Determination of mechanical stress in the silicon nitride films with a scanning electron microscope

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Abstract

A method to measure mechanical stress in thin films was proposed. Method is based on the geometry variance of thin film's fragment after it's been released from substrate. A scanning electron microscope (SEM) was used to measure linear dimensions. Samples were prepared with help of focused ion beam (FIB). Mechanical stress of silicon nitride thin film measured using our method is -1.64 GPa, relative measurement error estimated as 1.2%. Measured value of stress correlates with other method's existing data. Method can be applied to various materials, that are being used in MEMS technology.

Key words: mechanical stress, SEM, profilometer, surface morphology, MEMS, FIB, silicon nitride.

1. Introduction

Micromechanics or so-called microelectromechanical systems (MEMS) are rapidly developing nowadays [1]. A mechanical stress that appears during IC and MEMS-based devices' fabrication, has a strong influence on device reliability and dynamic characteristics [2, 3].

Existing techniques of mechanical stress measurement are based on wafer bending detection (such as profilometry [2]), or evaluation of lattice parameters (x-ray diffractometry, transmission electron microscopy).

In this paper we suggest a method of determining a value of mechanical stress in thin films of various materials, that is based on visual analysis of thin film fragment after it's been released from substrate. Algorithm for determining stress in MEMS structures with a SEM includes developing layout, preparation of structures, measuring linear dimensions of film fragment before and after removal of the substrate, and stress analysis by Hooke's law.

Layout and its controlled dimensions are shown on picture 1.

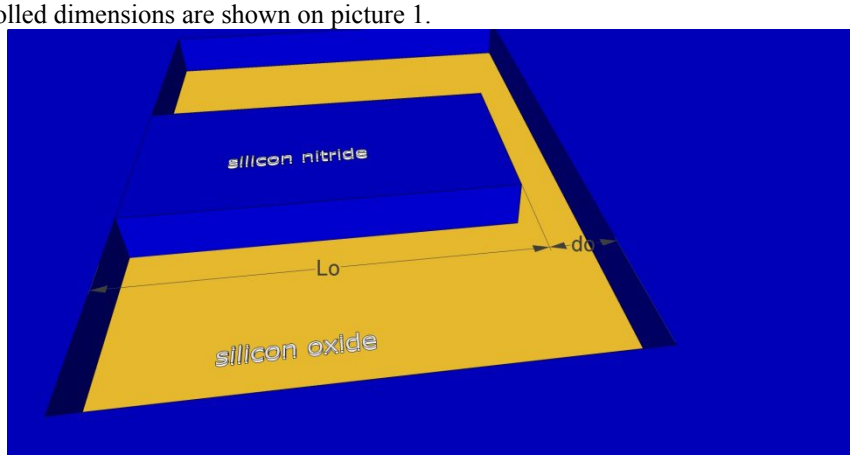

Figure1. Maket of balk structures.

Balk was chosen as a thin film fragment, and its dimensions should be as large as possible, but not larger than the maximum value required for SEM measurement within one field size. Also, the balk structure should survive the process of substrate etching.

From [4] it can be seen that the average value of compression stress in silicon nitride film fabricated by PECVD processes is -1.25 GPa, according to profilometry data. Two-axial modulus constant $E_f/(1-\mu_f)$ of silicon nitride film is about $1.9 \cdot 10^{11}$ Pa [5].

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After substrate removal, part of the film is being released and structure will stretch or constrict depending on mechanical stress sign [6]. Therefore, gap between the periphery and the edge of a balk will decrease or increase respectively, so the calculation of mechanical stress may be done with help of this formula:

$$\sigma = -\frac{L - L0}{L} \cdot \frac{E_f}{l - \mu_f} \tag{1}$$

where L is length of balk after substrate removal, L_0 is initial length of the balk, E_f is Young's modulus of the film, μ_f is Poisson's ratio of the film.

Value of L_0 can be expressed from formula 1. We have estimated minimum value of initial balk length on the basis of FEI Quanta 3D FEG dual beam system restrictions. Initial balk length is better to be 10 um or more. In this work initial balk length is 69.5 um. According to formula 1, value of stretching will be about 460 nm, and hence, will be easily measured using SEM technique.

Thereafter, an estimation of measurement accuracy was performed. Consider mechanical stress σ a function of two variables. It is known, that absolute error during indirect measurements (i.e. when value is calculated from known dependency between this value and values that are experimentally measured) is defined from local derivatives on each experimentally measured value. Absolute measurement error can be expressed as:

$$\Delta\sigma(L, Lo) = \sqrt{(\{(-\frac{L-Lo}{L})\cdot\frac{E_f}{1-\mu_f}\}'_{Lo} \cdot \Delta Lo)^2 + (\{(-\frac{L-Lo}{L})\cdot\frac{E_f}{1-\mu_f}\}'_{L} \cdot \Delta L)^2} \tag{2}$$

Moving two-axial modulus from square root and simplifying the equation gives

$$\Delta\sigma = \sqrt{(\frac{1}{L}\cdot\Delta\mathrm{Lo})^2 + (-\frac{Lo}{L^2}\cdot\Delta\mathrm{L})^2}\cdot\frac{E_f}{1-\mu_f} \tag{3}$$

Considering that $\Delta L \approx \Delta Lo$, we have

$$\Delta\sigma = \sqrt{2}\frac{\Delta L}{L}\cdot\frac{E_f}{1-\mu_f} \tag{4}$$

Formula 4 was obtained considering that $\Delta L \approx \Delta Lo$, $L \approx Lo$ and $\sqrt{\frac{L_0^2 + L_1^2}{L_0^2}} \approx \sqrt{2}$.

To increase method precision, one should measure not the balk length change itself, but the gap between periphery and the edge of the balk. Considering an operator's measurement error being 1 pixel, we have ΔL about 5 nm with gap size of 5 um. Calculations according to formula 4 give a relative measurement error of about 1.5% or less with (L-Lo)~0.5 um.

2. Experiment

A silicon substrate with 0.6 um thermal oxide (SiO_2) was prepared. Then a silicon nitride film with 0.13 um thickness was deposited by low pressure chemical vapor deposition (LPCVD) technique. After that we performed a photolithography process on the back-side of wafer and plasma-chemical etching (PCE) of back-side silicon nitride. Finally, we used PCE technique to remove SiO_2 and Si from back-side until we have reached front-side SiO_2. Roadmap of described process is illustrated on picture 2.

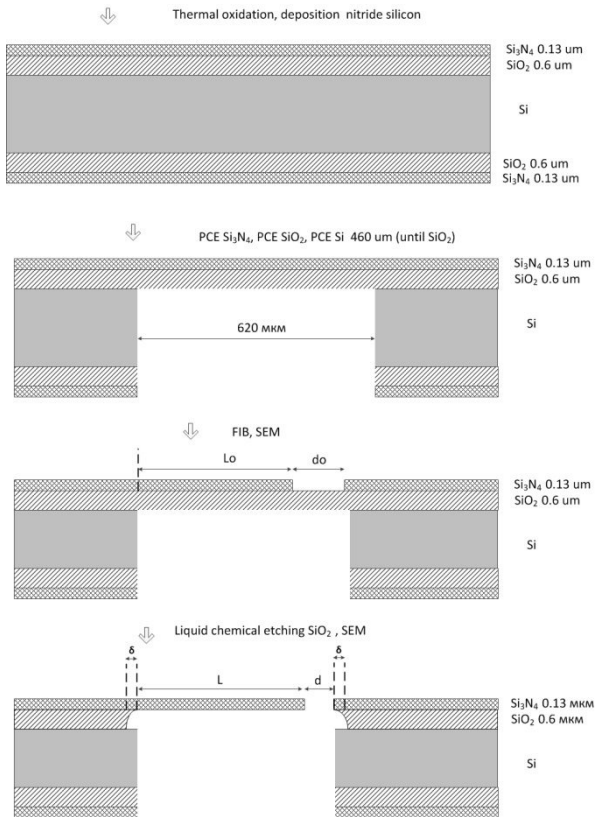

Figure 2. Roadmap of technological process

As a result, silicon walls of "wells" are almost ideally vertical, which allows seeing transparent isolator film with an eye. Silicon nitride on the front-side was etched by FIB, forming a balk 69.47 um long and 44.83 wide (picture 3).

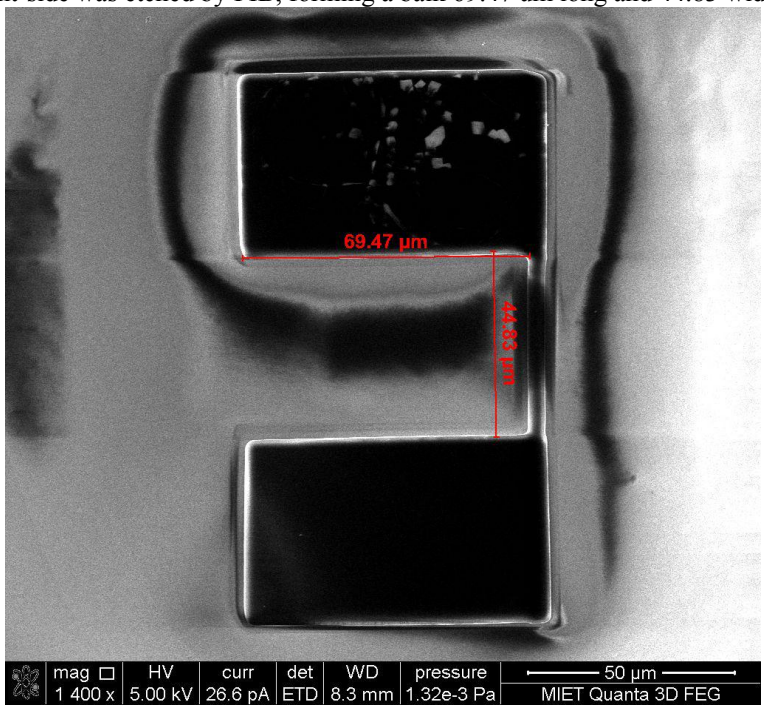

Figure 3. Geometry of balk

Thereafter, a SEM measurement of the gap on front-side of wafer was performed (picture 4). Initial gap length is 4.89um.

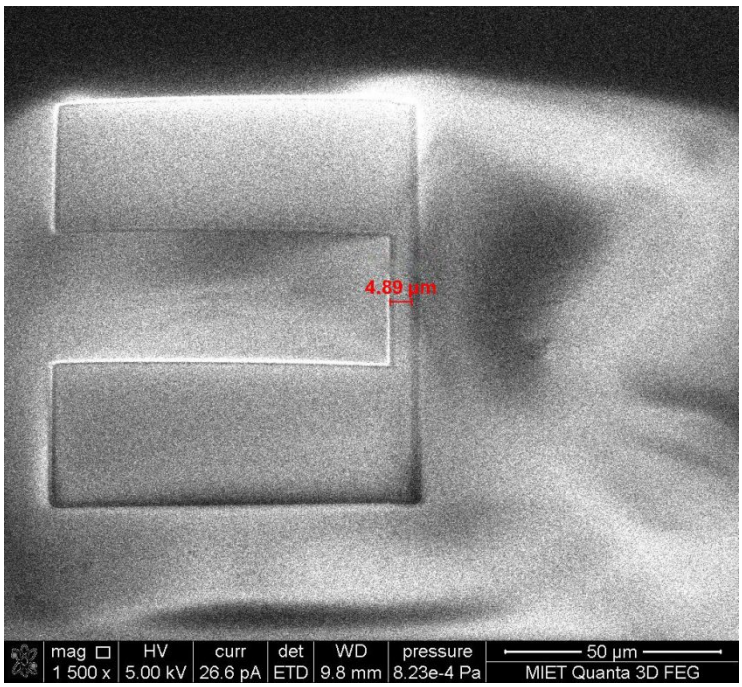

Figure 4. Determination of initial gap between edge balk and the periphery

To release silicon nitride, wafer was etched in hydrofluoric acid (HF) in order to remove SiO_2 on the front-side. It is worth mentioning that the value change of δ caused by wet chemical etching process is negligible, because the relation between gap length and balk length is less than 0.01. After silicon nitride film was released, we have repeated a SEM measurement of the gap (picture 5).

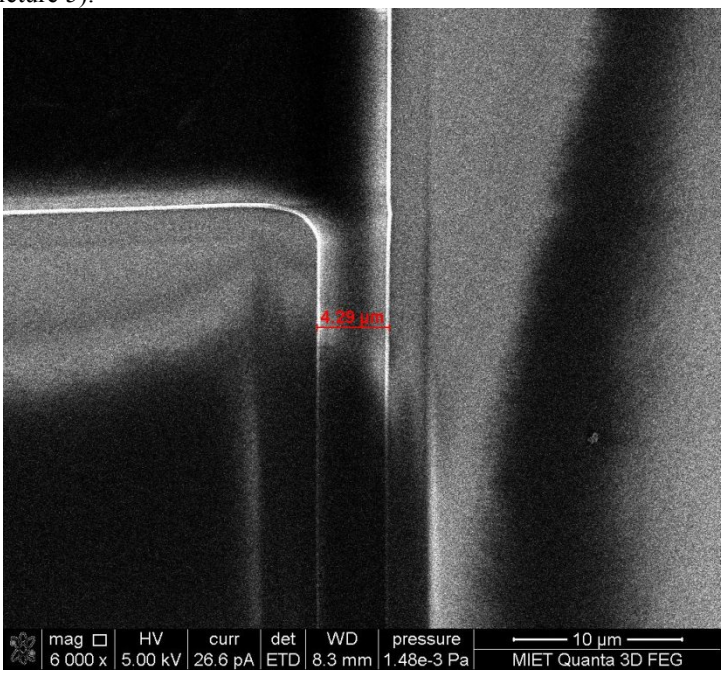

Figure 5. Repeated determination gap between edge of the bulk and the periphery after etching SiO_2

Picture 5 shows that the gap was reduced by ~0.6 um after silicon nitride was released.
Calculated value of mechanical stress according to formula 1 is -1.64 GPa. Absolute measurement error of mechanical stress according to formula 3 is about 20 MPa. Therefore, relative measurement error is 1.2%.
With help of optical profilometer Veeco Wyko NT9300 we have analyzed surface morphology of the balk, which is illustrated on picture 6.

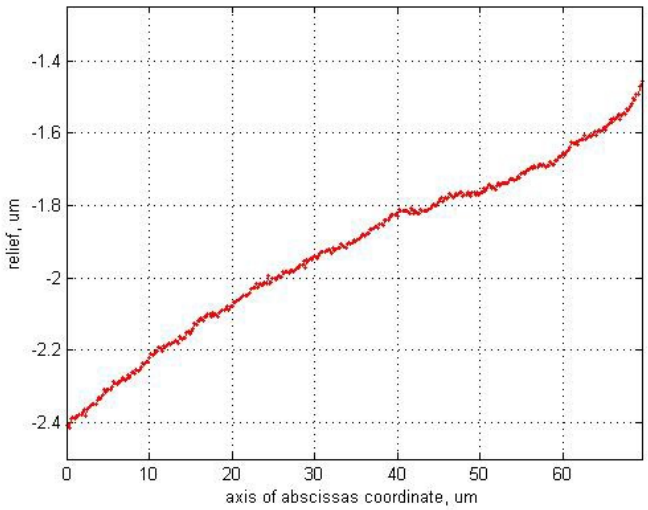

Figure 6. Relief of balk surface

From picture 6 it can be seen that free edge of the balk is curving upwards. Besides that, the value of curvature is proportional to the distance to fixed edge of the balk. Overall deflection is about 1 um.
Next we have estimated the deflection influence on the accuracy of our balk length measurements. Consider the right-angled triangle, where hypotenuse is 69.47 um and height equals to deflection (i.e. 1 um). Difference between hypotenuse and bigger leg is about 10 nm, which is at the order of SEM linear measurement error. Hence, silicon nitride balk curving is not sufficient for described method.

3. Summary

A method of mechanical stress measurement was developed. It can be applied to various materials in MEMS technology; approvement of method was performed on silicon nitride example. There are several advantages that used technique of preparing test structures is providing: time for chemical etching of silicon is significantly reduced with help of replacing of wet etching [7, 8] by plasma-chemical etching; localizing the area of investigation using focused ion beam.
A value of structure deformation was measured using SEM technique. Calculated value of mechanical stress in silicon nitride is -1.64 GPa with relative measurement error at 1.2%. Value of mechanical stress obtained with this method correlates well with data, that was indirectly measured on profilometer [4]. It is established that silicon nitride balk curving can be neglected while measuring length of the balk.

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