Improvement of Specimen Preparation Process for Bulge Test Using the Combination of XeF_2 and Deep Reactive Ion Etching

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The bulge test is a convenient approach to determine the thin film mechanical properties. This study presents a fabrication process to prepare the circular membrane made of metal as well as dielectric films for bulge test. The process successfully combines the dry etching of deep reactive ion etching (DRIE) and XeF_2 to release the test metal films. The Si_3N_4 film is used to protect the metal layers during the release process. Thus, the ion bombardment of the test metal films by DRIE is prevented. In addition, this process is also designed to prevent the pre-deformation of the Si_3N_4 and the metal films before release. By changing the recipe of XeF_2 etching, the circular Si_3N_4 test membrane can also be fabricated. In applications, the circular membranes of Al, Au, and Si_3N_4 films were successfully prepared using the presented approach. This study also performed the bulge test by using these specimens to determine the thin film elastic modulus.

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1. Introduction

The micro electro mechanical systems (MEMS) technology is regarded as an enabling tool for not only various consumer products, but also some emerging research fields (e.g., nano- and bio-technologies). In general, the MEMS devices are made of thin film materials. The performance as well as the reliability of MEMS devices is influenced by the characteristics of thin film materials. There were many methods to measure the mechanical properties of thin films including uniaxial tensile tests,[1] beam bending tests,[2] nanoindentation tests,[3,4] and frequency resonant methods.[5,6] Many MEMS devices have also been developed to characterize thin film material properties.[7] Despite the availability of numerous methods to measure the mechanical properties of thin film materials, the test setup, specimen preparation, and even the modeling of the problems are still challenges in the field.

The non-contact bulge test to determine the thin film material properties has been presented in refs. 8–10. The mechanics formulation associated with the bulge test has been established to show the relation of pressure-load and film-deflection. Based on the bulge test, the elastic modulus and residual stresses can be determined from the relationship of pressure-load and film-deformation. However, it remains a challenge to prepare a single-layer metal film for bulge test. Presently, the mechanical properties of metal films can be extracted from the double-layer composite formula.[10,11] Nevertheless, errors will result from the double-layer composite formula. In addition, the specimen preparation using the anisotropic bulk silicon etching will lead to a test film of rectangular shape. From the mechanics point of view such as singular point and boundary conditions, it is more complicated to analyze the rectangular membrane than the circular one.

This study presents a new approach by combining the dry etching processes of deep reactive ion etching (DRIE) and XeF_2 to fabricate the circular metal membrane for bulge test. During the DRIE etching process, the Si_3N_4 acts as the protection film for metal layers. The ion bombardment of the test metal films by DRIE is thus prevented. The protected Si_3N_4 film is then removed by XeF_2. In general, most of the metal films will not be removed by XeF_2 gas.[12] The process is also designed to prevent the pre-deformation of the Si_3N_4 and the metal films before the release of circular membrane. By changing the recipe of XeF_2 etching, the circular Si_3N_4 test membrane can also be fabricated. In applications, the suspended Si_3N_4, Al, and Au circular membranes for bulge test have been fabricated.

2. Theory of Bulge Test

In general, the bulge equation for the circular membrane with radius a, as indicated in Fig. 1, can be expressed as[10)]

$$P = 4\sigma_0 t \frac{h}{a^2} + C_1 Y t \frac{h^3}{a^4}, \tag{1}$$

where P is the applied hydraulic pressure, t is the thickness of the thin film, h is the maximum deformation of height, and Y is the biaxial modulus, $Y = E/(1 - v)$ and E is the elastic modulus of thin film. The parameter C_1 was related to the bi-axial modulus (Y) of thin film. As the variation of deflection h with the pressure load P is measured, the elastic modulus and residual tensile stress of thin film materials can be determined from the curve fitting of measurement results to eq. (1). The energy method is employed to model the deformation behavior of a thin film in the bulge test. In ref. 13, the displacement function for circular membranes is expressed as

$$w(r) = h(1 - r^2/a^2)^2, \tag{2}$$

where $w(r)$ is the out-of-plane displacement function. On the other hand, Lin *et al.*[10] analyzed the potential strain energy by neglecting the contribution of bending energy. The out-of-plane displacement of a circular membrane deformed by hydraulic pressure has been expressed as

$$w(r) = h(1 - r^2/a^2). \tag{3}$$

The parameter C_1 in eq. (1) can be yielded from either eq. (2) or (3). Thus, the elastic modulus is determined from eq. (1) after the relationship between applied pressure P and maximum deflection h is measured.

3. Preparing Specimens and Experiments

This study established an approach to prepare the specimen containing circular membrane for bulge test, especially for

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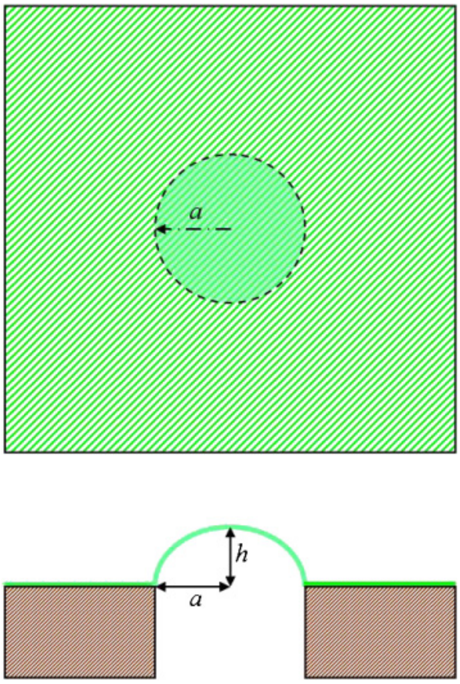


Fig. 1. (Color online) The spherical cap model for bulge test.

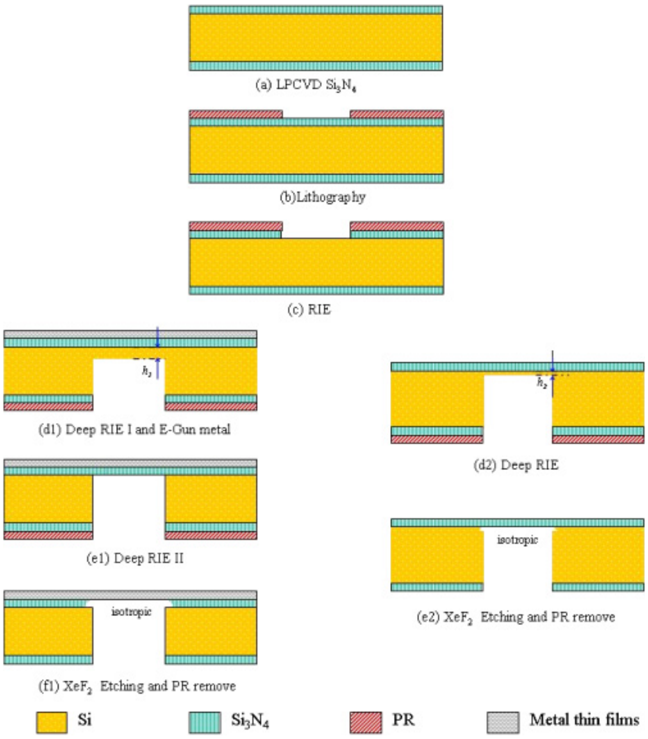


Fig. 2. (Color online) The proposed fabrication process steps to prepare the circular membrane.

metal films. In application, the Al, Au, and Si_3N_4 thin film specimens were fabricated, and the mechanical properties of these films were characterized using the bulge test. The thin films were also measured using the nano-indenter to confirm the results from bulge test.

3.1 Preparing specimens

This study employed the dry etching processes including the DRIE and the XeF_2 to fabricate the thin film circular membrane for bulge test. As shown in Fig. 2(a), a low stress Si_3N_4 was deposited on a double polished Si wafer using low pressure chemical vapor deposition (LPCVD). The deposition conditions were 875° centigrade and 0.7 Torr, with a ratio of $SiH_2Cl_2 : NH_3 = 3.5 : 1$. The Si_3N_4 on the backside of the wafer was defined by photolithography and patterned by reactive ion etching (RIE), as illustrated in Figs. 2(b) and 2(c). Then, the deep RIE was employed to remove part of the silicon substrate, as shown in Fig. 2(d-1). The Si of thickness h_1 was left to increase the stiffness of membrane region to prevent its deformation during the following metal deposition. After that, the metal film to be characterized for bulge test was deposited on the substrate. As shown in Fig. 2(e-1), the Si of thickness h_1 was then fully removed by DRIE. In this process, the Si_3N_4 was used as the protection layer to prevent the ion bombardment of DRIE on the metal film. Finally, as shown in Fig. 2(f-1), the XeF_2 was used to isotropically remove the Si_3N_4 film (recipe: 3 Torr XeF_2 only) to fully suspend the circular metal membrane. The shape of the suspended membrane was defined by the mask in Fig. 2(b). The similar process steps can also be employed to prepare the Si_3N_4 membrane for bulge test. In this case, the Si_3N_4 film to be tested was deposited and patterned, as shown in Figs. 2(a)–2(c). After that, the DRIE was used to remove most of the Si substrate, as shown in Fig. 2(d-2). The remaining Si h_2 was used to prevent the ion bombardment on the Si_3N_4 film. Finally, as shown in Fig. 2(e-2), the XeF_2 was used to isotropically remove the remaining Si (recipe: 3 Torr XeF_2 together with 2 Torr N_2, for a good etching selectivity between Si_3N_4 and Si) to fully suspend the circular Si_3N_4 membrane. Finally, the substrate was cut into $1 \times 1\,cm^2$ chips as the testing specimen. In applications, the suspended Al (5000 nm), and Au (2000 nm), and Si_3N_4 circular membranes were fabricated, as shown in Fig. 3. Figure 4 shows the front- and back-side views of the bulge test specimen prepared using the presented process.

3.2 Experiments

The experimental setup of the present bulge test is shown in Fig. 5. The vacuum system consisted of a vacuum chamber, a vacuum pump, two pairs of needle and pneumatic valve. It was operated by the program logic controller (PLC) which controlled the pressure sensor to turn the pneumatic valve on/off. The value of pressure sensor was calibrated by a traceable digital manometer (Yokogawa MT110). The manometer was calibrated with an uncertainty of 2.7 Pa. The specimen chip was glued on a poly(methyl methacrylate) (PMMA) holder with a 3 mm in diameter central hole. In addition, the pressure can be controlled inside and outside of the chamber, thus the cycling load can be applied to the membrane by this system. Besides this, negative pressure can also be performed. The deformation of the membrane was measured by a commercial optical interferometer. The resolutions were 3 nm in vertical scanning interferometer (VSI) mode and 0.3 nm in phase shifting interferometer (PSI) mode, respectively. A standard calibration specimen with 10.7 µm depth was utilized to calibrate the displacement measurement of interferometer in the VSI mode. In this study, the optical system for measuring the thickness

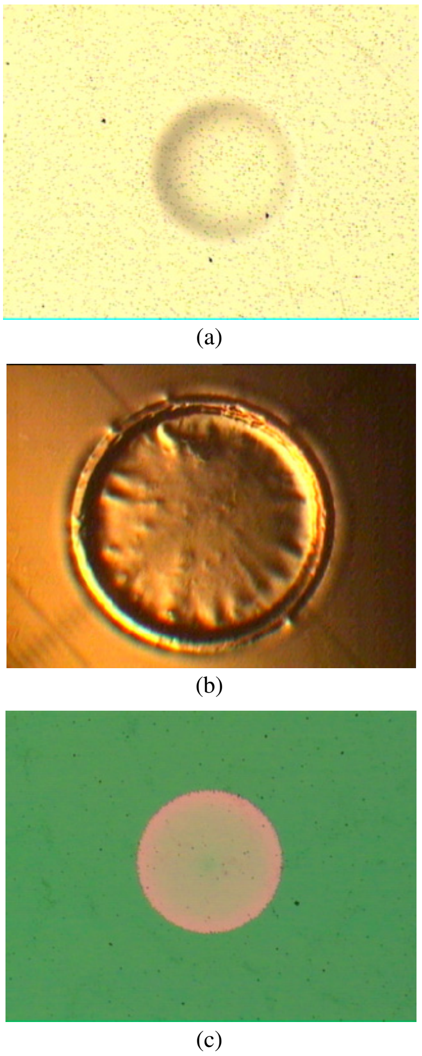


Fig. 3. (Color online) The photos of typical fabricated specimens with, (a) Al, (b) Au, and (c) Si_3N_4 thin film circular membranes.

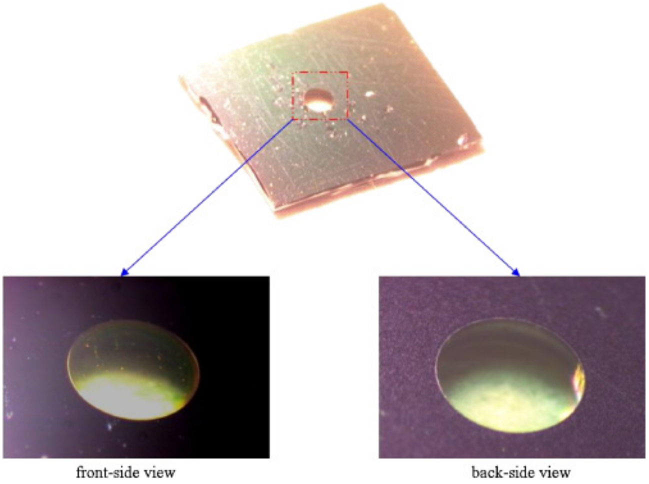


Fig. 4. (Color online) The photos to show the front- and back-side views of the bulge test specimen prepared using the presented process.

of transparent nitride film was the MP100-ME system (Labguide, with 1 nm readout). The interferometer system was used to measure the thickness of opaque metal films. The diameter of circular membranes can be obtained by measuring microscopes (Olympus STM6, with 0.1 µm readout).

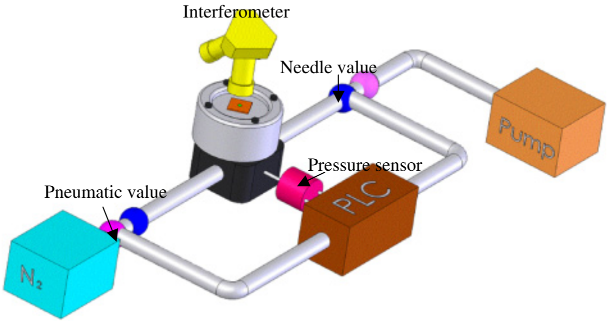


Fig. 5. (Color online) The experimental setup for bulge test.

As a comparison, the mechanical properties of thin film determined by a commercial nano-indenter (MTS) are also presented. The nano-indenter has a force resolution of 50 nN and a displacement resolution of less than 0.01 nm. A diamond tip of the Berkovich type was employed for the indentation test. In detail, the nanoindentation was performed by setting a 4 × 4 array test. There were sixteen experimental data for each thin film in this article. The average elastic modulus of thin films was obtained from these experimental data. The elastic modulus of the specimen, E, can be determined from the reduced modulus, E_r:[3)]

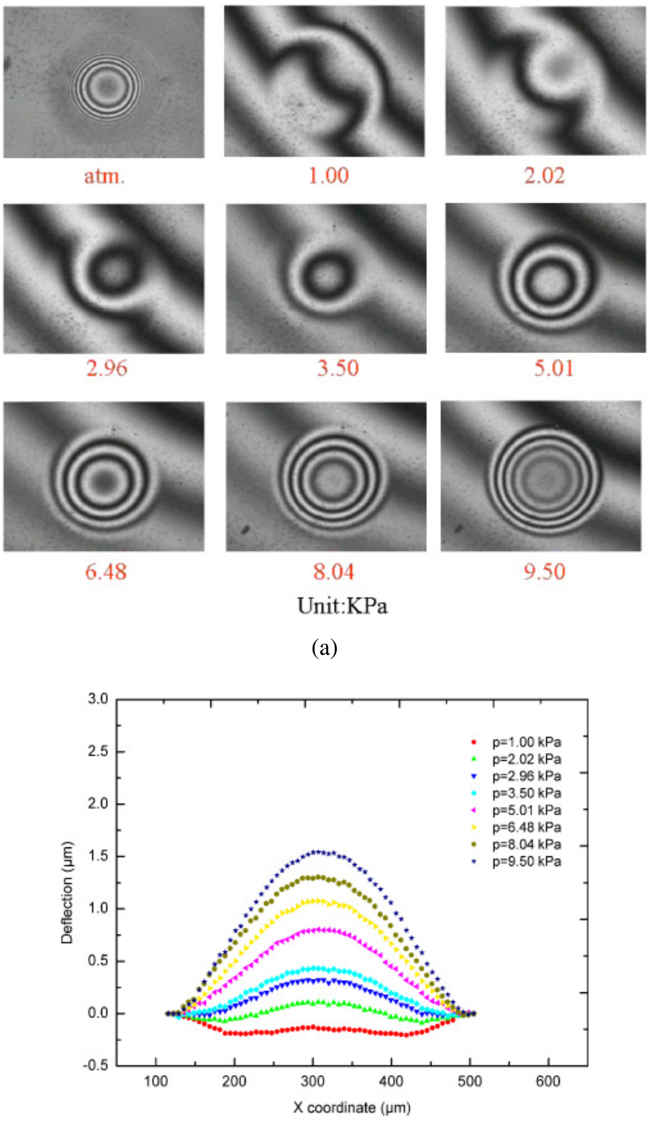
$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}, \tag{4}$$

where ν is the Poisson's ratio for the testing material; E_i and ν_i are the elastic modulus and the Poisson's ratio of the material of indenter, respectively. In this study, $E_i = 1141$ GPa and $\nu_i = 0.07$ are used for the diamond tip.

4. Results and Discussion

The corresponding relations between pressure and deformation profile of the fabricated circular membrane for Al, Au, and Si_3N_4 thin films are shown in Figs. 6–8, respectively. These figures also show the measured optical interference patterns of membranes. Thus, the maximum displacements of the membrane at different pressure loads can be obtained. As indicated in Fig. 6, the circular Al membrane has an initial deformation (as P at atm) due to the thin film residual stress. The deflection shape of Al film was influenced by its residual stress when $P < 5$ kPa. Therefore, the measurement results of Al film for $P < 5$ kPa were not satisfied with the relation of eq. (1). Similarly, the conditions of Au, and Si_3N_4 circular membranes during test can also be monitored from Figs. 7 and 8.

Figure 9 shows the measured deflection profile of Al, Au, and Si_3N_4 membranes. The deflection profiles predicted from Lin's and Timoshenko's models in eqs. (2) and (3) are also demonstrated. The results in Fig. 9 indicate that the Lin's model agree much well with the measured values. Thus, this study employs the Lin's model to extract the elastic modulus from the measured deflection amplitude h at different pressure load P. Figure 10 further shows the variation of the pressure load P and the deflection amplitude h of Al film. The three curves indicate the measurements of



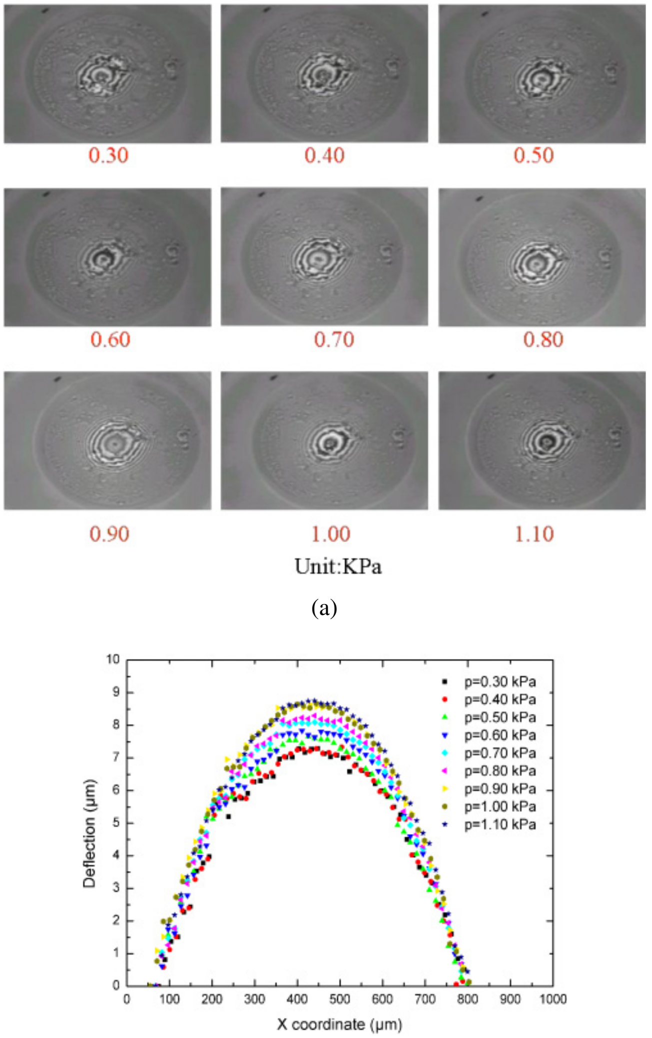
(a)

(b)

Fig. 6. (Color online) The variation of (a) interference fringe patterns, and (b) deflection profiles, of Al film under different pressure.

three specimens containing Al membrane of different diameter. In addition, Figs. 11 and 12 respectively show the measurement results for Au, and Si_3N_4 films. The elastic modulus of thin film was determined from the curve fitting of eq. (1) to the measurement results of in Figs. 10–12. According to Lin's membrane displacement function in eq. (3), the parameter C_1 in eq. (1) is determined as $C_1 = (7 - \nu)/3$. The elastic modulus of Al film was 121.72 ± 8.95 GPa, as the Poisson's ratio of 0.25. Similarly, the elastic modulus of Au film was 93.02 ± 5.15 GPa, as the Poisson's ratio of 0.42; and the elastic modulus of Si_3N_4 film was 250.92 ± 22.54 GPa, as the Poisson's ratio of 0.25. According to Lin's model, the elastic modulus is proportioned to $(1 - \nu)/(7 - \nu)$. As the Poisson's ratio has a 20% deviation (i.e. 0.25 ± 0.05), the extracted elastic modulus possess a near ±6% variation.

The nano-indentation test was used to confirm the measured elastic modulus. Figure 13 shows the typical loading and unloading curves of metal films. According to the measurements, the average elastic modulus of Al film was 118.87 GPa as $\nu = 0.25$ (from bulk material). The average elastic modulus of Au film was 92.35 GPa as $\nu = 0.42$ (from bulk material). According to eq. (4), the elastic modulus of thin film determined from this approach is proportioned to $(1 - \nu^2)$. As the Poisson's ratio has a 20% deviation (i.e., 0.25 ± 0.05), the elastic modulus extracted from eq. (4) has a less than ±3% variation.



(a)

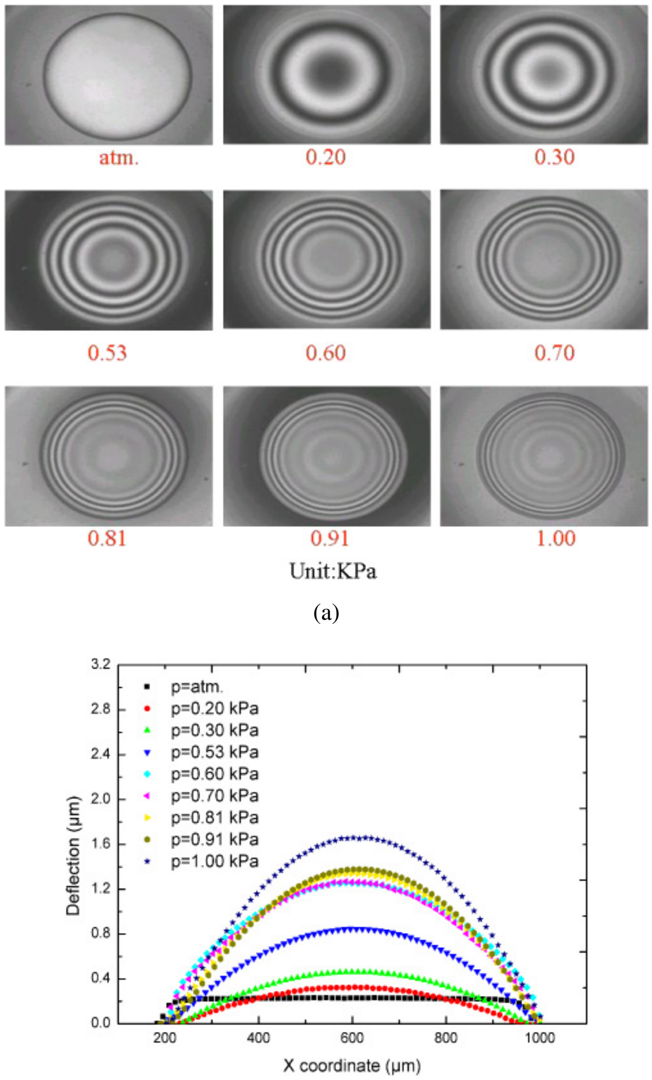
(b)

Fig. 7. (Color online) The variation of (a) interference fringe patterns, and (b) deflection profiles, of Au film under different pressure.

In comparison, the elastic modulus of Al thin film has a 2.34% deviation between the values determined from the bulge and nanoindentation tests when $\nu = 0.25$. Moreover, the elastic modulus of Au thin film has a 0.72% deviation between the values determined from the bulge and nano-indentation tests when $\nu = 0.42$. Despite another less than 10% deviations could be induced from the variation of the Poisson's ratio, the elastic modulus of metal thin film determined from these two approaches agrees well. In summary, the present dry etching release process is a feasible approach to implement circular metal membrane for bulge test.

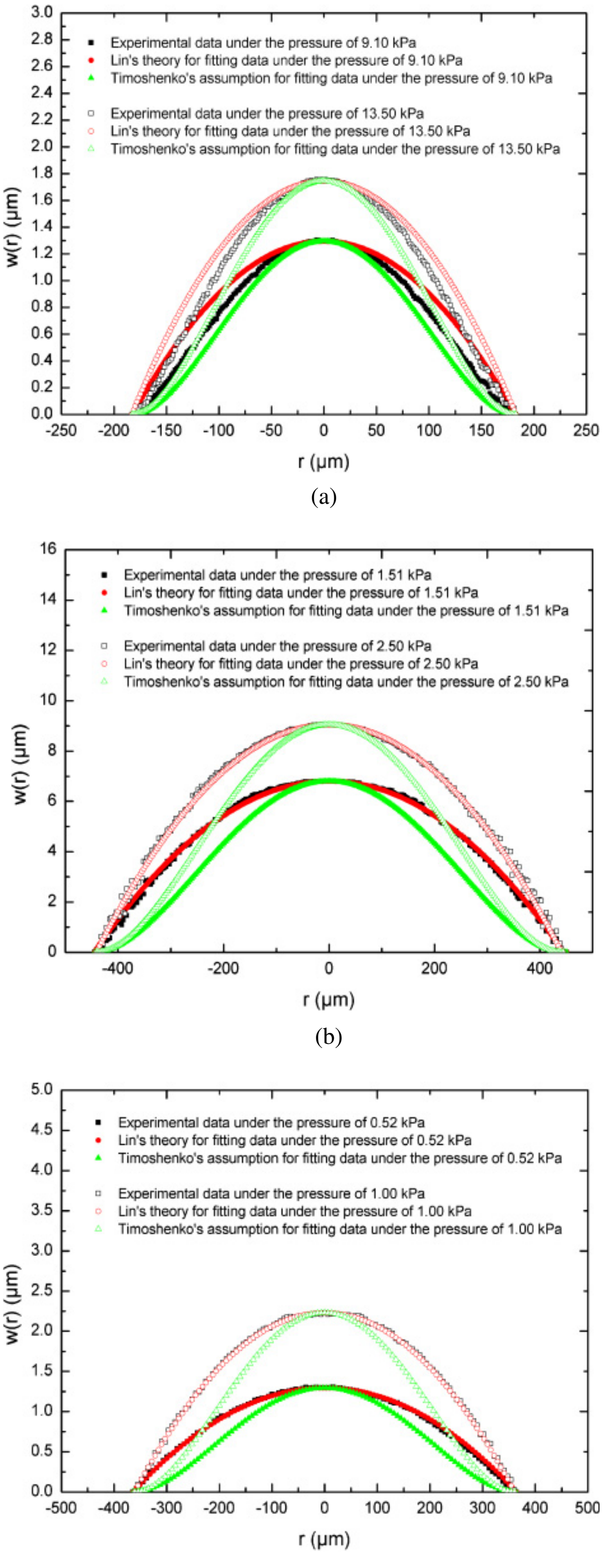
5. Conclusions

The main contribution of this study is to establish a new



(b)

Fig. 8. (Color online) The variation of (a) interference fringe patterns, and (b) deflection profiles, of Si_3N_4 film under different pressure.



(c)

Fig. 9. (Color online) Comparison of the measured and predicted deflection profiles of circular membranes by pressure load, (a) Al, (b) Au, and (c) Si_3N_4 thin film circular membranes.

fabrication process to manufacture the free-standing single-layer circular membranes of metal films. The process successfully combines the dry etching of DRIE and XeF_2 to release the test metal films. During the release process, the metal films are protected by the Si_3N_4 layer. As a result, the test metal films will not experience the ion bombardment by DRIE. In addition, this process is also designed to prevent the pre-deformation of the Si_3N_4 and the metal films before release. Thus, the mechanical properties of the test film will not be changed during the processes for specimen preparation. The circular Si_3N_4 test membrane can also be fabricated by changing the recipe of XeF_2 etching. In applications, the circular membranes of Al, Au, and Si_3N_4 films were fabricated and then characterized by bulge test. The results also confirmed with the measurements from nano-indentation test.

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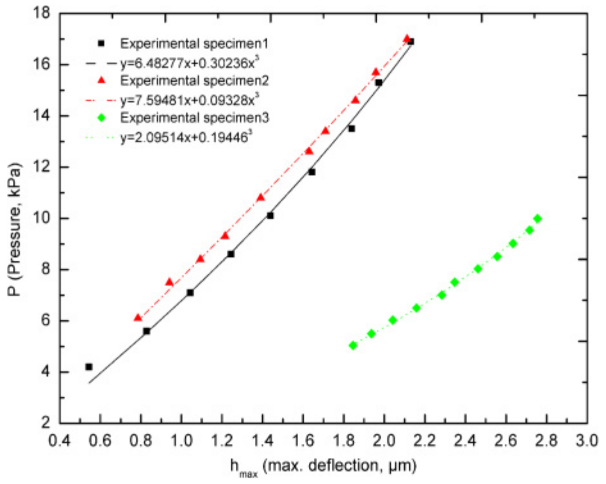


Fig. 10. (Color online) Variation of pressure and film-displacement for three Al film specimens during bulge test.

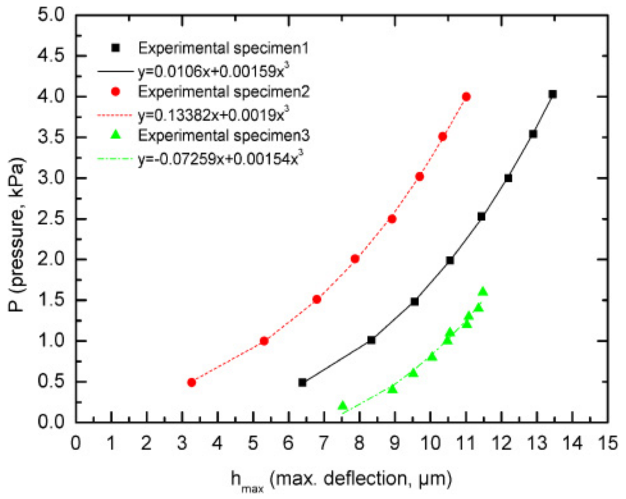


Fig. 11. (Color online) Variation of pressure and film-displacement for three Au film specimens during bulge test.

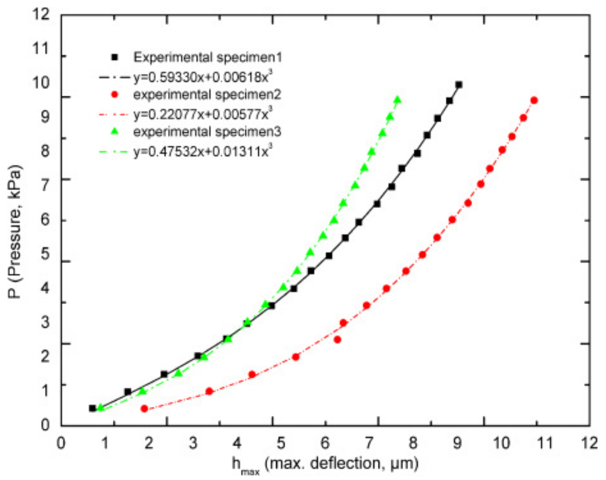
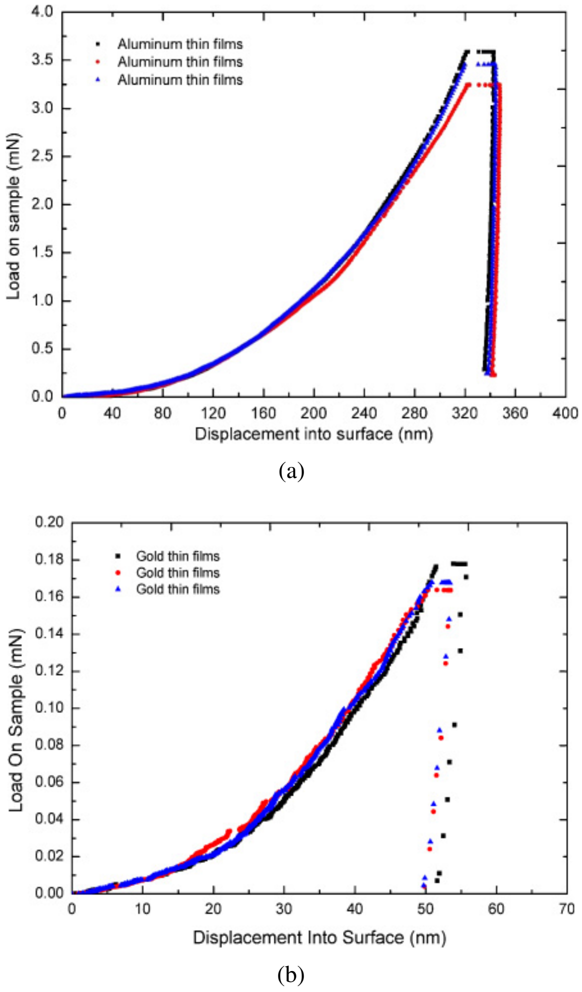


Fig. 12. (Color online) Variation of pressure and film-displacement for three Si_3N_4 film specimens during bulge test.



(a)

(b)

Fig. 13. (Color online) Typical load/displacement curves from nano-indentation tests: (a) Al film and (b) Au film.

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