Bistability study of buckled MEMS diaphragms

Dilan Ratnayake1, Masoud Derakhshani2, Thomas A Berfield2 and Kevin M Walsh1

1 Department of Electrical and Computer Engineering, University of Louisville, Louisville, KY, United States of America
2 Department of Mechanical Engineering, University of Louisville, Louisville, KY, United States of America

E-mail: kevin.walsh@louisville.edu

Keywords: MEMS, bistable, diaphragms, no-power, threshold-driven, stress

Abstract

Bistable elements are candidate structures for the evolving field of MEMS-based no-power event-driven sensors. In this paper, we present a strategy for producing bistable elements and investigate two compatible bilayer material systems for their realization using MEMS technology. Both bilayer systems leverage thermally-grown silicon dioxide as the principal stress-producing layer and a second material (either polyimide or aluminum) as the main structural layer. Arrays of buckled circular diaphragms, ranging in diameter from 100 μm to 700 μm in 50 μm increments, were fabricated and their performances were compared to modeled and FEA-simulated results. In all cases, the diaphragms buckled when DRIE-released as expected, and their buckled experimental heights were within 9.1% of the theory and 1.8% of the FEA prediction. Interestingly, the smaller diameter structures exhibited a directional bias which we investigate and forecast using FEA. These bistable mechanical elements have the ability to serve as building blocks for no-power threshold-driven smart switches. New contributions to the field include: (a) introduction of a new bistable material system made from aluminum and compressive oxide, (b) investigation of diaphragm diameter size as it related to the phenomena of bistability versus non-bistability, (c) FEA analysis of the critical transition between bistability and non-bistability, and (d) introduction of the 'dome factor' term to describe dome quality.

1. Introduction

Many experts predict that the world will contain a trillion IoT (Internet of Things) devices by 2035 [1]. If that becomes the new reality, either the energy needs for those sensors must be addressed, or a new type of no electrical power (NEP) sensor will need to be developed. The latter is the obvious preferred solution if the sensor can be 'powered' by the event it is designed to sense. Such threshold types of devices can be called no electrical power (NEP) event-driven sensors. An important integral structure to the design of such NEP event-driven sensors is a bistable element capable of changing states upon the detection of a threshold-sensing event, such as a specific pressure, temperature, humidity, or radiation exposure. Our research group has been very active in developing bistable candidate elements for NEP event-driven sensors [2–5]. We have previously developed various fabrication strategies for processing domed diaphragm structures which serve as the heart of the bistable devices using resist reflow and grayscale lithography [6]. Since these strategies use MEMS (microelectromechanical systems) fabrication techniques, we call them 'Buckle MEMS'. With this research, we further advance the Buckle MEMS NEP event-driven field by introducing a new fundamental bistable building block that combines stress-free polyimide and stress-engineered thin films (oxide and aluminum) for the production of robust large-displacement wrinkle-free bistable MEMS diaphragms.

2. Theory of operation

While MEMS sensors take advantage of the mechanical properties of their materials, most all are designed to operate within their linear structural response regime. With this research, we intentionally develop MEMS
structures designed to operate outside that regime. We do so by purposely introducing internal compressive stress into our deposited thin films so that they mechanically buckle when released. This provides the foundation for a truly bistable element. Figure 1 presents a general overview of our strategy. We begin with a traditional silicon wafer (figure 1(a)). We deposit a film or combination of films at a prescribed elevated deposition temperature (figure 1(b)). Upon cooling to room temperature, internal compressive stresses build up in the thin film if the deposited material has a lower coefficient of thermal expansion (CTE) than the much thicker silicon (figure 1(c)). Upon release using deep reactive ion etching (DRIE), the film will buckle out of plane if its effective internal compressive stress exceeds the stress required for buckling (figure 1(d)) and h is the buckling height which is measured from the $y = 0$ axis as shown in figure 1(d). A combination of films can be used to introduce the required stress and mechanical stability needed for uniform wrinkle-free buckled dome structures.

Although the process appears straightforward, there are many aspects to consider in order to produce structures that are reliable and robust. While this process can be used to fabricate buckled beams and other buckled structures, this paper focuses on the design and manufacturing of buckled circular diaphragms.

3. Buckling criteria for circular diaphragms

Buckling is a mechanical phenomenon that can occur in structures (beams or diaphragms) based on a combination of compressive stress, material stiffness, and the slenderness ratio of the structure (radius of gyration/length or thickness/diameter) [7]. As lateral compressive forces are applied to a diaphragm, for
example, the diaphragm is simply compressed. However, when the compressive stress is further increased, an unstable state for the structure can result. When the critical buckling stress is reached, the transverse deflection will increase rapidly and the diaphragm will buckle to its 1st stable state, which has a lower total strain energy than the simple compressed state [8, 9]. This can occur with MEMS-deposited thin films as well. If sufficient internal compressive stress is introduced during the deposition process, through mismatched thermal expansion coefficients for example, the films will buckle upon their release, as previously illustrated in figure 1.  
Such buckled diaphragms can be readily modeled using traditional mechanical engineering equations. Below we examine some of the more important equations needed for the design of MEMS-based buckled diaphragms.

The effective internal compressive stress \( \sigma_{\text{eff}} \) needed to produce diaphragm buckling is given by [6]:

\[
\sigma_{\text{eff}} = -\frac{4}{3} \frac{t^2}{R^2} Y_{\text{eff}} \left(1 - v_{\text{eff}}^2\right)
\]

where \( R \) is the radius of the circular diaphragm, \( t \) is the total thickness, \( Y_{\text{eff}} \) is the film’s effective Young’s Modulus and, \( v_{\text{eff}} \) is the film’s effective Poisson Ratio.

For a bilayer film [2, 6],

\[
Y_{\text{eff}} = \left( \frac{t_{\text{film1}} \times Y_{\text{film1}} + t_{\text{film2}} \times Y_{\text{film2}}}{t_{\text{film1}} + t_{\text{film2}}} \right)
\]

\[
v_{\text{eff}} = \left( \frac{t_{\text{film1}} \times v_{\text{film1}} + t_{\text{film2}} \times v_{\text{film2}}}{t_{\text{film1}} + t_{\text{film2}}} \right)
\]

The height of the buckled dome structure \( (h_0) \) can be calculated using:

\[
h_0 = \pm \frac{\sqrt{35}}{4} \sqrt{\frac{\sigma_{\text{eff}}}{\sigma_{\text{eff}}} - 1}
\]

Where \( \sigma_{\text{eff}} \) is the effective residual stress. Note that compressive stresses are negative in value.

The height of the dome when it abruptly transitions from one bistable state to the other is called the ‘snap’ height \( h_{\text{snap}} \), and it is given by:

\[
h_{\text{snap}} = \pm \frac{\sqrt{35}}{34} \sqrt{\frac{\sigma_{\text{eff}}}{\sigma_{\text{eff}}} - 1} = \frac{1}{\sqrt{3}} h_0
\]

(Both \( h_0 \) and \( h_{\text{snap}} \) are defined from the \( y = 0 \) axis as shown in figure 1(d)).

For a bilayer film [2, 6], we need to find the effective residual stress by considering the residual stress and thickness of each film as given in equation (6).

\[
\sigma_{\text{eff}} = \left( \frac{t_{\text{film1}} \times \sigma_{\text{film1}} + t_{\text{film2}} \times \sigma_{\text{film2}}}{t_{\text{film1}} + t_{\text{film2}}} \right)
\]

Finally, the critical force required to snap the buckled diaphragm from one state to the other can be calculated using [6]:

\[
F_C = \pm \frac{\pi}{3} \sqrt{\frac{35}{3}} \frac{t^2}{ \sigma_{\text{eff}} C} \left( \frac{\sigma_0}{\sigma_C} - 1 \right)^{1/2} = \frac{PR^2}{3}
\]

where \( P \) is surface pressure exerted on the buckled diaphragm.

Using the above equations as a guide, we designed an appropriate photomask for the fabrication of an array of diaphragms to build and test. To determine the minimum size for our diaphragm array, recall that the effective compressive stress in the film combination must exceed the critical stress expression for buckling. Using a combination of polyimide and thermal oxide for our initial film (details to be discussed in the next section), figure 2 shows that our smallest diaphragm size needed to be 100 \( \mu \)m. Accordingly, we designed our photomask array to include 13 different diameter sizes, ranging from 100 \( \mu \)m to 700 \( \mu \)m in 50 \( \mu \)m increments.

### 4. Candidate materials for buckled diaphragms

This paper explores two sets of material systems for the fabrication of buckled MEMS diaphragms. This first system consists of a combination of polyimide and thermally grown silicon dioxide. The second system consisted of thermally grown oxide along with aluminum, should a conductive top layer be required for a given application. In both cases, the thermal oxide layer serves as the primary source of compressive stress required for buckling, and the polyimide and aluminum films act primarily as the structural layer to assure mechanical integrity. Below we discuss the important features of each of the 3 candidate materials.
4.1. Thermally grown oxide
Thermal oxidation using oxygen or water vapor in combination with silicon is a common way to produce SiO\textsubscript{2}. It is a chemical process where SiO\textsubscript{2} is grown on a silicon wafer in the presence of oxygen at a high elevated temperature, typically ranging from 900 to 1200 °C. Furthermore, thermal cycling of the substrate along with any thermal expansion coefficient mismatches between the grown film and the relatively thick substrate can lead to the development of internal stresses within the thin film. This interesting phenomena can be leveraged to produce buckled MEMS diaphragms [7].

As mentioned above, thermal oxidation takes place at a relatively high temperature and then cooled down to room temperature at the end of the process. Silicon (2.3 \times 10^{-6} K^{-1}) has a higher coefficient of thermal expansion (CTE) compared to silicon dioxide (5.6 \times 10^{-7} K^{-1}). Due to the mismatch of the CTEs, the much thicker silicon substrate contracts more than the thin thermal oxide film during the cooling process, dominating the mechanical response. This produces compressive stress in the thin thermal oxide film, which is leveraged to produce buckled structure upon release from the substrate.

4.2. Polyimide
While thermally grown oxide is a convenient material for introducing compressive stress, it presents some challenges if used all by itself. Thin oxide films are very fragile and tend to wrinkle, distort, and even crack when released using DRIE as shown in figure 3 [4]. To overcome this challenge, we introduce a second low stress material, polyimide, to provide the needed mechanical and structural integrity. Polyimide films are commonly used on wafers as passivation layers, stress buffer layers, dry etch masks, structural layers, and re-distribution layers for chip scale packaging and wafer level packaging [10]. In this research, we explore the use of polyimide as a stress-free structural layer to mechanically enhance the thin oxide films so that smooth dome-like buckled structures are possible. Specifically, we examine PI 2600 polyimide because of its high molecular weight and the rigid rod polyimide structure of cured PI-2600 exhibits desirable film properties such as low stress, low coefficient of thermal expansion (CTE), low moisture and high modulus [11].

4.3. Aluminum
For applications where a top conductive surface is needed, we explored a third candidate material, aluminum. Aluminum thin films are used in variety of applications, especially in microelectronics, because of their low resistivity, high reflectance and low cost [12]. These applications include connections in semiconductor and integrated circuit devices, electrodes, and back contacts in solar cells. Similar to polyimide, upon deposition, the aluminum layer can be comparatively free of stress [8, 13]. Therefore, aluminum should also be a viable candidate material for the structural support layer, although it cannot be deposited as thick as polyimide.

5. Fabrication process
Our fabrication process is shown in figure 4. It begins with an n-type double side polish 4” silicon wafer which was cleaned using the RCA cleaning process (figure 4(a)). The wafer was loaded into a three-zone tube furnace at 600 °C. Nitrogen gas was introduced to the furnace so that it flows through the furnace until oxidation starts; thus, creating an inert clean environment for the oxidation process. In order to create a high-quality oxidation layer, a dry/wet/dry thermal cycle was used (2 h. oxidation at 1000 °C) (figure 4(b)). The silicon wafer was removed at 600 °C after the oxidation process was completed, and a Filmetrics system was used to determine the thickness of the oxide, which was measured to be 0.45 μm. Next, the structural layer was added (figure 4(c)). For
the case of a polyimide structural layer, PI-2610 polyimide, which is highly viscous due to its high molecular weight, was selected to provide the needed mechanical support. To ensure the wafer was completely dry (thus ensuring the polyimide would correctly adhere) the wafer was placed on a hotplate for a dehydration bake for three minutes at 115 °C. This was done to eliminate the moisture on the wafer and make it ready for the application of the polyimide. Then PI 2610 was spun on the wafer. PI 2610 has the ability to provide a 1.0 to 2.5 μm thick layer by adjusting the spread and spin parameters. Spread and spin parameters were adjusted until 1.7 μm thick polyimide layer was obtained. After the spin, a soft bake was performed using a hot plate at 130 °C for another 3 min to remove any solvent remaining in the film. Following the soft bake, the wafer was transferred to a Yes Polyimide Oven, which is a high temperature vacuum oven (550 °C maximum temperature), for thermal curing. The wafer was loaded at 50 °C and cured at 350 °C for an hour in vacuum.

For the case of an aluminum structural layer, aluminum was deposited using a 4″ diameter pure aluminum (99.995%) target after completing the oxidation process. Sputtering was performed with a PVD 75 RF/DC sputtering system from Kurt J Lesker. For all experiments, the power was 300W DC and the pressure was 5 mT while the deposition time varied for each sample to change the thickness of the film. Before each deposition, a pre-sputtering process was performed to clean the target’s surface in case there is an oxide layer or contamination present.

Figure 3. Typical localized distortions found with releasing thin oxide diaphragms without any mechanical support layer [4].

Figure 4. Left side: cross sectional view of the device. Right side: fabrication process flow chart.
After completing the structural layer deposition, the wafer was transferred to the lithography bay, where arrays of circular patterns on the wafer backside were opened so that the bulk silicon can be etched using DRIE to form the diaphragms as shown in figure 4(d). Details about this process are as follows. First, the wafer was placed on a hotplate at 115 °C for three minutes for a dehydration bake. Then AZ4620 photoresist was spun onto the backside of the sputtered wafer using a spinner. Then a soft bake was performed for 10 min at 100 °C on a hotplate which is below the glass transition temperature of the AZ4620. Standard binary lithography was performed using an MA6/BA6 Suss Mask Aligner, which provides the UV light for exposure, and the photomask (containing circular features from 100 μm to 700 μm diameter). Once the sample was exposed to UV light, AZ400k Developer was used to develop the exposed structures, and a QDR (Quick Dump Rinse) and SRD (Spin Rinse Dryer) were used to clean the wafer using DI water. To carry out the hard bake, the patterned wafer was placed on the hotplate at 115 °C for one minute. This removes any remaining solvents and hardens the resist, thus improving its ability to function as an etch mask. Finally, the wafer was immersed in BOE (Buffer Oxide Etch) bath for four minutes to etch the oxide, and a QDR and SRD were used to once again clean the wafer.

Prior to DRIE etching, the processed wafer was first diced into several samples so more experiments could be conducted. Samples were transferred to the DRIE System to etch the bulk silicon from the backside. A sample was attached to a handle wafer using polyimide tape and loaded to the DRIE System. This DRIE process was continued until it reached the top etch stop layer, which was the thermally grown oxide. Samples were transferred to the DRIE System to etch the bulk silicon from the backside. A sample containing circular features from 100 μm to 700 μm diameter was attached to a handle wafer using polyimide tape and loaded to the DRIE System. This DRIE process was conducted. Samples were transferred to the DRIE System to etch the bulk silicon from the backside. A sample consisting of 0.45 μm of thermally grown silicon dioxide and 1.7 μm of cured polyimide. As mentioned above, the bilayer structures were released using DRIE. Encouragingly, all of the diaphragms in the arrays buckled as predicted by our modeling shown in figure 2. We observed that approximately 10%–15% of diaphragms buckled in their down state, and the remainder buckled in their up state. However, this observation was not consistent from one process run to another. We attribute this to some of the fabrication subtleties in the overall process which could impact the final buckle direction. For example, the device wafer needed to be attached and then released from a handle wafer. The manual release process could affect the final buckle direction. Also, the various pressure differentials that the device wafer experienced during the DRIE process could potentially affect the final buckle direction as well. The buckling could be readily observed using either light microscopy or profilometry. Figure 5 shows an optical image of a typical buckled diaphragm using an Olympus BX51 high-power optical microscope equipped with DIC (differential interference contrast). The DIC feature of the microscope allowed easy determination of the direction of buckling, which was difficult to assess otherwise. As shown in figure 5, the color pattern on the diaphragm surface changed relative to the direction of buckle (buckle up versus buckle down).

The second method for assessing the buckling profile states was by profilometry using a Dektak system. Figure 6 presents the profilometry data for a typical 700 μm diaphragm in both its buckled up and down states. The diaphragm state was switched by simply pulling light vacuum on the structure. From the profilometry data, the buckle height could be measured and compared to our model and predictions. Next, the diaphragms were observed using the Zygo 3D optical interferometer, and 3D profiles of buckled up and buckled down diaphragms are shown in figure 7.

Before using the modeling, equations introduced in section 3, we needed to adjust the previous equations using oxide-polyimide bilayer film properties. For the case of the effective Young’s Modulus for the oxide-PI bilayer, the following equation and values were used [7]:

\[
Y_{\text{eff}} = \frac{t_{\text{SiO}_2} \times Y_{\text{SiO}_2} + t_{\text{PI}} \times Y_{\text{PI}}}{t_{\text{SiO}_2} + t_{\text{PI}}} \tag{8}
\]

where:
- \( t_{\text{SiO}_2} \) = Oxide thickness (0.45 μm)
- \( Y_{\text{SiO}_2} \) = Young’s modulus of silicon dioxide (73 GPa) [6]
- \( t_{\text{PI}} \) = Polyimide thickness (1.7 μm)
- \( Y_{\text{PI}} \) = Young’s modulus of polyimide (8 GPa) [6]

6. Results and discussion

6.1. Oxide-PI diaphragms

The above fabrication process was first implemented to produce oxide-polyimide (Oxide-PI) diaphragms consisting of 0.45 μm of thermally grown silicon dioxide and 1.7 μm of cured polyimide. As mentioned above, the bilayer structures were released using DRIE. Encouragingly, all of the diaphragms in the arrays buckled as predicted by our modeling shown in figure 2. We observed that approximately 10%–15% of diaphragms buckled in their down state, and the remainder buckled in their up state. However, this observation was not consistent from one process run to another. We attribute this to some of the fabrication subtleties in the overall process which could impact the final buckle direction. For example, the device wafer needed to be attached and then released from a handle wafer. The manual release process could affect the final buckle direction. Also, the various pressure differentials that the device wafer experienced during the DRIE process could potentially affect the final buckle direction as well. The buckling could be readily observed using either light microscopy or profilometry. Figure 5 shows an optical image of a typical buckled diaphragm using an Olympus BX51 high-power optical microscope equipped with DIC (differential interference contrast). The DIC feature of the microscope allowed easy determination of the direction of buckling, which was difficult to assess otherwise. As shown in figure 5, the color pattern on the diaphragm surface changed relative to the direction of buckle (buckle up versus buckle down).

The second method for assessing the buckling profile states was by profilometry using a Dektak system. Figure 6 presents the profilometry data for a typical 700 μm diaphragm in both its buckled up and down states. The diaphragm state was switched by simply pulling light vacuum on the structure. From the profilometry data, the buckle height could be measured and compared to our model and predictions. Next, the diaphragms were observed using the Zygo 3D optical interferometer, and 3D profiles of buckled up and buckled down diaphragms are shown in figure 7.

Before using the modeling, equations introduced in section 3, we needed to adjust the previous equations using oxide-polyimide bilayer film properties. For the case of the effective Young’s Modulus for the oxide-PI bilayer, the following equation and values were used [7]:

\[
Y_{\text{eff}} = \frac{t_{\text{SiO}_2} \times Y_{\text{SiO}_2} + t_{\text{PI}} \times Y_{\text{PI}}}{t_{\text{SiO}_2} + t_{\text{PI}}} \tag{8}
\]

where;
- \( t_{\text{SiO}_2} \) = Oxide thickness (0.45 μm)
- \( Y_{\text{SiO}_2} \) = Young’s modulus of silicon dioxide (73 GPa) [6]
- \( t_{\text{PI}} \) = Polyimide thickness (1.7 μm)
- \( Y_{\text{PI}} \) = Young’s modulus of polyimide (8 GPa) [6]
This produced an effective Young’s Modulus of 20.53 GPa for our oxide-PI bilayer film.

Similarly, the effective residual stress of the oxide-polyimide diaphragm was calculated using the following equation [14]:

\[
\sigma_{\text{eff}} = \left( \frac{t_{\text{SiO}_2} \times \sigma_{\text{SiO}_2} + t_{\text{PI}} \times \sigma_{\text{PI}}}{t_{\text{SiO}_2} + t_{\text{PI}}} \right)
\]  

(9)

where;
- \(\sigma_{\text{SiO}_2}\) = Stress in the oxide film (MPa)
- \(\sigma_{\text{PI}}\) = Stress in the polyimide film (MPa)

The polyimide (PI-2610) is a stress free material according to the manufacture specifications [11]. Therefore, the value of stress in the above equation was considered to be zero for this calculation. A Toho FLX-2320-S stress analyzer was used to determine the residual stress of the thermal oxide, and it was measured to be \(-275\) MPa. These values were then substituted into the equation (9) to determine the effective residual stress on the bilayer, which was calculated to be \(-57.56\) MPa. Both Poisson’s ratios of oxide and polyimide were found to be nearly identical, so the effective Poisson ratio of 0.2 was used. These effective values of the material properties of the bilayer film were then used in equation (4) to determine the theoretical buckling height.
Figure 8. Comparison of the experimental, theoretical and FEA buckling height versus diaphragm diameter for 0.45μm oxide/1.7μm polyimide diaphragms. The 'bistability' region labeled in the figure was determined experimentally by applying vacuum to the diaphragms.

As mentioned previously, the experimental buckle height was measured using a Dektak profilometer and then vacuum was applied underneath individual diaphragms to test their bistability. Equation (4) was used to predict the initial theoretical buckling height for each diaphragm. Figure 8 shows a comparison of our experimental and theoretical buckling height for each Oxide-PI diaphragm. Two different experimental sample arrays were tested. As shown in figure 8, the simple model predicted by equation (4) did a respectful job at predicting the experimental behavior, considering effective values for the mechanical properties of the bilayer film had to be used. In general, the model overestimated the buckle height except at the very low end. Figure 8 also presents our testing for 'bistability'. We did such by applying vacuum to mechanically change the buckled state of the diaphragm (from buckled up to buckled down or vice versa). While we were able to mechanically change the state of all the diaphragms, not all would remain in their 2nd bistable state when vacuum was removed. Some of the samples would immediately return to their original buckled condition. Those samples were labeled as being 'not bistable' in figure 8. Experimentally, the diaphragms with diameter from 300 μm to 700 μm were found to be truly 'bistable', whereas the diaphragms with diameters from 100 μm to 250 μm were 'not bistable'. Diaphragms, not all would remain in their 2nd bistable state when vacuum was removed. Some of the samples would immediately return to their original buckled condition. Those samples were labeled as being 'not bistable' in figure 8. Experimentally, the diaphragms with diameter from 300 μm to 700 μm were found to be truly 'bistable', whereas the diaphragms with diameters from 100 μm to 250 μm were 'not bistable'.

The 'non-bistable' buckled diaphragms suggests that a biased direction exists within the structures. Such bias is attributed to a gradient of stress distribution through the total diaphragm thickness and small symmetry imperfections, both of which are unaccounted for in the simple theoretical model employed.

To probe these effects, a nonlinear finite element analysis of the bistable diaphragm was performed in ANSYS with SHELL281 selected as the element type in the model considering very small thickness to diameter ratios. Since in the experimental setup the compressive stress was only induced in the oxide layer, the imposed stress was applied to this layer in the FEA model as well to have more accurate simulation results. To perform this properly, a thermal expansion coefficient was introduced for the oxide layer in the modeling process and a certain temperature was applied to find the equivalent stress measured experimentally [15]. To do so, first, a thin-walled diaphragm made of two sections (oxide and polyimide) was modeled with the clamped boundary condition at the edge and zero out of plane deflection condition for the whole surface. The temperature was then applied to the model to mimic the equivalent stress measured in the experimental setup. Finally, the out of plane deflection constraint was removed, and the model was solved nonlinearly to find the buckling height of the diaphragm under the applied loading conditions.

As can be seen from figure 9, FEA results are in better agreement with the experimental data compared to the ones obtained from the simple theory. This is due to several simplifications considered in the developed theory, including the order of the nonlinearity and the effective stress/thickness consideration, which resulted in a less accurate theoretical analysis. As discussed above, diaphragms with smaller diameters were found as not bistable structures. While the experimentally measured stress in the oxide layer was much higher than the calculated critical stress, biased conditions created in the fabricated samples coming from experimental imperfections and different material properties and stress level in the deposited oxide/PI layers would result in requiring higher stress value than the critical one for having bistable structures. This effect is expected to be more noticeable for smaller diameters as the critical values are higher and the biased condition becomes more intensified.
To better understand this experimental observation, finite element analysis of two different cases, 200 \, \mu m and 300 \, \mu m diameters which were found as not bistable and bistable structures respectively, are considered in this section. This bistability threshold behavior was accurately captured by multiple FEA modeling methods, including either by imposing a stress gradient within the structure (requires 3D model) or by imposing a geometry imperfection of 98\% polyimide coverage of the diaphragm surface (2D model). Both FEA cases considered were found to have a negligible effect on the calculated critical stress and buckling height compared to completely symmetric FEA model case. For both cases, three steps of analysis were performed as shown in figure 9. First, the diaphragms were buckled into their first states (buckled up) by applying the equivalent thermal stress measured in the experimental setup. The structures were then switched to their second states (buckled down) by applying sufficient surface pressure in the models. The last step was removing this surface pressure from the developed models to check the structures bistability. As can be observed in the figure, the 200 \, \mu m diameter diaphragm returned to its first state after removing the surface pressure (figure 9(c)) while the 300 \, \mu m diameter diaphragm remained in its second state after the pressure removal (figure 9(f)), consistent with the experimental findings in figure 8. Furthermore, the maximum buckling heights are different for the two buckling states in this case, as is expected to be found for a general bistable system with symmetry imperfections.

This analysis demonstrates that the existence of small imperfections biased in the structure can significantly affect its bistability and result in having monostable diaphragms at smaller diameters. Further FEA results showed that bistability could occur in diaphragms with larger diameters at lower stress levels, which verified the experimental threshold shown in figure 8 for this behavior.

6.2. Oxide-al diaphragms
For our second manufacturing strategy, aluminum was explored as the upper mechanical structural layer in place of polyamide. We maintained the thickness of the underlying oxide layer constant at 0.45 \, \mu m. Since aluminum must be deposited using sputtering or evaporation, it cannot be deposited as thick as spin-on polyimide. Therefore, we anticipated that there might be a minimum threshold thickness needed for the aluminum mechanical layer to produce nicely shaped dome structures, especially given the very wide range of diameter sizes in our test array. As anticipated, we discovered experimentally that very thin aluminum produced released dome structures that were wrinkled and deformed, as shown in the right image of figure 10. Figure 10 captures the interesting transition diameter for the case of 0.675 \, \mu m thick aluminum, where the 500 \, \mu m diameter structure produced a well-formed dome, but its 600 \, \mu m companion exhibits the onset of higher-order buckling modes and/or local buckling (wrinkling) due to insufficient effective plate stiffness for the given residual stress and aspect ratio of this case.

To further study this phenomenon, we varied the thickness of the top aluminum layer and introduced a binary ‘dome factor’ term to help evaluate them. We defined the ‘dome factor’ to be ‘1’ when the diaphragm diameter produced smooth well-formed buckled domes, and ‘0’ otherwise. Figure 11 shows the results for the cases of 0.45 \, \mu m, 0.675 \, \mu m and 0.9 \, \mu m thick aluminum. As shown in the figure, for the cases of 0.45 \, \mu m and 0.675 \, \mu m thick aluminum, only the smaller diameter structures produced well-formed domes (dome factor = 1). On the other hand, for the case of 0.9 \, \mu m thick aluminum, the entire array of diameters resulted in...
smooth well-formed domes. This result makes sense as it is consistent with our previous experience demonstrated in figure 3 that oxide only diaphragms (i.e. aluminum thickness equal zero) exhibited wrinkling. Simply put, 0.45 $\mu$m and 0.675 $\mu$m of aluminum thickness were insufficient to overcome the oxide mechanical instability. However, when the aluminum thickness was increased to 0.9 $\mu$m, near perfect domes resulted for the case of all diameter sizes.

As done in the previous section, we then compared our experimental buckled height to our theoretical model. The initial buckle height was once again measured using a Dektak profilometer and then vacuum was applied on individual diaphragms to test their bistability. Equation (4) was used to calculate the initial buckling height for each diaphragm and FEA modeling was used to produce finer predictions. Figure 12 compares the two predictions with the experimental results, as well as the experimentally determined bistability region. As shown in the figure, our simple model using effective values provides a relatively close prediction while the FEA analysis

**Figure 10.** Optical images of 500 $\mu$m and 600 $\mu$m in diameter buckled diaphragms using DIC brightfield illumination.

**Figure 11.** Dome factor versus diameter of the diaphragm for different thickness of aluminum.
provide a better estimation as expected due to the less simplifications considered in the modeling process. Furthermore, the 200 μm to 700 μm diameter diaphragms were found to be bistable with the applied vacuum, while diameters below 150 μm were not.

### 7. Conclusions

This research focused on designing bistable dome-shaped diaphragms and characterizing their properties. Two different types of MEMS-based diaphragms were studied: oxide-polyimide diaphragms and oxide-aluminum diaphragms. In all cases, the compressive stress in the thermally grown oxide layer was engineered to cause the released films to buckle. However, due to the thin oxide’s fragile nature, a stress-free structural layer was needed to produce nice uniform hemispherical domes. Polyimide was shown to be an excellent candidate material for the structural layer. Arrays of oxide-polyimide bilayer domes with diameters ranging from 100 μm to 700 μm were fabricated and found to be mechanically bistable. Relatively thick patterned aluminum was also investigated as the structural layer in place of polyimide. All diaphragms were characterized, and the experimental data were compared with both the theoretical and FEA predictions. While the simple theory provided predictions in good agreement with the experimental data (within 9.1%), the FEA analysis provided an even closer match (within 1.8%).

Interestingly, we determined that the domed structures exhibited two distinct regions of operation. For the case of oxide-polyimide structures, diameters greater than 300 μm were truly bistable, while diaphragms less than 250 μm exhibited a bias that caused them to return to their previous state. This behavior was confirmed using nonlinear FEA analysis. In particular, we found that introducing even mild disruptions to the structure symmetry greatly influences the predicted bi-stability behavior threshold, helping match the experimentally observed behavior without disrupting other commonly measured parameters such as initial buckled height. This research also introduced a new parameter called the ‘dome factor’, which characterized the shape of the structure upon its release. We experimentally determined that the dome factor was directly dependent upon the thickness of the structural film.

### Acknowledgments

This research was partially funded by National Science Foundation under Grant No. 1542164. The authors also wish to acknowledge the University of Louisville Micro /Nano Technology Centre (MNTC) and its cleanroom staff for their assistance with the many fabrication processes.

### ORCID iDs

Dilan Ratnayake [https://orcid.org/0000-0003-4270-0227](https://orcid.org/0000-0003-4270-0227)

### References

[1] Berger I W 2020 The internet of things is changing the world The Wall Street Journal [https://blogs.wsj.com/cio/2020/01/10/the-internet-of-things-is-changing-the-world/](https://blogs.wsj.com/cio/2020/01/10/the-internet-of-things-is-changing-the-world/)

[2] Gowrishetty U R et al 2010 Fabrication of Polyimide Bi-stable Diaphragms using Oxide Compressive Stresses for the Field of ‘Buckle MEMS’ [20 075013](https://doi.org/10.1116/2010.20075013)
[3] Ratnayake D 2015 Engineering stress in thin films for the field of bistable MEMS J. Micromech. Microeng. 25 125025
[4] Gowrishetty U, Jackson D and Walsh K 2011 Mems bi-stable buckled diaphragms for no power applications Advanced in Nanotechnology and Applications III 89–100
[5] Ratnayake D and Walsh K M 2016 Invar thin films for MEMS bistable devices SoutheastCon 2016
[6] Loomis J et al 2016 Grayscale lithography—automated mask generation for complex three-dimensional topography J. Micro/Nanoﬁlms. MEMS MOEMS 15 1–10
[7] Gowrishetty U, Walsh K and Jackson D 2010 No-power vacuum actuated bi-stable MEMS SPDT switch Proc. of IEEE Sensors pp 1745–9
[8] Arya R et al 2006 Thermally actuated, bistable, oxide/silicon/metal membranes J. Micromech. Microeng 16 40–7
[9] Ding X et al 1990 A study on silicon-diaphragm buckling IEEE 4th Technical Digest on Solid-State Sensor and Actuator Workshop
[10] Hubbard R L et al 2004 Low temperature curing of polyimide wafer coatings IEEE/CPMT/SEMI 29th Int. Electronics Manufacturing Technology Symp. (IEEE Cat. No.04CH37585)
[11] MicrooSystems H 2009 PI-2600 Series-Low Stress Applications
[12] Kang T et al 2014 Modification of optical and mechanical surface properties of sputter-deposited aluminum thin ﬁlms through ion implantation Int. J. Precis. Eng. Manuf. 15 889–94
[13] Hodge T C, Bidstrup-Allen S A and Kohl P A 1997 Stresses in thin ﬁlm metallization IEEE Trans. Compon. Packag. Manuf. Technol. A 20 241–251
[14] Omar Z et al 2007 Investigating thin ﬁlm stresses in stacked silicon dioxide/silicon nitride structures and quantifying their effects on frequency response J. Micromech. Microeng 17 1042–51
[15] Porter D A et al 2012 Mechanics of buckled structure MEMS for actuation and energy harvesting applications ASME 2012 Int. Mechanical Engineering Congress and Exposition (American Society of Mechanical Engineers Digital Collection)