,i} \), it depends in turn on \( h \). The errors in \( \bar{w}_{cr0} \) and \( \sigma_0 \) almost compensate for each other when \( \nu(\sigma_0, \bar{w}_{cr0}) \) is computed.

5.4 Numerical Results

Convergence

The convergence behavior of \( \nu(\bar{w}_{0cr}, \sigma_0) \) with respect to the truncation parameters \( m_{max} \) and \( p_{max} \) in Eqs. 5.9 and 5.10 was investigated. Poisson’s ratio was computed for different values of \( \bar{w}_{0cr} \) and \( \sigma_0 \) while \( m_{max} \) was varied between 1 and 15. The parameter \( p_{max} \) was set equal to \( m_{max} \). As an example, Fig. 5.7 shows the convergence behavior of \( \nu(\bar{w}_{0cr}, \sigma_0) \) at \( \bar{w}_{0cr} = 24 \) and \( \sigma_0 = -500 \). At high \( m_{max} \) the computed \( \nu \) still increases noticeably with \( m_{max} \). A fit of the form \( c_1 + c_2/(m_{max} - c_3)^{c_4} \), where \( c_1, \ldots, c_4 \) are the fit parameters excellently models the data, but results in an asymptotic value of \( \nu = 0.2440 \). This is about 0.3% higher than the value obtained for \( m_{max} = 15 \). From the numerical convergence experiments with different \( \bar{w}_{0cr} \) and \( \sigma_0 \), it can be concluded, that \( m_{max} \) must be larger than 10, if \( \nu \) has to be computed with an absolute uncertainty of 0.001. If an uncertainty of 0.003 is acceptable, \( m_{max} \) can be set to 5.

The expansions of the displacements \( \bar{w}_x \) and \( \bar{u}_{x,r} \) in Eqs. 5.9 and 5.10 only contain the first Fourier term with respect to the \( \bar{y} \)-direction. To check whether higher Fourier terms are in fact negligible we computed \( \nu(\bar{w}_{0cr}, \sigma_0) \) using the expansions
Fig. 5.7 Convergence of $v$ at $w_{0\text{cr}} = 24$ and $\sigma_0 = -500$ with respect to the truncation of the series in Eqs. 5.9 and 5.10. The truncation parameter $p_{\text{max}}$ is set equal to $m_{\text{max}}$.

\[
\bar{u}_{x,r}(\bar{x}, \bar{y}) = \sum_{p=1}^{p_{\text{max}}} \sum_{q=1}^{q_{\text{max}}} u_{pq} \sin(2p\pi\bar{x}) \cos(q\bar{k}\bar{y}) \tag{5.23}
\]

and

\[
\bar{w}_{r}(\bar{x}, \bar{y}) = \sum_{m=1}^{m_{\text{max}}} \sum_{n=1}^{n_{\text{max}}} w_{mn} g_{m}(\bar{x}) \cos(n\bar{k}\bar{y}) \tag{5.24}
\]

The calculated $v$ was found to be independent of terms with $n, q > 1$.

In the following, numerical results for the critical point are presented. Firstly, the particular case of the ripple transition at zero differential pressure is discussed. Secondly, the general case with differential pressure is described.
5.4 Numerical Results

Fig. 5.8 Ripple transition of a long rectangular membrane with $v = 0.25$. The solid lines show the Euler buckling deflection and the triangular markers indicate the ripple transitions at $\overline{P} = 0$. These critical points are numbered with Arabic numerals. The dotted lines show the ripple transition for $\overline{P} \neq 0$.

$\overline{P} = 0$

Long membranes have three different critical points with $\overline{P} = 0$, at which they become unstable with respect to the ripple. Fig. 5.8(a) and (b) shows these critical points for the case $v = 0.25$. Figs. 5.8(b) is a close up of the boxed region in Fig. 5.8(a). If $\overline{\sigma}_0$ is decreased from zero to negative values, the ripple occurs at $\overline{\sigma}_{cr1} = -3.0901$ independent of $v$. Note that this value is less negative than the critical Euler stress $-\pi^2/3 \approx -3.290$ (see Section 4.2). The wavevector of the ripple occurring at $\overline{\sigma}_{cr1}$ is $\overline{k} = 2.399$.

The second critical point occurs at $\overline{\sigma}_{cr2} \approx -3.45$. The wavevector is $\overline{k} = 2.7$. Both $\overline{k}$ and $\overline{\sigma}_{cr2}$ are almost independent of $v$. In the region between $\overline{\sigma}_{cr1}$ and $\overline{\sigma}_{cr2}$, the membrane shows an $x$- and $y$-dependent buckling profile at $\overline{P} = 0$. However, the membrane can be driven into the state of plane strain by the application of a sufficiently high differential pressure. The dashed line in Fig. 5.8(b) shows the smallest center deflection of the pressurized membrane at which the plane strain deflection is stable.
The third critical point occurs at a strongly \( \nu \)-dependent critical stress \( \bar{\sigma}_{cr3}(\nu) \). In the case of \( \nu = 0.25 \) the critical stress is \(-21.4\), as shown in Fig. 5.8(a). We experimentally observed that a membrane with \( \bar{\sigma}_0 \) between \( \bar{\sigma}_{cr2} \) and \( \bar{\sigma}_{cr3} \) can assume two stable buckling profiles at \( \bar{P} = 0 \): the plane strain buckling profile described in Chapter 4 and a more complicated \( x \)- and \( y \)-dependent profile. A membrane showing the latter profile can be brought into the plane strain profile by the application of a brief pressure pulse.

At reduced residual stresses \( \bar{\sigma}_0 < \bar{\sigma}_{cr3}(\nu) \), the plane strain buckling profile is unstable. A differential pressure is required to drive a membrane with such a \( \bar{\sigma}_0 \) into the state of plane strain. The dashed line in Fig. 5.8(a) shows the smallest center deflection at which the plane strain deflection is stable as a function of \( \bar{\sigma}_0 \).

Along the Euler buckling curve \( (\bar{P} = 0) \), \( \bar{w}_0 \) and \( \bar{\sigma}_0 \) are uniquely related by Eq. 4.12. Therefore, the number of independent parameters characterizing a critical point lying on the Euler curve reduces to one. Thus, Poisson’s ratio can be inferred from the third \( \nu \)-dependent critical point by measuring one critical parameter, for instance, either \( \bar{\sigma}_{cr3} \) or \( \bar{w}_{cr0} \). Fig. 5.9 shows these parameters of the third critical point as functions of \( \nu \). The \( \nu \)-dependence of \( \bar{w}_{cr0} \) is excellently modeled by
\[
\bar{w}_{cr0}(\nu)\bigg|_{\bar{p} = 0} = 2.127 + 1.884\nu + 1.597\nu^2 + 2.871\nu^4. \tag{5.25}
\]

The third critical point of a long micromachined membrane may be experimentally determined by heating (or cooling) of the membrane and simultaneous monitoring of the buckling profile. Since the thermal expansion of thin film and substrate are usually different a temperature variation changes the reduced residual stress of the membrane. This enables \(\bar{\sigma}_0\) to be adjusted to \(\bar{\sigma}_{cr3}\).

Fig. 5.10 Euler buckling (light gray surface) and ripple transition (dark gray surface) plotted in a \(\bar{\sigma}_0, \nu, w_0\) diagram.
\( \bar{P} \neq 0 \)

Figure 5.10 shows the critical center deflection \( \bar{w}_{cr0} \) as a function of \( \bar{\sigma}_0 \) and \( \nu \). The center deflection for Euler buckling is also shown. Note that Euler buckling is independent of \( \nu \). In contrast, \( \bar{w}_{cr0} \) depends strongly on \( \nu \). The thick solid line in Fig. 5.10 shows \( \bar{w}_{cr0} \) of the third critical point for \( \bar{P} = 0 \) discussed in the previous subsection. This line marks the onset of the ripple transition surface. The reduced residual stress corresponding to this line is \( \bar{\sigma}_{cr3}(\nu) \). For \( \bar{\sigma}_0 < \bar{\sigma}_{cr3}(\nu) \), the Euler buckling profile is not the equilibrium profile of a membrane.

Fig. 5.11(a) shows \( \bar{w}_{cr0} \) as a function of \( \bar{\sigma}_0 \) and \( \nu \) for a larger range of reduced residual stresses. The critical center deflection increases with increasing negative residual stress and decreasing Poisson’s ratio. For membranes with \( \nu = 0 \) very large center deflection must be realized to drive such samples into the state of plane strain.

The dependence of \( \bar{k} \) on \( \bar{\sigma}_0 \) and \( \nu \) is shown in Fig. 5.11(b). The wavevector increases with increasing \( |\bar{\sigma}_0| \) and decreasing \( \nu \).

**Ripple Shape**

Ripple profiles \( \bar{w}_r(\bar{x},0) \) along the \( \bar{x} \) axis are shown in Fig. 5.12. The profiles are normalized to \( \bar{w}_r(0,0) \). Fig. 5.12(a) shows the profiles at \( \bar{\sigma}_0 = \bar{\sigma}_{cr1} \) and \( \bar{\sigma}_0 < \bar{\sigma}_{cr3} \). At \( \bar{\sigma}_{cr1} \), the profile \( \bar{w}_r(\bar{x},0) \) is given by the first Fourier term, \( g_1(\bar{x}) \) of Eq. 5.10, with good accuracy and is independent of \( \nu \). At large negative reduced stresses \( \bar{\sigma}_0 \), \( \bar{w}_r(\bar{x},0) \) changes only weakly with \( \nu \) and \( \bar{\sigma}_0 \).

In the special case of the ripple transition at \( \bar{\sigma}_{cr3}(\nu) \) (\( \bar{P} = 0 \)), the ripple profile develops a local minima at \( \bar{x} = 0 \) and shows a strong dependence on Poisson’s ratio as shown in Eq. 5.12.

**Error Propagation**

This subsection discusses how errors in the parameters \( \bar{\sigma}_0 \) and \( \bar{w}_{cr0} \) propagate to errors in the evaluated \( \nu(\bar{\sigma}_0, \bar{w}_{cr0}) \). According to the Gaussian law of error propagation, errors in \( \bar{\sigma}_0 \) and \( \bar{w}_{cr0} \) denoted as \( \Delta \bar{\sigma}_0 \) and \( \Delta \bar{w}_{cr0} \) cause an uncertainty
Fig. 5.11 Plots of (a) $\bar{w}_{c,0}$ versus $\bar{\sigma}_0$ and (b) the critical $\bar{k}$ versus $\bar{\sigma}_0$ for different values of $\nu$. 

5.4 Numerical Results
\[
\Delta \nu = \sqrt{\left( \frac{\partial \nu}{\partial \bar{w}_{cr0}} \mid_{\bar{\sigma}_0} \right)^2 \left( \frac{\Delta \bar{w}_{cr0}}{\bar{w}_{cr0}} \right)^2 + \left( \frac{\partial \nu}{\partial \bar{\sigma}_0} \mid_{\bar{w}_{cr0}} \right)^2 \left( \frac{\Delta \bar{\sigma}_0}{\bar{\sigma}_0} \right)^2}
\]

of the computed \( \nu \). The factors \( d_1 \) and \( d_2 \) are shown in Fig. 5.13 as a function of \( \nu \). Using these plots, the achievable measurement accuracy of \( \nu \) can easily be determined. As an example, a thin film with an estimated Poisson’s ratio of 0.2 is considered. Further it is assumed that \( \bar{\sigma}_0 \) and \( \bar{w}_{cr0} \) can be measured with relative uncertainties of 3\% and 1\%, respectively, and that the sample has \( \bar{\sigma}_0 = -400 \). According to Fig. 5.13, \( d_1 = -0.5 \). From Fig. 5.11, it can be found that \( \bar{w}_{cr0} = 23 \) and therefore \( d_2 = 0.3 \) (see Fig. 5.13(b)). Thus, using Eq. 5.26, the overall achievable accuracy of \( \nu \) is found to be ±0.01.

As shown in Fig. 5.13, \( d_1 \) and \( d_2 \) decrease with increasing \( |\bar{\sigma}_0| \) and \( \bar{w}_{cr0} \), respectively. Therefore, a larger compressive reduced stress \( \bar{\sigma}_0 \) enables \( \nu \) to be determined with higher accuracy.

As explained in Section 5.3, errors in \( \bar{h} \) exert only a minor influence on the accuracy of the determined \( \nu \). Similarly, errors that are due to a wrong linear scaling of the measured center deflections only weakly affect the determined \( \nu \) values. Such errors may occur, for example, due to a wrong calibration of the profilometer used to measure the center deflections. Therefore, if the uncertainty of \( \nu \) is calculated using Eq. 5.26, errors of \( \bar{w}_{cr0} \) that are due to a wrong linear scaling can be excluded from \( \Delta \bar{w}_{cr0} \).
Fig. 5.12 Ripple profiles $\overline{w_r}(x, 0)$ at (a) $\overline{\sigma}_0 = \overline{\sigma}_{cr1}$ and $\overline{\sigma}_0 \ll \overline{\sigma}_{cr3}$ and (b) $\overline{\sigma}_0 = \overline{\sigma}_{cr3}$.
Fig. 5.13 The factors (a) $d_1$ and (b) $d_2$ of Eq. 5.26 as a function of $v$ and $\sigma_0$. 
5.5 Experimental Results

The model of the ripple transition described in Section 5.2 is now applied to the characterization of a silicon nitride thin film. Seven samples of type C (see Table 2.2), with width $a$ between 535 µm and 841 µm and aspect ratios between 10:1 and 16:1, were investigated. The reduced residual stress $\bar{\sigma}_0$ was extracted from the load-deflection behavior of these samples in the plane strain regime ($|P| \geq P_{cr}$). The results are shown in Table 4.3. Only samples No. 1 to 7 of Table 4.3 are investigated in this section in view of the ripple transition.

The critical deflection $w_{cr0}$ was determined from longitudinal and transverse profiles at pressures close to $P_{cr}$. As an example, Fig. 5.2 shows profiles of sample No. 1. From such data the pressure-dependent wavelength $\lambda(P)$ and ripple amplitude $A(P)$ were determined by Fourier transformation. These allowed the determination of the critical parameters $P_{cr}$, $\lambda_{cr}$, and $w_{cr0}$. Fig. 5.14 shows the pressure-dependent $A$ and $\lambda$ for membrane No. 2 under negative differential pressures. $A$ decreases strongly with increasing pressure for $|P| < P_{cr}$ and shows a sharp transition to zero at the critical pressure. In contrast, the wavelength of the ripple shows a weak pressure dependence. The critical pressure and wavelength are in this case $P_{cr} = -28.1$ kPa and $\lambda_{cr} = 178.8$ µm. The corresponding $w_{cr0}$ is

![Graph](a) and ![Graph](b)

Fig. 5.14 Pressure-dependent (a) ripple amplitude $A$ and (b) wavelength $\lambda$ of sample No. 2.
Table 5.3 Aspect ratio $b:a$, width $a$, measured critical deflection $<w_{cr0}>$, pressure $<P_{cr}>$, wavelength $<\lambda_{cr,meas}>$, calculated ripple wavelength $\lambda_{cr,calc}$, and Poisson’s ratio $\nu$ of silicon nitride samples.

<table>
<thead>
<tr>
<th>No.</th>
<th>$b:a$</th>
<th>$a$ [\mu m]</th>
<th>$&lt;w_{cr0}&gt;$ [\mu m]</th>
<th>$&lt;P_{cr}&gt;$ [kPa]</th>
<th>$&lt;\lambda_{cr,meas}&gt;$ [\mu m]</th>
<th>$\lambda_{cr,calc}$ [\mu m]</th>
<th>$\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10:1</td>
<td>841</td>
<td>18.9±0.1</td>
<td>20.4±0.2</td>
<td>185.4±2</td>
<td>181.7</td>
<td>0.230</td>
</tr>
<tr>
<td>2</td>
<td>10:1</td>
<td>841</td>
<td>21.2±0.1</td>
<td>27.8±0.3</td>
<td>178.9±1</td>
<td>172.7</td>
<td>0.269</td>
</tr>
<tr>
<td>3</td>
<td>10:1</td>
<td>839</td>
<td>18.9±0.2</td>
<td>20.7±0.3</td>
<td>185.0±3</td>
<td>181.0</td>
<td>0.237</td>
</tr>
<tr>
<td>4</td>
<td>10:1</td>
<td>639</td>
<td>13.7±0.2</td>
<td>22.6±0.2</td>
<td>168.3±2</td>
<td>162.6</td>
<td>0.255</td>
</tr>
<tr>
<td>5</td>
<td>10:1</td>
<td>637</td>
<td>13.8±0.1</td>
<td>23.2±0.6</td>
<td>164.7±2</td>
<td>161.8</td>
<td>0.259</td>
</tr>
<tr>
<td>6</td>
<td>12:1</td>
<td>632</td>
<td>13.8±0.2</td>
<td>24.1±0.4</td>
<td>165.1±1</td>
<td>160.1</td>
<td>0.234</td>
</tr>
<tr>
<td>7</td>
<td>16:1</td>
<td>535</td>
<td>12.6±0.1</td>
<td>32.3±0.3</td>
<td>147.5±1</td>
<td>143.2</td>
<td>0.290</td>
</tr>
</tbody>
</table>

-21.14 \mu m. Since positive and negative pressures were applied, two sets of critical parameters were obtained for each sample. Table 5.3 lists their averages $<P_{cr}>$, $<\lambda_{cr,meas}>$, and $<w_{cr0}>$. The uncertainties in Table 5.3 show the deviation from the mean or the extraction uncertainty, whichever was larger. Clearly, the critical point can be determined accurately.

Finally, with the $\bar{\sigma}_0$ values from Table 4.3 and the deflections $<w_{cr0}>$ from Table 5.3, $\nu$ and $\lambda_{cr,calc}$ were calculated for each sample using Eqs. 5.21 and 5.22. The average Poisson’s ratio is 0.254 with a standard deviation of 0.022. Note that the calculated critical wavelength $\lambda_{cr,calc}$ is not used to determine $\nu$. However, it can be compared with the measured critical wavelength and thus provides an independent consistency test. The calculated $\lambda_{cr,calc}$ and the measured $<\lambda_{cr,meas}>$ in Table 5.3 agree within less than 4% discrepancy and are therefore considered consistent. The deviation of $<\lambda_{cr,meas}>$ from $\lambda_{cr,calc}$ is larger than the measurement uncertainty of $<\lambda_{cr,meas}>$. A possible reason for this deviation is that the wavelength is influenced by the finite length of the samples. This is discussed in the next section.
Fig. 5.15 Discontinuity of the ripple wavelength as a function of the applied differential pressure. The dashed lines are visual guides only.

Vlassak et al. [27], Tabata et al. [61], and Baltes et al. [15] have reported Poisson's ratios of thin LPCVD silicon nitride films of 0.28±0.05, 0.35, and 0.13±0.02, respectively. Baltes et al. [15] measured a Poisson's ratio of $\nu = 0.13 \pm 0.07$ for PECVD silicon nitride [15]. All these values were determined using the nonlinear load-deflection behavior of rectangular membranes with different aspect ratios (see Section 2.2). The large scattering of these values between 0.13 and 0.35 gives rise to the question: if Poisson's ratio of silicon nitride films is a strongly process-dependent material property or if this measurement method may lead to large uncertainties. To answer this question further investigations are necessary.

5.6 Wavelength Selection

Finally, the influence of the finite sample length on the determined $\nu$ is discussed. Two observations were made. Firstly, as shown in Fig. 5.2, the ripple is sensitive to end effects and spreads from the sample center. Small deviations of in-plane stresses from the ideal plane strain situation affect the instability transition.
Based on this, a minimum aspect ratio \( b:a \) of 10:1 is recommended. Secondly, we observed jumps in the pressure-dependent \( \lambda \). Fig. 5.15 shows the wavelength as a function of the applied pressure of sample No. 5. Between 18 kPa and 19 kPa, \( \lambda(P) \) is discontinuous and jumps by roughly 2.8 \( \mu \text{m} \) which is about 1.6% of the total wavelength.

As in other mechanical instabilities [120], the finite length of the samples leads to wavelength selection. Fig. 5.16 illustrates this phenomenon. It schematically shows the ripple profile in the center part of a long membrane. The ripple is allowed to develop only in a range of finite length \( L \). Roughly speaking, the discrete wavelengths \( \lambda_n \), which fit into this region, fulfill the condition \( n\lambda_n/2 = L \), where \( n \) is the number of half periods that fit into the buckled region. Thus, the difference \( \Delta \lambda \) of two adjacent wavelengths is

\[
\Delta \lambda = \lambda_n - \lambda_{n+1} = \frac{2L}{n(n + 1)}. \tag{5.27}
\]

In the case of the wavelength jump shown in Fig. 5.15, \( L = 4370 \mu \text{m} \) and \( n = 50 \). The theoretically expected jump is therefore \( \Delta \lambda = 3.4 \mu \text{m} \), which is in reasonable agreement with the observed jump.

To discuss the influence of wavelength selection on the critical wavelength we consider an infinitely long membrane with critical wavelength \( \lambda_{cr} \). If we assume, that a real finite sample with the same material properties buckles with the particular \( \lambda_{select} \) of its discrete wavelengths \( \lambda_n \) that is closest to \( \lambda_{cr} \), the maximum possible deviation of \( \lambda_{select} \) from \( \lambda_{cr} \) is \( \Delta \lambda/2 \). This results in a systematic error in
5.6 Wavelength Selection

Fig. 5.17 Range of $\bar{k}$ for which buckling is energetically advantageous versus $\bar{w}_0/\bar{w}_{cr0}$ of a long membrane with $\bar{\sigma}_0 = -500$ and $\nu = 0.25$. The thick lines show the discrete $\bar{k}$ values allowed by a finite buckling length of $L = \pi a/2$. In this case the plate ripples with $\bar{k}_{select}$ at $\bar{w}_0 = 0.9997\bar{w}_{cr0}$, where $\bar{w}_{cr0}$ is the critical center deflection of an infinitely long sample with the same material parameters, width, and thickness.

the experimentally determined critical wavelength. Therefore, the critical wavelength is not a reliable input parameter for Eqs. 5.21 and 5.22 to compute $\nu$.

In contrast to the critical wavelength, the critical center deflection is virtually unaffected by wavelength selection. As an example, a membrane of finite length with $\nu = 0.25$, $\bar{\sigma}_0 = -500$, and $L = \pi a/2$ is considered. In the experiments described in Section 5.5, $L$ was of the same magnitude at the critical transition. Fig. 5.17 shows the region in the $\bar{k}$-$\bar{w}_0$-plane in which $\det(H) \leq 0$. For a $\bar{k}$ value in this range indicated by the gray color it is energetically advantageous for the membrane to ripple. At the particular center deflection $\bar{w}_0 = \bar{w}_{cr0}$ buckling is only possible with one $\bar{k}$. This is the critical point of an infinitely long membrane with the same material properties as the considered finitely long sample. With decreasing $\bar{w}_0$ the range of possible $\bar{k}$ values, with which the membrane can buckle,
increases. The discrete $k_n = 2n$ of the sample are also shown in Fig. 5.17. The critical center deflection of the finite sample is the largest $w_0$ for which $\det(H) \leq 0$ at one of these $k_n$. In this case, the real critical $w_0$ is 99.97% of the critical center deflection of the corresponding infinitely long membrane.

Consequently, $\overline{w_{cr0}}$ and $\overline{\sigma_0}$ are reliable input parameters for the determination of $v$ using Eqs. 5.21 and 5.22. In contrast, the determination of $v$ using $\overline{k}$ and $\overline{\sigma_0}$ or $\overline{k}$ and $\overline{w_{cr0}}$ as input parameters is an unstable procedure. Within the limits of wavelength selection, the calculated values $\lambda_{cr,calc}$ in Table 5.3 are consistent with the measured averages $\langle \lambda_{cr,meas} \rangle$. 
6 Postbuckling of Square Plates

In this chapter, the buckling of compressively prestressed square plates with built-in edges is experimentally investigated and theoretically analyzed. The buckling depends weakly on Poisson's ratio and is essentially a function of the reduced prestrain $\varepsilon_0$. As $\varepsilon_0$ becomes increasingly negative, two, symmetry breaking, buckling transitions were observed. The bifurcation points of these transitions and buckling profiles are calculated using analytical energy minimization and nonlinear finite-element simulation.

The buckling of micromachined PECVD silicon nitride membranes is interpreted in terms of the theoretical results. Two types of buckled micromachined membranes are investigated. The first of these is the sample type D of Table 2.2. The thickness of the membranes is $1.008 \pm 0.003 \mu m$ and their side lengths is between 601 $\mu m$ and 1980 $\mu m$. Due to the highly compressive residual film stress these membranes develop complicated buckling profiles as shown in Fig. 6.1. Good consistency between measured and calculated buckling profiles is found. The extracted strain values are consistent irrespective of the size and buckling mode of the membranes. The second set of samples are of type B of Table 2.2. Some of them which do not buckle were investigated by bulge testing as described in Chapter 4. Due to a stress gradient of the silicon nitride film across the wafer several type B samples show buckling. Their temperature dependent buckling behavior is analyzed to determine the coefficient of thermal expansion with high accuracy.

Buckling and postbuckling of plates is an important topic in structural engineering and aircraft construction since buckling considerably influences the stability of plate structures and limits the maximum load such structures can carry [124]. However, the buckling of micromachined membranes is in some aspects different from the classical buckling of engineering structures. Firstly, the load causing buckling is due to intrinsic stresses of the membranes and not due to external forces. Secondly, micromachined membranes often have high intrinsic stresses.
which might be some hundred times larger than their critical buckling stress. Due to these stresses the membranes show complicated postbuckling profiles some of which have been documented in literature [125-127]. However, these postbuckling ranges are little of interest for load carrying structures. This may explain why the results of the extensive research on postbuckled plates do not describe the postbuckled micromachined membranes investigated in this chapter.

The buckling of square and rectangular clamped membranes was analyzed using the variational method [54,128]. A brief investigation of weakly postbuckled square plates under uniaxial compression is given in Ref. [129].

The spatial symmetries of experimental buckling profiles are discussed in Section 6.1. Based on this, models to simulate square membrane buckling are developed in Section 6.2. Numerical results for instability transitions and buckling profiles as a function of $\bar{e}_0$ and $\nu$ are presented in Section 6.3. In Section 6.4, these results are compared with experimental buckling profiles to verify the reliability of the models and to determine residual film strains. Section 6.5 describes the extraction of the coefficient of thermal expansion from the temperature dependent buckling of silicon nitride membranes.

### 6.1 Deflection Symmetries

We consider a square plate with side length $a$ as defined in Section 3.1 and schematically shown in Fig. 3.1. As shown in Section 3.2, the buckling of such a plate is only a function of two parameters, namely Poisson’s ratio $\nu$ and the reduced residual strain $\bar{e}_0$. While $\nu$ is a fixed material property, $\bar{e}_0$ can be varied experimentally over a wide range by fabricating membranes with different lateral dimensions or thicknesses. In view of the scaling factor $(a/h)^2$, the reduced prestrain values of the four structures in Fig. 6.1 span more than a decade.

With increasingly negative $\bar{e}_0$, profound changes in the deflection shape are observed. The smallest membrane (Fig. 6.1(a)) has the least negative $\bar{e}_0$ and shows a deflection profile with reflection and rotational symmetries. At more negative reduced prestrains (Figs. 6.1(b) to (d)), the reflection symmetries have disappeared, leaving only the rotational symmetries. The analysis of a large number of such membrane profiles has shown that three $\bar{e}_0$ regions may be distinguished.
6.1 Deflection Symmetries

Fig. 6.1 Optical micrographs of compressively prestrained PECVD silicon nitride membranes with lateral dimensions of (a) 601 µm, (b) 1014 µm, (c) 1425 µm, and (d) 1980 µm. The buckling profile of the smallest membrane shows reflection and rotational symmetries, the other profiles show only rotational symmetries.

The first region is that of tensile or weakly compressive prestrain. It is defined by $\varepsilon_{cr1}(v) < \varepsilon_0$, where $\varepsilon_{cr1}(v) < 0$ is a $v$-dependent critical prestrain. In this region the membrane is stable in the flat position and shows all the symmetries of a
square. These are the symmetries under reflection in the planes \( x = 0, y = 0, \) and \( z = 0 \) (\( \sigma_x, \sigma_y, \) and \( \sigma_z \) symmetries [130]), under rotation through \( \pi/2 \) (\( C_{4z} \) symmetry [130]), and the symmetries under reflection in the planes \( x = \pm y \) resulting from \( \sigma_x, \sigma_y, \) and \( C_{4z} \). The boundary \( \bar{\varepsilon}_{cr1}(v) \) marks a first mechanical instability point below which it is energetically advantageous for the membrane to buckle.

The second region extends from \( \bar{\varepsilon}_{cr1}(v) \) down to \( \bar{\varepsilon}_{cr2}(v) \), i.e., \( \bar{\varepsilon}_{cr2}(v) < \bar{\varepsilon} < \bar{\varepsilon}_{cr1}(v) \). Here, the membrane can assume two stable buckling modes: one upward, the other downward. Their symmetries are \( \sigma_x, \sigma_y, \) and \( C_{4z} \), while \( \sigma_z \) has vanished. In terms of the displacement fields, \( \sigma_x, \sigma_y, \) and \( C_{4z} \) mean

\[
\begin{bmatrix}
  u_x(x, y) \\
  u_y(x, y) \\
  w(x, y)
\end{bmatrix}
= \begin{bmatrix}
  -u_x(-x, y) \\
  u_y(-x, y) \\
  w(-x, y)
\end{bmatrix}
= \begin{bmatrix}
  u_x(x, -y) \\
  -u_y(x, -y) \\
  w(x, -y)
\end{bmatrix}
\]  

(6.1)

and

\[
\begin{bmatrix}
  u_x(x, y) \\
  u_y(x, y) \\
  w(x, y)
\end{bmatrix}
= \begin{bmatrix}
  u_y(-x, y) \\
  -u_x(-x, y) \\
  w(-y, x)
\end{bmatrix},
\]

(6.2)

respectively. The critical reduced prestrain \( \bar{\varepsilon}_{cr2}(v) \) marks the onset of a second mechanical instability.

Below \( \bar{\varepsilon}_{cr2}(v) \), the membrane minimizes its strain energy by choosing an even less symmetric displacement, by sacrificing \( \sigma_x \) and \( \sigma_y \). The displacement field can then be separated into two parts, both \( C_{4z} \) symmetric. The first is a displacement with \( \sigma_x \) and \( \sigma_y \) symmetries, while the second is skew symmetric with respect to reflections at the \( x \) and \( y \) axes, i.e.,

\[
\begin{bmatrix}
  u_x(x, y) \\
  u_y(x, y) \\
  w(x, y)
\end{bmatrix}
= \begin{bmatrix}
  u_x(-x, y) \\
  -u_y(-x, y) \\
  -w(-x, y)
\end{bmatrix}
= \begin{bmatrix}
  -u_x(x, -y) \\
  u_y(x, -y) \\
  -w(x, -y)
\end{bmatrix}.
\]  

(6.3)
6.2 Models

The observations described in the previous section are now mathematically analyzed. Firstly, membrane deflections are computed using an energy minimization method (EMM) based on the Ritz method. Secondly, finite element simulations (FES) are carried out in order to provide a complementary method and independent test.

Energy Minimization Method

The reduced elastic energy \( \bar{U}_{tot} = \bar{U}_b + \bar{U}_0 \) of a buckled square plate is given by Eqs. 3.17 and 3.19 in terms of the reduced quantities defined in Table 3.1. These equations can be simplified by integration by parts with use of the \( C_4 \)-symmetry and the boundary conditions (Eq. 3.1). Without terms independent of the displacement field the energies \( \bar{U}_b \) and \( \bar{U}_0 \) then read

\[
\bar{U}_b = \frac{1}{12} \int_{-1/2}^{1/2} \int_{-1/2}^{1/2} \left[ \left( \frac{\partial^2 \bar{\varphi}}{\partial \bar{x}^2} \right)^2 + \frac{\partial^2 \bar{\varphi}}{\partial \bar{y}^2} \right] d\bar{x} d\bar{y} \tag{6.4}
\]

and

\[
\bar{U}_0 = \frac{1}{2} \int_{-1/2}^{1/2} \int_{-1/2}^{1/2} \left\{ 2 \left( \frac{\partial \bar{u}_x}{\partial \bar{x}} \right)^2 + (1 - \nu) \left( \frac{\partial \bar{u}_x}{\partial \bar{y}} \right)^2 + (1 + \nu) \frac{\partial \bar{u}_x}{\partial \bar{y}} \frac{\partial \bar{u}_y}{\partial \bar{y}} + \frac{\partial^2 \bar{w}}{\partial \bar{x}^2} \right\} d\bar{x} d\bar{y} \tag{6.5}
\]
The variational space for the out-of-plane displacement \( \bar{w}(\bar{x}, \bar{y}) \) is defined as follows. A set of trial functions \( g_{mn}^s(\bar{x}, \bar{y}) \) with \( \sigma_x, \sigma_y \), and \( C_{4z} \) symmetry is defined as

\[
g_{mn}^s(\bar{x}, \bar{y}) = (\cos 2m\pi \bar{x} - (-1)^m)(\cos 2n\pi \bar{y} - (-1)^n) + (\cos 2n\pi \bar{x} - (-1)^n)(\cos 2m\pi \bar{y} - (-1)^m)
\]

with \( n \geq m \). Similarly,

\[
g_{mn}^a(\bar{x}, \bar{y}) = \left[ \sin((2m + 1)\pi \bar{x}) - (-1)^m \sin(\pi \bar{x}) \right] \times \left[ \sin((2n + 1)\pi \bar{y}) - (-1)^n \sin(\pi \bar{y}) \right] - \left[ \sin((2n + 1)\pi \bar{x}) - (-1)^n \sin(\pi \bar{x}) \right] \times \left[ \sin((2m + 1)\pi \bar{y}) - (-1)^m \sin(\pi \bar{y}) \right]
\]

with \( n > m \) constitute a base for the deflections with \( C_{4z} \) symmetry and skew reflection symmetries. The out-of-plane displacement is written as a truncated sum of these trial function, viz.,

\[
\bar{w}(\bar{x}, \bar{y}) = \sum_{m=1, n=m}^{m_{\max}} w_{mn}^s g_{mn}^s(\bar{x}, \bar{y}) + \sum_{m=1, n=m+1}^{m_{\max}} w_{mn}^a g_{mn}^a(\bar{x}, \bar{y}).
\]

When truncated at \( m_{\max} \), \( \bar{w} \) consists of a total of \( m_{\max}^2 \) terms, with the same number of variational parameters \( w_{mn}^s \) and \( w_{mn}^a \).

Analogously, the in-plane displacements \( \bar{u}_x \) and \( \bar{u}_y \) are expanded as

\[
\bar{u}_x(\bar{x}, \bar{y}) \equiv \bar{u}_y(-\bar{y}, \bar{x}) = \sum_{p, q = 1}^{p_{\max}} u_{pq}^s f_{pq}(\bar{x}, \bar{y}) + \sum_{p, q = 1}^{p_{\max}} u_{pq}^a f_{pq}(\bar{y}, \bar{x})
\]

in terms of the trial functions

\[
f_{pq}(\bar{x}, \bar{y}) = \sin(2p\pi \bar{x}) \times \cos((2q-1)\pi \bar{y})
\]
with variational parameters $u_{pq}^s$ and $u_{pq}^a$. In view of their constituent functions, $w$, $u_x$, and $u_y$, all satisfy the required boundary conditions. The sets of coefficients $w_{mn}^s$, $w_{mn}^a$, $u_{pq}^s$, and $u_{pq}^a$ are $m_{max}^2 + 2p_{max}^s$ variational parameters available for the minimization of the energy and thus for the approximation of the buckling deformation.

The total energy $\bar{U}_{tot} = \bar{U}_b + \bar{U}_0$ is obtained by inserting Eqs. 6.8 and 6.9 into Eqs. 6.4 and 6.5. In view of Eqs. 6.4 and 6.5, $\bar{U}_{tot}$ is a polynomial with terms up to second order in $u_{pq}^s$ and $u_{pq}^a$, and fourth order in $w_{mn}^s$ and $w_{mn}^a$. The coefficients $w_{mn}^s$, $w_{mn}^a$, $u_{pq}^s$, and $u_{pq}^a$ are determined by the condition that $\bar{U}_{tot}$ is minimal with respect to them. In the case of the in-plane coefficients $u_{pq}^s$ and $u_{pq}^a$ this means

$$\frac{\partial}{\partial u_{pq}^s} \bar{U}_0 = \frac{\partial}{\partial u_{pq}^a} \bar{U}_0 = 0.$$  \hspace{1cm} (6.11)

This is a system of equations linear in $u_{pq}^s$ and $u_{pq}^a$. Solving for $u_{pq}^s$ and $u_{pq}^a$ shows that they are sums of terms quadratic in $w_{mn}^s$ and $w_{mn}^a$. This has three implications: firstly, the out-of-plane deflection unambiguously defines the in-plane displacements; secondly, the in-plane displacements grows quadratically with the out-of-plane deflection; finally, upward and corresponding downward deflections have identical in-plane deformations.

Substitution of $u_{pq}$ in $\bar{U}_{tot}$ yields

$$\bar{U}_{tot}(\vec{e}_0, \nu) = M_{ijkl}(\vec{e}_0, \nu)w_{ijkl}^s + M_{ijkl}(\vec{e}_0, \nu)w_{ijkl}^a + T_{ijklmns}(\nu)w_{ijkl}^sw_{mn}^sw_{rs} + T_{ijklmns}(\nu)w_{ijkl}^aw_{mn}^aw_{rs} + T_{ijklmns}(\nu)w_{ijkl}^aw_{mn}^aw_{rs}$$ \hspace{1cm} (6.12)

where the indices of the coefficients $w_{pq}^s$ and $w_{pq}^a$ are restricted to $p \leq q$ and $p < q$, respectively. This is a polynomial with quadratic and quartic terms in the out-of-plane variational parameters $w_{mn}^s$ and $w_{mn}^a$. Its coefficients are given by the elements of multidimensional matrices $M^s$, $M^a$, $T^s$, $T^a$, and $T^{aa}$. For fixed $\vec{e}_0$ and $\nu$, the stable deflection profiles are defined by those sets of $w_{mn}^s$ and $w_{mn}^a$ minimizing $\bar{U}_{tot}$. The global minimum was found using the conjugated gradient
method [131]. The computational time to calculate $\bar{U}_{tot}$ increases with the fourth power of the number of out-of-plane trial functions. Truncation at $m_{\text{max}} = 8$ therefore limited the number of $w^{s}_{mn}$ and $w^{a}_{mn}$ to 36 and 28, respectively. With this expansion, $\bar{U}_{tot}$ consists of roughly $1.3 \times 10^7$ non zero products and more than $3 \times 10^6$ sums. Since the number of in-plane functions only weakly increases the total computational time, we systematically chose $p_{\text{max}} = 2m_{\text{max}}$. The accuracy of the result was then limited by the number of out-of-plane functions.

Finite-Element Simulations

Nonlinear finite-element simulations were performed using ANSYS® 5.4. The plate was meshed uniformly with elements of type Shell 63. These 4-node shells have three translational and three rotational degrees of freedom at each node. Clamped edges were implemented as boundary conditions. For the side length $a$ and thickness $h$ of the plate we chose $a = 1000 \ \mu m$ and $h = 1 \ \mu m$. Young's modulus was set to $E = 100 \ \text{GPa}$. Poisson's ratio $\nu$ was varied between 0 and 0.49. To ensure the proper convergence of the solution, the total number of elements was varied from $10 \times 10$ to $80 \times 80$.

In-plane residual strains were simulated thermally. For this purpose a coefficient of thermal expansion $\alpha = 10^{-5} \ \text{K}^{-1}$ was assigned to the material of the plate and the temperature of the plate was changed stepwise while the plate remained constrained by the fixed edges. The thermal thin film stress resulting from a temperature increase $\Delta T$ is $\sigma_0 = -\Delta T E \alpha / (1 - \nu)$. The corresponding residual strain is $\epsilon_0 = -\Delta T \alpha$.

The plate was brought into the buckling mode by the application of thermal and external loads. To avoid numerical instabilities at the bifurcation points $\epsilon_{cr1}$ and $\epsilon_{cr2}$, these were bypassed by the procedure outlined in Fig. 6.2. This diagram schematically shows the center deflection of the plate as a function of the residual strain $\epsilon_0$ and an externally applied differential pressure $P$. The deflection of the plate at a strain $\epsilon'_0$ between $\epsilon_{cr1}$ and $\epsilon_{cr2}$ was reached by a three-step procedure. Firstly, the plate was gradually loaded with a differential pressure $P = 200 \ \text{Pa}$. Secondly, the thermal prestrain was decreased from zero to $\epsilon'_0$. Thirdly, the differential pressure was unloaded step by step to let the plate settle into its buckling mode at $\epsilon'_0$. Similarly, the second bifurcation point at $\epsilon_{cr2}$ was bypassed by the
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Fig. 6.2  Loading sequence used in FE simulations to avoid numerical instabilities at the critical points $\varepsilon_{crj}$ and $\varepsilon_{cr1}$. The diagram schematically shows the center deflection $w_0$ of a square plate as a function of the differential pressure $P$ and the residual strain $\varepsilon_0$ close to $\varepsilon_{crj}$.

application of an appropriate asymmetric load that enabled the plate to settle in profiles without $\sigma_x$ and $\sigma_y$ symmetry.

Note that the numerical results obtained for this particular plate are translated straightforwardly into those of plates with other geometrical or material parameters, by applying the scaling laws defined in Section 3.2.

6.3 Numerical Results

Comparison of EMM and FES

The convergence of both numerical methods was investigated. As an example, Figure 6.3 shows the convergence of the center deflections $\bar{w}_{0,EMM}$ and $\bar{w}_{0,FES}$ calculated using the EMM and FES, respectively. The reduced residual strain is -175 and Poisson’s ratio is 0.25. The EMM deflection shows an oscillatory con-
Fig. 6.3 Convergence of two models of square buckled plate. The reduced center deflection \( \bar{w}_0 \) of the buckling profile was computed for \( \nu = 0.25 \) and \( \bar{\varepsilon}_0 = -175 \) as a function of (a) the number of shell elements in the FES and (b) the number of out-of-plane trial functions \( g_{nm}^s \) in the EMM. The solid lines are visual guides.

Convergence with increasing number of trial function; in contrast, \( \bar{w}_{0,\text{FES}} \) decreases monotonically with growing number of shell elements.

To check the consistency of the two methods, we calculated the relative difference \( \Delta \bar{w}_0 = (\bar{w}_{0,\text{FES}} - \bar{w}_{0,\text{EMM}})/\bar{w}_{0,\text{EMM}} \) over the prestrain range \(-800 < \bar{\varepsilon}_0 < \bar{\varepsilon}_{cr1} \) and for \( \nu = 0, 0.25, 0.49 \). The number of out-of-plane trial functions and shell elements used to compute \( \Delta \bar{w}_0 \) were 64 and 6400, respectively. At small reduced residual strains \(-250 < \bar{\varepsilon}_0 < \bar{\varepsilon}_{cr1} \) the center deflections are highly consistent. For all \( \nu \)-values, \( |\Delta \bar{w}_0| \) is smaller than 0.3%. \( |\Delta \bar{w}_0| \) increases towards higher negative residual strains. For \( \nu = 0.25 \), \( \Delta \bar{w}_0 \) is between -0.2% and +0.35%. For the other \( \nu \)-values, \( \Delta \bar{w}_0 \) is significantly larger: the maximum values of \( \Delta \bar{w}_0 \) are +1.15% and
6.3 Numerical Results

-0.8% at $\{\varepsilon_0, v\} = \{-800, 0\}$ and $\{-600, 0.49\}$, respectively. The tendency of $\Delta w_0$ to increase towards more negative residual strains can be explained by two reasons. Firstly, as $\varepsilon_0$ becomes more negative, the buckling profile folds up more and more. Since the truncation of the sum in Eq. 6.8 limits the number of spatial periods of the out-of-plane displacement functions, strongly folded buckling profiles are less well modeled by the EMM. On the other hand, the discontinuity of the derivatives of the finite element shape functions at the element boundaries may also lead to an increasing error with increasing complexity of the buckling profile.

**Instability Transitions**

Figure 6.4 shows the first few expansion coefficients of the two sums in Eq. 6.8 as a function of $\varepsilon_0$ for $v = 0.25$. In Fig. 6.4(a) coefficients $w^s_{mn}$ are plotted against $\varepsilon_0$. Above a clearly defined critical prestrain $\varepsilon_{cr1}$, the coefficients are zero, i.e., the plate is at rest in the flat position. A closer inspection shows that below $\varepsilon_{cr1}$ the squares of all coefficients are to first order linear in $(\varepsilon_{cr1} - \varepsilon_0)$. This means that the emergence of the buckling profile is proportional to $(\varepsilon_{cr1} - \varepsilon_0)^{1/2}$. Note that in this region all coefficients $w^a_{mn}$ are zero, i.e., the buckling profile has all the reflection and rotational symmetries of the trial functions $g^s_{mn}$.

A similar behavior was found with the coefficients $w^a_{mn}$, the first three of which are shown in Fig. 6.4(b). The coefficients $w^a_{mn}$ are zero at strains less negative than a critical prestrain $\varepsilon_{cr2}$. Their squares are proportional to $\varepsilon_{cr2} - \varepsilon_0$ for $\varepsilon_0 < \varepsilon_{cr2}$. This means that the contribution of trial functions $g^a_{mn}$ to the profile emerges as $(\varepsilon_{cr2} - \varepsilon_0)^{1/2}$, which again is the clear signature of a mechanical instability transition. The appearance of $g^a_{mn}$ below $\varepsilon_{cr2}$ also influences the symmetric part of the profile: the derivatives of $w^s_{mn}$ with respect to $\varepsilon_0$ are discontinuous at $\varepsilon_{cr2}$.

The first transition point, $\varepsilon_{cr1}$, can be calculated using $U_{tot}$ as defined in Eq. 6.12. In fact, $\varepsilon_{cr1}$ is the prestrain at which $U_{tot}$ ceases to be a minimum in the undeflected plate state $w^s_{mn} = w^a_{mn} = 0$ and becomes a saddle point. This means, that at $\varepsilon_{cr1}$ the lowest eigenvalue of the Hessian of the total energy, $H(U_{tot})$, evaluated at $w^s_{mn} = w^a_{mn} = 0$, is equal to zero. As a consequence the determinant of $H(U_{tot})$ vanishes at $\varepsilon_{cr1}$. Solving $\det[H(U_{tot}(\varepsilon_0, v))] = 0$ for $\varepsilon_0$ leads to
Fig. 6.4 Expansion coefficients of buckling profile calculated using the EMM, as a function of the reduced residual strain $\bar{\varepsilon}_0$ with $\nu = 0.25$: (a) $w_{11}^s$, $w_{12}^s$, and $w_{22}^s$ close to $\bar{\varepsilon}_{crl}$; (b) $w_{11}^o$, $w_{13}^o$, and $w_{23}^o$ close to $\bar{\varepsilon}_{cr2}$.
6.3 Numerical Results

The second transition was determined using FES. Figure 6.5 shows $\tilde{\varepsilon}_{cr2}$ for several $\nu$ values as a function of the number of shell elements in the FE-model. As is obvious from the slow convergence of $\tilde{\varepsilon}_{cr2}$, at least 6400 elements are necessary to compute $\tilde{\varepsilon}_{cr2}$ with an uncertainty smaller than 1%. Fig. 6.5 also shows that $\tilde{\varepsilon}_{cr2}$ is only weakly influenced by Poisson’s ratio. Whatever the value of $\nu$, $\tilde{\varepsilon}_{cr2}$ is between -206 and -226. Materials with $\nu = 0.25$ have least negative critical strain $\tilde{\varepsilon}_{cr2}$; most negative $\tilde{\varepsilon}_{cr2}$ values are found for $\nu = 0$ and $\nu = 0.49$.

\[ \tilde{\varepsilon}_{cr1}(\nu) = \frac{4.363}{1 + \nu}, \]  

This is in good agreement with the approximated solution $\tilde{\varepsilon}_{cr1} = -4.386/(1+\nu)$ reported in [54]. The small discrepancy results from the neglect of contributions to the buckling profile other than $g_{11}$ in [54].

Fig. 6.5 Second critical strain $\tilde{\varepsilon}_{cr2}(\nu)$ approximated using FES, as a function of the number of shell elements and for various $\nu$ values. The solid lines are visual guides.
Buckling Profiles

As the prestrain $\overline{e}_0$ is lowered, the buckling profiles undergo profound changes; they are increasingly folded and become less and less symmetric. Extended numerical experiments have shown that the profiles are only weakly influenced by Poisson's ratio, except close to $\overline{e}_{cr1}(v)$. For this reason, the following discussion focuses only on one Poisson's ratio, namely $v = 0.25$.

Figure 6.7 shows buckling profiles calculated using the EMM. The first profile corresponds to $\overline{e}_0 = 2 \overline{e}_{cr1}$. It is dominated by the out-of-plane function $g_{11}(\tilde{x}, \tilde{y}) = 2(\cos 2\pi \tilde{x} + 1)(\cos 2\pi \tilde{y} + 1)$. This is just the buckling shape assumed in previous models of the buckling of square plates [54, 128]. At more negative prestrains the plate folds up more strongly, as shown in Fig. 6.7(b) corresponding to $\overline{e}_0 = -200$ which is close to $\overline{e}_{cr2}$. Characteristic structures appear, such as depressions at the corners and valleys at the middle of the edges. At still more negative residual strains, the reflection symmetries $\sigma_x$ and $\sigma_y$ are broken and only the rotational symmetry $C_{4z}$ remains. As an example, Fig. 6.7(c) shows the buckling profile at $\overline{e}_0 = 2 \overline{e}_{cr2}$.

Fig. 6.7 gives a more detailed account of how buckling profiles evolve as a function of $\overline{e}_0$. Profiles along $\tilde{y} = 0$ ($\tilde{x}$ axis), $\tilde{y} = \tilde{x}$ (diagonal) and $\tilde{y} = 1/4$ are shown. The reduced residual strains range from -10.8 to -769.5 and lead to reduced deflections $\overline{w}_0 = 2, 4, 6, \ldots, 18$. Apart from their different amplitudes the $\tilde{y} = 0$ profiles changes weakly with $\overline{e}_0$. In contrast, the $\tilde{y} = \tilde{x}$ and $\tilde{y} = 1/4$ profiles undergo profound changes. At low residual strain both decrease monotonically from the center to the edge of the plate. With increasingly negative residual strain, however, a succession of local minima appears along the diagonal. Similarly, at sufficiently negative prestrain, a symmetric local minimum first develops in the middle of the $\tilde{y} = 1/4$ profiles. At prestrains $\overline{e}_0 < \overline{e}_{cr2}$, these profiles are asymmetric due to the loss of the reflection symmetries at $\overline{e}_{cr2}$.

Center Deflection

A point of special practical interest is the center deflection $w_0$ of the deflected plates since it can be measured straightforwardly and accurately. Figure 6.8 shows $\overline{w}_0$ as a function of $\overline{e}_0$ for $v = 0, 0.25,$ and 0.49, as computed with FES. At the onset of the buckling, $\overline{w}_0$ first increases as $\{\overline{e}_{cr1}(v) - \overline{e}_0\}^{1/2}$, continues to increase strongly with $|\overline{e}_0|$, and varies weakly with $v$. 
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Fig. 6.6 Buckling profiles calculated using the EMM with $v = 0.25$ and $\varepsilon_0 = 2 \varepsilon_{cr1}$ (a), $\varepsilon_0 = -200$ (b), and $\varepsilon_0 = 2 \varepsilon_{cr2}$ (c). Profiles (a) and (b) have $\sigma_x$, $\sigma_y$, and $C_{4z}$ symmetries, while profile (c) has only the $C_{4z}$ symmetry.
Fig. 6.7 Profiles of buckled square plates along $\bar{y} = 0$ (x-axis), $\bar{y} = \bar{x}$ (diagonal) and $\bar{y} = 1/4$ for the sequence of reduced center deflections $\bar{w}_0 = 2, 4, 6, ..., 18$, resulting from the reduced residual strains listed in the legend. The profiles were calculated using FES with $v = 0.25$. 

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6.3 Numerical Results

![Graph showing reduced center deflection w_0 as a function of the reduced prestrain \( \bar{\varepsilon}_0 \), for \( v = 0, 0.25, \) and \( 0.49 \). The values were calculated using FES with 80x80 shell elements.]

Experimental results can in principle be evaluated using one of the two methods described in Section 6.2, which establish a relation between the deflection \( \bar{w}_0 \) of a plate and the parameters \( \bar{\varepsilon}_0 \) and \( v \). However, implementing the variational calculation or FE simulation for a particular plate case is admittedly a time-consuming task. For this reason, to provide an efficient method of calculating \( \bar{w}_0 \) as a function of \( \bar{\varepsilon}_0 \) and \( v \), we carried out extensive simulations with the EMM in the ranges \( \bar{\varepsilon}_{cr2} \leq \bar{\varepsilon}_0 \leq 0 \) and \( 0 \leq v \leq 0.5 \). From roughly 1000 simulations covering this two-dimensional parameter range, we extracted the fit function

\[
\bar{w}_{0,fit} = \sqrt{\Delta \bar{\varepsilon}_0 \left[ c_1 + c_2 \tanh \{ c_3 \Delta \bar{\varepsilon}_0 \} + \frac{c_4 \Delta \bar{\varepsilon}_0 + c_5 \Delta \bar{\varepsilon}_0^2}{1 - c_6 \Delta \bar{\varepsilon}_0^3} \right]} \tag{6.14}
\]

where \( \Delta \bar{\varepsilon}_0(v) = \bar{\varepsilon}_0 - \bar{\varepsilon}_{cr1}(v) \) with \( \bar{\varepsilon}_{cr1}(v) \) defined by Eq. 6.13. The \( v \)-dependent coefficients \( c_1 \) to \( c_6 \) are given by
The approximate center deflection \( \bar{w}_{0\text{fit}} \) matches the true deflection \( \bar{w}_0 \) as given by the EMM with an uncertainty smaller than 0.5% over the entire simulation range.

### 6.4 Comparison with Experiments

In this section, measured profiles of buckled micromachined membranes of type D (see Table 2.2) with lateral dimensions between 601 \( \mu \text{m} \) and 1425 \( \mu \text{m} \) are analyzed using the results of the previous sections. Residual strain and Young's modulus are extracted from these structures.

Profiles of buckled membranes were measured along the \( \bar{y} = 0 \) (x-axis), \( \bar{y} = \bar{x} \) (diagonal) and \( \bar{y} = 1/4 \). At the membrane edges, the measured profiles were distorted by interferences due to the silicon substrate. These distortions were eliminated by subtracting profiles measured in the upward and downward deflected state. Brief differential pressure pulses enabled switching between these states.

Fig. 6.9 shows profiles of four membranes with side lengths of 601 \( \mu \text{m} \), 841 \( \mu \text{m} \), 1014 \( \mu \text{m} \), and 1301 \( \mu \text{m} \). For comparison, profiles simulated using finite elements are also shown. For these simulations, \( v \) was set to 0.25. This value is consistent with the value 0.254±0.022 measured on the samples of type C (see Table 6.2) as described in Section 5.4. These samples are identically processed but have a different film thickness. The residual strain was adjusted to let the center deflection of simulated and measured profiles coincide. Excellent agreement between experiment and simulation in terms of deflection symmetries, existence and position of local extrema, and deflection amplitudes was obtained. Thus the numerical models of Section 6.2 are well suited to quantitatively model the buckling
6.4 Comparison with Experiments

Fig. 6.9 Comparison of measured (solid lines) and simulated (dashed lines) buckling profiles. Profiles of membranes Nos. 1, 2, 3, and 6 in Table 1 were recorded along (a) $\tilde{y} = 0$, (b) $\tilde{y} = \bar{x}$, and (c) $\tilde{y} = 1/4$. FE simulations were performed with $\nu = 0.25$. 
Table 6.1 Measured center deflections $w_0$, extracted reduced and physical residual strains $\bar{\varepsilon}_0$ and $\varepsilon_0$, and symmetries of buckled square membranes with various side lengths $a$. Membranes Nos. 1, 3, and 7 are shown in Figs. 6.1(a), (b), and (c), respectively. Profiles of membranes Nos. 1, 2, 3, and 6 are shown in Fig. 6.9.

<table>
<thead>
<tr>
<th>No.</th>
<th>$a$ [µm]</th>
<th>$w_0$ [µm]</th>
<th>$\bar{\varepsilon}_0$</th>
<th>Symmetry</th>
<th>$\varepsilon_0 [10^{-4}]$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>601</td>
<td>7.76</td>
<td>-129.6</td>
<td>$\sigma_x$, $\sigma_y$, $C_4$</td>
<td>-3.67</td>
</tr>
<tr>
<td>2</td>
<td>841</td>
<td>10.72</td>
<td>-254.8</td>
<td>$C_4$</td>
<td>-3.69</td>
</tr>
<tr>
<td>3</td>
<td>1014</td>
<td>12.59</td>
<td>-358.6</td>
<td>$C_4$</td>
<td>-3.57</td>
</tr>
<tr>
<td>4</td>
<td>1170</td>
<td>14.84</td>
<td>-505.0</td>
<td>$C_4$</td>
<td>-3.77</td>
</tr>
<tr>
<td>5</td>
<td>1171</td>
<td>14.51</td>
<td>-482.1</td>
<td>$C_4$</td>
<td>-3.60</td>
</tr>
<tr>
<td>6</td>
<td>1301</td>
<td>16.06</td>
<td>-593.8</td>
<td>$C_4$</td>
<td>-3.59</td>
</tr>
<tr>
<td>7</td>
<td>1425</td>
<td>17.58</td>
<td>-715.8</td>
<td>$C_4$</td>
<td>-3.61</td>
</tr>
<tr>
<td>8</td>
<td>1423</td>
<td>17.85</td>
<td>-738.0</td>
<td>$C_4$</td>
<td>-3.73</td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$-3.65 \pm 0.07$</td>
</tr>
</tbody>
</table>

behavior of square membranes. They can be used for the reliable determination of mechanical thin film properties using such structures.

Residual strain values were extracted from seven membranes with side lengths between 601 µm and 1425 µm. The membranes were located in an small area of 36×24 mm² on the wafer to minimize the influence of a possible strain gradient across the wafer on the homogeneity of the measured strain values. Results are shown in Table 6.1. The residual strain was derived from the measured center deflection. A further input was $\nu = 0.25$. The evaluated reduced residual strain values $\bar{\varepsilon}_0$ cover the wide range between -131 and -612, due to the quadratic dependence of $\bar{\varepsilon}_0$ on $a$. The physical residual strain values $\varepsilon_0 = \bar{\varepsilon}_0 (h/a)^2$ are all in excellent agreement, despite the large $\bar{\varepsilon}_0$ range and the different buckling modes and symmetries. The average prestrain is $-(3.65\pm0.07)\times10^{-4}$. It is noteworthy that
the extracted film prestrain only weakly depends on thickness \( h \): an error in \( h \) of 1\% in the case of membrane No. 4 gives rise to an error of only 0.06\% in \( \varepsilon_0 \).

To obtain Young’s modulus of the PECVD silicon nitride, the film stress was determined using the wafer curvature technique (see Section 2.2). Curvatures were measured along two orthogonal directions of a second wafer covered with an identically processed PECVD silicon nitride. Using Stoney’s formula (Eq. 2.20) a stress of \( \sigma_0 = -60.7 \pm 1 \text{ MPa} \) was found. To determine the average residual strain of the nitride film the center deflection of 33 membranes spread over the wafer were measured. The determined strain values were between \(-3.0 \times 10^{-4}\) and \(-4.1 \times 10^{-4}\) showing a roughly linear dependence on the membrane position. The average film strain was calculated to be \( \varepsilon_{0, \text{ave}} = -3.50 \times 10^{-4} \). Using this value and \( v = 0.25 \), a Young’s modulus of \( E = (1-v)\sigma_0/\varepsilon_{0, \text{ave}} = 130.0 \text{ GPa} \) was obtained. A slightly higher value \( E = 133.6 \text{ GPa} \) was determined for the same silicon nitride by bulge testing as described in Section 4.4.

Finally, the influence of the neglected Au layer on the extracted residual strain is discussed. We assume the Au layer to have the elastic properties of cast bulk material, i.e., \( E = 74.4 \text{ GPa} \) and \( v = 0.42 \) [132]. Changes of the flexural rigidity and effective biaxial modulus due to the inclusion of the Au layer were calculated to be 0.3\% and 0.1\%, respectively. The influence of the residual strain of the Au layer is estimated by introducing an average effective residual strain

\[
\varepsilon_{\text{eff}} = \frac{\varepsilon_{0, \text{SiN}} h_{\text{SiN}} E_{b, \text{SiN}} + \varepsilon_{0, \text{Au}} h_{\text{Au}} E_{b, \text{Au}}}{h_{\text{SiN}} E_{b, \text{SiN}} + h_{\text{Au}} E_{b, \text{Au}}},
\]

where the indices refer to the respective materials. The residual strain value extracted from experimental membrane deflections is \( \varepsilon_{\text{eff}} \) instead of \( \varepsilon_{0, \text{SiN}} \). With a residual stress \( \sigma_0 \) of the Au layer assumed to be somewhere between -300 MPa and 300 MPa [133] the error of \( \varepsilon_{0, \text{SiN}} \) introduced by the neglect of the Au layer is smaller than 1.5\%.

### 6.5 Measurement of Thermal Expansion

In this section, measurements of the coefficient of thermal expansion of a silicon nitride thin film are presented. The thermal expansion was extracted from the
temperature dependent center deflection of buckled membranes. Four square membranes of type C (see Table 2.2) were investigated.

The chips that contain the membranes were mounted into the temperature chamber described in Section 2.4. The temperature of the unit was controlled between room temperature and 140°C. At 140°C all membranes were flat. Buckling profiles were recorded at stabilized chip temperatures. Between successive profile measurements the chamber was purged with dry nitrogen to prevent excessive oxidation of the Ti layer. However, after the first temperature cycle of the membranes we observed a change in the surface reflectivity. Also, the center deflections at room temperature changed slightly between 0.2 μm and 0.4 μm. This is due to the oxidation of the Ti layer. During following temperature cycles no further changes were observed. Therefore, all membranes were once ramped up to 140°C before the first measurement was performed.

Fig. 6.10(a) shows $w_0$ versus temperature for sample No. 1. The center deflection of the membrane decreases with increasing temperature since the coefficient of thermal expansion of the nitride film is lower than that of silicon. The residual strain $\varepsilon_0(T)$ of the membrane was then computed for each measured $w_0$ using Eq. 6.15 and $h = 3.546 \mu m$. For Poisson's ratio a value of 0.254 was assumed because this value was determined for the similarly processed nitride samples of type C (see Table 2.2) as described in Section 5.5. The only difference in the deposition condition is that the samples of type C were deposited with 60% LF power to the total power while the samples characterized in this section were deposited with 55% LF power. According to Eq. 2.18 the relative thermal expansion $\Delta l/l$ of the film with respect to the room temperature $T_0$ is given by

$$\frac{\Delta l(T)}{l(T_0)} = \int_{T_0}^{T} \alpha_f(T')dT' = \varepsilon_0(T_0) - \varepsilon_0(T) + \int_{T_0}^{T} \alpha_{Si}(T')dT'. \quad (6.17)$$

The integral on the right hand side was calculated using Eq. 2.19. Fig. 6.10(b) shows the relative length change of the film determined using sample No. 1.

To determine the coefficient of thermal expansion of the silicon nitride we assume that the 15 nm-thin titanium layer on top of the nitride has the same in-plane elastic properties as the silicon nitride. The total thickness used to calculated the film strain was taken as the sum of the nitride and titanium thicknesses.
6.5 Measurement of Thermal Expansion

Fig. 6.10 (a) Measured center deflection $w_0$ of sample No. 1 of Table 6.2 as a function of temperature. (b) Relative thermal expansion of the PECVD silicon nitride with respect to room temperature (21.5°C), extracted from the data in (a). The solid line is a second order polynomial fit to the experimental data.

This assumption is justifiable since isotropic bulk titanium has a biaxial modulus of 161 GPa [134]. In comparison, the measured plane strain modulus of the nitride layer is 134 GPa (see Section 4.4) which results in a biaxial modulus of 168 GPa if $\nu$ is set equal to 0.25. We further assumed that the Ti layer has the same coefficient of thermal expansion as polycrystalline bulk titanium which is $\alpha_{Ti} = 8.6 \times 10^{-6} \, \text{K}^{-1}$ at 25°C [135]. Since $\alpha_{Ti}$ changes weakly with temperature its temperature dependence is neglected. The thermal expansion of the film sandwich consisting of 3.531 μm silicon nitride and 0.015 μm titanium is therefore

$$\alpha_f = \frac{h_{SiN} \alpha_{SiN} + h_{Ti} \alpha_{Ti}}{h_{SiN} + h_{Ti}}. \quad (6.18)$$

Note, that $\alpha_{SiN} = \alpha_f$ since $h_{Ti}/h_{SiN} = 0.004$. To obtain $\alpha_f$ a second order polynomial was fitted to $\Delta l/l(T)$. The derivative of this polynomial with respect to $T$ is $\alpha_f(T)$. Finally $\alpha_{SiN} = \alpha_0 + \alpha_1 (T-T_0)$, where $\alpha_0$ and $\alpha_1$ denote the linear and quadratic expansion coefficients, respectively, and $T_0 = 25^\circ \text{C}$, was calculated using Eq. 6.18. Table 6.2 shows these coefficients evaluated for four samples. The coef-
Table 6.2 Width $a$, center deflection $w_0$ at room temperature, and the extracted linear and quadratic coefficient of thermal expansion.

<table>
<thead>
<tr>
<th>No.</th>
<th>$a$ [$\mu$m]</th>
<th>$w_0$ [$\mu$m]</th>
<th>$\alpha_0$ [$10^{-6}$ K$^{-1}$]</th>
<th>$\alpha_1$ [$10^{-9}$ K$^{-2}$]</th>
</tr>
</thead>
</table>
|     |              | ($T = 21.5^\circ$C) | ($T = 25^\circ$C) | ($T = 25^\circ$C) |}
| 1   | 2621         | 16.52           | 1.794                         | 7.59                          |
| 2   | 2623         | 17.53           | 1.806                         | 7.32                          |
| 3   | 2622         | 14.21           | 1.809                         | 8.03                          |
| 4   | 2623         | 9.27            | 1.801                         | 6.95                          |
|     | average      |                 | 1.803                         | 7.5                           |
|     | ± standard deviation |            | ±0.006                        | ±0.5                          |

The coefficient of thermal expansion of the PECVD silicon nitride is $\alpha_0 = (1.803 \pm 0.006) \times 10^{-6}$ K$^{-1}$ and $\alpha_1 = (7.5 \pm 0.5) \times 10^{-9}$ K$^{-2}$. Also shown in Table 6.2 are the widths and the center deflections at room temperature. The differences in $w_0$ at room temperature are due to a stress gradient across the wafer.

Finally, the errors introduced by the parameters $v$, $a$, and $h$ are discussed. An error in $v$ of 10% results in an error of 0.4% in $\alpha_{SiN}(T)$. Errors of 1% in $h$ and $a$ cause errors of 0.07% and 1% in $\alpha_{SiN}(T)$, respectively. It is concluded that the method is robust against uncertainties in the film thickness as long as the thickness is homogeneous over the membrane.

M. Maeda et al. reported coefficients of thermal expansion for PECVD silicon nitride for different rf bias during film deposition [20]. Assuming that their silicon substrate has the coefficient of thermal expansion given in Eq. 2.19 we inferred values for the linear coefficient $\alpha_0$ at $25^\circ$C between $2.19 \times 10^{-6}$ K$^{-1}$ and $3.34 \times 10^{-6}$ K$^{-1}$ and for the quadratic coefficient $\alpha_1$ between $7.1 \times 10^{-9}$ K$^{-2}$ and $11.2 \times 10^{-9}$ K$^{-2}$ from their measurements. But we also inferred a significant cubic coefficient. Polycrystalline bulk silicon nitride has been reported to have a coefficient of thermal expansion of $0.8 \times 10^{-6}$ K$^{-1}$ and $1.8 \times 10^{-6}$ K$^{-1}$ at $27^\circ$C and $127^\circ$C, respectively [136].
7 Summary and Outlook

The load-deflection behavior, buckling and postbuckling of micromachined membranes were experimentally investigated and numerically analyzed. The numerical results were used to extract thin film mechanical properties from micromachined membranes with compressive residual stress or significant flexural rigidity. The film properties are the residual stress and strain, Young's modulus, Poisson's ratio, and the coefficient of thermal expansion. In case of the residual stress and strain, the variation of these properties across wafers was determined.

An analytical model for the load-deflection of long plates undergoing a plane strain deformation under a uniform load was formulated. The model consistently takes into account the flexural rigidity and residual stress of the plate material. It describes buckled, weakly compressive, and tensile films clamped to a rigid support. To qualify for the plane strain model, load-deflection test structures should have an aspect ratio $b/a$ larger than four. Based on the model, compressive films can now be studied without the need to include them in a multilayer. Their plane strain modulus is obtained with similar accuracy as was previously possible with tensile single layers, with uncertainties at the percent level. Also, smaller membranes with nonnegligible bending energy can be reliably analyzed. With small samples, the evaluation of the distribution of the residual stress and plane strain modulus over wafers is feasible.

Using the plane strain model the mechanical properties of three PECVD silicon nitride films were measured. The films were deposited with 50%, 55%, and 60% low frequency (LF) power to the total power. The plane strain modulus of these films was between 134 GPa and 142 GPa and increases slightly with increasing percentage of LF power. The stress changes from slightly tensile (25 MPa) at 50% LF power to compressive stress (-63.2 MPa) at 60% LF power. The resolution of the stress measurement was sufficient to determine a stress gradient of 0.38 MPa/mm for the silicon nitride film deposited with 50% LF power.

Under strongly compressive residual stress long plates develop complex buckling shapes from which the extraction of mechanical properties is difficult. However,
we found that such plates can be driven into a state of plane strain by the application of a sufficiently high differential pressure. The transition to the state of plane strain is accompanied by a mechanical instability. At pressures below their instability point the plates develop a longitudinal ripple profile. The transition is therefore referred to as ripple transition. A mechanical model of this phenomenon was developed. Using this model the instability points were computed as a function of the residual stress, Poisson’s ratio, geometry, and deflection of the plate. It was found that the instability points strongly depend on Poisson’s ratio of the plate material and thus provides a useful measurement principle to determine this mechanical parameter. Using the ripple transition Poisson’s ratio of a PECVD silicon nitride film of 0.703 μm thickness was determined with high accuracy. It is \( \nu = 0.254 \pm 0.022 \).

Strongly buckled silicon nitride membranes were fabricated to compare theoretical and experimental buckling profiles. The PECVD silicon nitride was deposited with 60% LF power to the total deposition power. The membranes cover a large range of reduced residual strains. Two residual strain regimes were found experimentally in which the post-buckling profiles show different spatial symmetries. At small negative reduced residual strains the buckling profile shows a fourfold rotational symmetry and all mirror symmetries of a square except the mirror symmetry with respect to the film plane. At more strongly negative reduced strains, only the fourfold rotational symmetry remains. The mirror symmetries are lost through a second instability transition at a weakly \( \nu \)-dependent critical residual strain.

To quantitatively interpret these observations the post-buckling profiles of square plates were analyzed using finite element simulations and the Ritz method. From extended simulations an easy-to-use function was derived which describes the center deflection of buckled square plates as a function of the residual film strain, Poisson’s ratio, and the geometry of the plate. Excellent agreement between computed and measured buckling profiles was found. The residual thin film strain was extracted from the center deflection of these membranes. The determined strain values are independent of size and buckling mode of the membranes and show a small standard deviation of 2% for membranes located close together on the wafer. The average strain of the wafer in combination with wafer curvature measurements leads to a Young’s modulus that is in excellent agreement with a value determined by bulge testing. Moreover, the coefficient of thermal expan-
sion of a PECVD silicon nitride deposited with 55% LF power was determined. The coefficient of thermal expansion at 25°C is \((1.803\pm0.006)\times10^{-6}\ \text{K}^{-1}\) and increases linearly with temperature. The slope is \((7.5\pm0.5)\times10^{-9}\ \text{K}^{-2}\).

The materials properties measured in this thesis were collected in the data base of thermophysical and mechanical material properties ICMAT. An internet interface provides easy access to the data and enables their use in FE modeling of IMEMS.

The methods developed and tested in this thesis can now be applied to study the mechanical properties of a wider range of thin films than previously possible using the membrane deflection method. If the theoretical results were extended to thin films with \(z\)-dependent elastic properties also multilayered films can be investigated. This is already done for the analytical plane strain model described in Chapter 4 but not yet for the ripple transition and the postbuckling behavior of square membranes. An important topic for future research is the influence of temperature cycling, stress cycling, and humidity on mechanical thin film properties since such long term effects may significantly affect the reliability of IMEMS.
APPENDICES

A.1 Numerical Solution of the Plane Strain Model

In this section, we propose algorithms to numerically solve the equations

\[ \bar{w}_0 = \bar{P}G_0(\bar{s}) \quad (A.1) \]

\[ \bar{s} = \bar{\sigma}_0 + \frac{1}{2} \bar{w}_0^2 H(\bar{s}) \quad (A.2) \]

where \( G_0(\bar{s}) = G(\bar{s}, 0) \). These two equations contain four parameters, namely \( \bar{w}_0 \), \( \bar{P} \), \( \bar{\sigma}_0 \), and \( \bar{s} \). If two of them are known the other can be calculated. Six cases of known parameter pairs can be distinguished.

In the cases where \( \{\bar{w}_0, \bar{s}\} \), \( \{\bar{P}, \bar{s}\} \), or \( \{\bar{\sigma}_0, \bar{s}\} \) are given, Eqs. A.1 and A.2 can be solved analytically. In the other cases the solution can only be obtained numerically. Since \( G_0(\bar{s}) \) diverges at \( \bar{s} = \bar{\sigma}_{cr} = -\pi^2/3 \) the numerical algorithms have to be chosen carefully to avoid numerical instabilities for \( \bar{s} \) values close to \( \bar{\sigma}_{cr} \).

The algorithms are based on the following properties of the functions \( H(\bar{s}) \) and \( G_0(\bar{s}) \). \( H(\bar{s}) \) depends only weakly on \( \bar{s} \) and \( G_0(\bar{s}) \) is well approximated by \( j(\bar{s}) = 1/9(\bar{s} - \bar{\sigma}_{cr}) \). Moreover, \( G_0(\bar{s}) > 1/9(\bar{s} - \bar{\sigma}_{cr}) \) for all \( \bar{s} > \bar{\sigma}_{cr} \). In the following it is assumed that either \( \bar{w}_0, \bar{P} \geq 0 \) or \( \bar{w}_0, \bar{P} \leq 0 \).

\( \{\bar{w}_0, \bar{P}\} \) are known

The solution \( \bar{s}^* \) to Eq. A.1 can be found using Newton’s iteration formula [138]

\[ \bar{s}^{(n+1)} = \bar{s}^{(n)} - \frac{G_0(\bar{s}^{(n)}) - \bar{w}_0}{\frac{\partial}{\partial \bar{s}} G_0(\bar{s}^{(n)})} \quad (A.3) \]

The series \( \bar{s}^{(n)} \) is monotonically increases and converges to \( \bar{s}^* \) if the starting value \( \bar{s}^{(0)} \) is between \( \bar{\sigma}_{cr} \) and \( \bar{s}^* \). Such an appropriate \( \bar{s}^{(0)} \) is given by
\[ s^{(0)} = \bar{\sigma}_{cr} + \frac{\bar{P}}{9.9\bar{w}_0}. \]  \hspace{1cm} (A.4)

\{\bar{w}_0, \bar{\sigma}_0\} \text{ are known}

In this case, a rapidly converging iteration to compute \( \bar{s}^* \) is

\[ s^{(n+1)} = \bar{\sigma}_0 + \frac{1}{2} \bar{w}_0^2 H(\bar{s}^{(n)}). \]  \hspace{1cm} (A.5)

A good the starting value is

\[ s^{(0)} = \bar{\sigma}_0 + \frac{1}{2} \bar{w}_0^2 H(\bar{\sigma}_{cr}) = \bar{\sigma}_0 + \frac{\bar{w}_0^2 \pi^2}{4}. \]  \hspace{1cm} (A.6)

\{\bar{P}, \bar{\sigma}_0\} \text{ are known}

Substitution of Eq. A.1 into Eq. A.2 yields

\[ \bar{s} = \bar{\sigma}_0 + \frac{1}{2} \bar{P}^2 G_0(\bar{s}^2) H(\bar{s}). \]  \hspace{1cm} (A.7)

We rewrite this equation in the following form

\[ g(\bar{s}) - \frac{2}{H(\bar{s})\bar{P}^2} = 0, \]  \hspace{1cm} (A.8)

where \( g(\bar{s}) = G_0(\bar{s})^2 / (\bar{s} - \bar{\sigma}_0) \). Since the second term of Eq. A.8 is almost independent of \( \bar{s} \), we consider it as a constant in Newton’s iteration scheme. This saves computing time. Thus, the iteration formula reads

\[ s^{(n+1)} = s^{(n)} - \frac{g(s^{(n)}) - 2/(H(s^{(n)})\bar{P}^2)}{\frac{\partial}{\partial s} g(s^{(n)})}. \]  \hspace{1cm} (A.9)

To ensure converge of this iteration the starting value should lie between \( \bar{\sigma}_{cr} \) and \( \bar{s}^* \). An appropriate value is provided by

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\[
\bar{s}^{(0)} = \frac{\gamma}{3^{4/3}11^{2/3}} + \frac{(11/3)^{2/3} \Delta \bar{\sigma}_0}{\gamma} + \frac{1}{3}(\bar{\sigma}_0 + 2\bar{\sigma}_{cr}),
\]
(A.10)

where

\[
\Delta \bar{\sigma}_0 = \bar{\sigma}_0 - \bar{\sigma}_{cr},
\]
(A.11)

\[
\gamma = (50\eta + 10\sqrt{\eta} \sqrt{25\eta + 363\Delta \bar{\sigma}_0^3 + 363\Delta \bar{\sigma}_0^3})^{1/3},
\]
(A.12)

and

\[
\eta = \frac{H(0) \bar{P}^2}{2} = \frac{256\bar{P}^2}{105}.
\]
(A.13)

The \(\bar{s}^{(0)}\) defined by Eq. A.10 is between \(\bar{\sigma}_{cr}\) and \(\bar{s}^{#}\) and is the largest real root of the following equation

\[
(\bar{s} - \bar{\sigma}_0)(\bar{s} + \bar{\sigma}_{cr})^2 = 9.9 \frac{2H(0) \bar{P}^2}{2}.
\]
(A.14)

Eq. A.14 is an approximation of Eq. A.8 and is obtained when \(G_0(\bar{s})\) is replaced by \(f(\bar{s})\) and \(H(\bar{s})\) by \(H(0)\), respectively. Note, that \(H(0)\) is the minimum of \(H\).

**A.2 Analytical Solution of the Plane Strain Model**

In this section, we develop an analytical solution of the plane strain equations

\[
\bar{w}_0 = \bar{P}G_0(\bar{s})
\]
(A.15)

\[
\bar{s} = \bar{\sigma}_0 + \frac{1}{2} \bar{w}_0^2 H(\bar{s}),
\]
(A.16)

where \(G_0(\bar{s}) \equiv G(\bar{s}, 0)\).
For $\sigma_0 \geq \sigma_{cr} = -\pi^2/3$, $P$ is a continuous and skew symmetric function of $\bar{w}_0$ and thus is expanded in the following Taylor series

$$P = \sum_{n=1, 3, \ldots} c_n \bar{w}_0^n,$$  \hspace{1cm} (A.17)

where the expansion coefficients $c_n$ are functions of the reduced residual stress $\sigma_0$ and are defined as

$$c_n(\sigma_0) = \frac{1}{n!} \left. \frac{d^n}{d\bar{w}_0^n} P \right|_{\bar{w}_0 = 0}.$$  \hspace{1cm} (A.18)

Similarly, $\bar{s}$ is expanded in powers of $\bar{w}_0$. Since $\bar{s}$ equals $\sigma_0$ at $\bar{w}_0 = 0$ and is independent of the sign of $\bar{w}_0$, it is given by

$$\bar{s} = \sigma_0 + \sum_{m=2, 4, \ldots} k_m \bar{w}_0^m,$$  \hspace{1cm} (A.19)

where again the expansion coefficients are functions of $\sigma_0$. These are given by

$$k_m(\sigma_0) = \frac{1}{m!} \left. \frac{d^m}{d\bar{w}_0^m} \bar{s} \right|_{\bar{w}_0 = 0}.$$  \hspace{1cm} (A.20)

Using Eqs. 4.8 and A.19, the coefficient $c_1$ becomes

$$c_1(\sigma_0) = \left. \frac{d}{d\bar{w}_0} P \right|_{\bar{w}_0 = 0} = \left. \frac{\partial}{\partial \bar{w}_0} P \right|_{\bar{w}_0 = 0} + \left. \frac{\partial}{\partial \bar{w}_0} \bar{s} \right|_{\bar{w}_0 = 0} \left( \frac{\partial}{\partial \bar{s}} P \right) = \frac{1}{G_0(\sigma_0)}. \hspace{1cm} (A.21)$$

Likewise, $c_3$ becomes

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where the coefficient $k_2$ can be derived using Eq. 4.9 as

$$k_2(\bar{\sigma}_0) = \left. \frac{1}{2} \frac{d^2}{d\bar{w}_0^2} \left( \bar{\sigma}_0 + \frac{1}{2} \bar{w}_0^2 H(\bar{s}) \right) \right|_{\bar{w}_0 = 0} = \frac{1}{2} H(\bar{\sigma}_0).$$  \tag{A.23}$$

Similarly, the coefficients $c_5$ and $k_4$ can be derived using Eqs. A.22 and A.23. Following this scheme for higher coefficients, we conjecture that $c_n$ and $k_m$ are generally given by

$$c_n = \frac{1}{(n-1)!!} \left( \frac{\partial}{\partial \bar{s}} \right)^{n-3} \left( H(\bar{s}) \frac{1}{2} \frac{\partial}{\partial \bar{s}} \left( \frac{1}{2} \frac{\partial}{\partial \bar{s}} G_0(\bar{s}) \right) \right) \bigg|_{\bar{s} = \bar{\sigma}_0}$$  \tag{A.24}$$

$$k_m = \frac{1}{m!!} \left( \frac{\partial}{\partial \bar{s}} \right)^{m-1} \left( H(\bar{s}) \frac{1}{2} \right) \bigg|_{\bar{s} = \bar{\sigma}_0}.$$  \tag{A.25}$$

The validity of Eqs. A.24 and A.25 was explicitly checked up to $n = 17$ and $m = 18$, respectively. In their general form, Eqs. A.24 and A.25 remain conjectures. Possibly, they may be proved using Faà di Bruno’s formula [137].
REFERENCES


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