Mechanical Integrity of Polysilicon Films Exposed to Hydrofluoric Acid Solutions

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This paper presents the results from a comparative study of Young's modulus, residual stress, and membrane burst pressure of undoped LPCVD polysilicon films exposed to various concentrations of hydrofluoric acid (HF). Load deflection measurements on square membranes of polysilicon with residual tensile stress were used to obtain estimates of Young's modulus, residual stress and burst pressure. The polysilicon membranes were exposed to four different solutions of the 49% by weight reagent HF including 10:1 DI water and HF, 1:1 DI water the HF, commercial 10:1 buffered oxide etchant, or pure HF (i.e. 49% by weight reagent). Two control groups were studied composed of membranes with no treatment and membranes exposed to DI water. Young's modulus changed from an average of 190 GPa for the control groups to an average of 240 GPa for films exposed to pure HF. Residual stress values exhibited a less pronounced and opposite change, with an average of 42 MPa for the control groups and an average of 27 MPa for films exposed to pure HF. Similarly, burst pressure was seen to decrease with increasing HF concentration, ranging in value from an average of 96.5 kPa (14 psi) for the control groups to an average of 34.5 kPa (5 psi) for films exposed to pure HF. It was found that the change in the investigated mechanical properties of polysilicon was approximately equal for HF:DI solutions of HF concentration above 10%. Furthermore, for solutions of equal HF concentration, the addition of the buffering agent decreases the effect on membrane burst pressures significantly.

Key words: Micromechanics, x-ray lithography, polysilicon, hydrofluoric acid

1. INTRODUCTION

Polysilicon is currently being utilized as structural material for such applications as micromechanical components, x-ray lithography masks, and sensors.1,2,3,4 Despite this extensive mechanical use of polysilicon, little is known about the effects of wet chemical processing on the mechanical properties of polysilicon. For example, a final step in the fabrication of polysilicon micromechanical components is the stripping away of the sacrificial silicon dioxide layers in a concentrated hydrofluoric acid (HF) solution. During our initial research on polysilicon micromechanisms,1 it was observed that the fracture strength of the structural polysilicon depended on the duration and concentration of the HF etch used to remove the silicon dioxide sacrificial layer, precipitating the study reported here.

This paper presents in detail the results of a comparative study of the effect of different solutions of the 49% by weight reagent HF ranging in concentrations of pure HF (i.e. 49% by weight reagent) to 10:1 DI water:HF on Young's Modulus, residual stress, and membrane burst pressure of undoped LPCVD polysilicon.5 Square membranes of polysilicon with residual tensile stress were fabricated using standard bulk-silicon micromachining techniques. The membranes were then exposed to one of four HF based solutions and tested to determine residual stress, Young's modulus, and burst pressure using standard load-deflection measurement techniques.5,7 The results were then compared with identically fabricated membranes which were not subjected to the HF soak. The mechanical properties of the membranes soaked in each solution and the control group were compared using statistical means.

2. LOAD-DEFLECTION METHOD

The load-deflection behavior of a flat square membrane with residual tensile stress has been previously examined.6,7,8 In the analysis of the load-deflection data obtained in this work, we have used a previously reported model.7 Figure 1 shows the geometry of the pressurized square membrane. In summary, the deflection, \( w \), of any point \( (x, y) \) on the membrane along the \( z \) axis is assumed to have the following shape:

\[
 w = h \cos \left( \frac{\pi x}{2a} \right) \cos \left( \frac{\pi y}{2a} \right). 
\]
Fig. 1 — Geometry of a pressurized square membrane.

where \( h \) is the membrane center deflection and \( a \) is the half-length of an edge. Assuming similar shapes for the in-plane displacements, \( u, v \), as

\[
u = C \cos \left( \frac{\pi y}{2a} \right) \sin \left( \frac{\pi x}{a} \right)
\]

(2)

and

\[
u = C \sin \left( \frac{\pi x}{a} \right) \cos \left( \frac{\pi x}{2a} \right),
\]

(3)

boundary and asymmetry conditions are satisfied.\(^7\)

Using energy minimization techniques the following relation is then developed for the membrane center deflection, \( h \), and the applied pressure, \( p \):

\[
p = \left( \frac{3t\sigma_e}{a^2} \right) h + \left( \frac{1.8Et}{a^4} \right) h^3,
\]

(4)

where \( t \) is the thickness of the membrane, \( \sigma_e \) is the residual stress, and \( E \) is Young’s Modulus. Poisson’s ratio, \( \nu \), has been assumed to be 0.25. For a known geometry membrane, Eq. (4) is fit to the load-deflection data to extract the residual stress and Young’s modulus.

A mechanical property of interest in this study is the fracture strain of polysilicon which is indicative of the mechanical integrity of the film. However, to estimate the polysilicon strain at burst, the location of the initial fracture in the membrane must be known. By recording the applied pressure at burst, as well as the location of fracture, the film’s fracture strain can be estimated by detailed structural finite-element analysis.

A high-speed video camera was used in an attempt to determine where fracture first occurs in the membrane at burst. However, even at 12,000 frames per second, it was not possible to determine the location where fracture originated. Therefore, we have selected to use the burst pressure of the membranes with similar geometries as the primary parameter for studying changes in the polysilicon mechanical integrity resulting from HF exposure. As a result, an implicit assumption is that the fracture process is always the same in the tested membranes. In addition, using the value of membrane deflection preceding burst and the pressure at burst, an average strain at fracture is calculated using standard geometrical methods to determine arc length strain along the meridian at fracture as

\[
\epsilon_f = \frac{2a(1 + \frac{h^2\pi^2}{16a^2}) - 2a}{2a} = \frac{h^2\pi^2}{16a^2},
\]

(5)

where \( \epsilon_f \) is the strain at fracture and \( h_f \) is the membrane center deflection just before fracture.

3. SAMPLE FABRICATION

The fabrication process used to form the membranes for this study is briefly described here. A more detailed discussion of sample fabrication is documented elsewhere.\(^10\) A film sandwich consisting of 2500Å silicon nitride, 4 µm undoped polysilicon, and 2500Å silicon nitride was deposited by LPCVD on single-side polished, lightly boron-doped <100> silicon substrates. The nitride was grown by reaction of SiH₂Cl₂ and NH₃ at 800 °C and 300 mTorr. The polysilicon was deposited by pyrolysis of 100% silane and hydrogen at 630 °C. By the nature of the LPCVD process used, the nitride/polysilicon/nitride sandwich was deposited on both sides of the substrate.

LPCVD polysilicon films deposited in processes similar to that described above are typically under compressive stress. In our initial samples, the compressive stress in the polysilicon lead to the buckling of the membranes when the highly tensile-stressed nitride films were removed from the two sides of the membrane. This buckling complicated the load-deflection measurements as well as analysis of the measurement data. We found that it was possible to cause a stress reversal in the polysilicon film by the addition of a 90 minute anneal at 1000 °C in a nitrogen environment, after the second silicon nitride film had been deposited. The annealed polysilicon films exhibit a residual tensile stress near 30 MPa measured by load-deflection on associated membranes. This type of anneal procedure was demonstrated by Guckel\(^11\) to cause a reversal of stress type for low-temperature (580 °C) LPCVD polysilicon. Similar anneal conditions were used by Howe\(^12\) to reduce residual stress to near zero in LPCVD polysilicon deposited at 630 °C but did not result in a stress reversal.

Once annealed, the front side of the wafer was coated with a 7 µm-thick film of photoresist in order to protect this surface from mechanical damage during the patterning of the backside films. The backside nitride/polysilicon/nitride layers were patterned in an SF₆ plasma to define the membrane mask. The wafers were then anisotropically etched in a 20% by weight KOH solution at 80°C to form the nitride/polysilicon/nitride membranes on the front. Finally, the nitride layers encasing the polysilicon were stripped in pure HF for 30 min, resulting in 3 mm × 3 mm, 4 µm-thick polysilicon membranes with low residual tensile stress. Note