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Fabrication of micro-nano-mechanical structures for sensing application

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Abstract

In this work, we presented the studies on mechanical properties of polysilicon, silicon nitride and silicon dioxide thin films prepared by low pressure chemical vapour deposition (LPCVD) and plasma enhanced chemical vapour deposition (PECVD) methods. Cantilevers were fabricated from these films using surface micromachining technique. Deflection of fabricated cantilevers were studied as a function of applied load. It was found that silicon dioxide film has low Young's modulus whereas silicon nitride film deposited by LPCVD method has highest Young's modulus. Deflection of fabricated microcantilever using silicon dioxide film was high due to self-loading or residual stress but silicon nitride based cantilever has zero deflection at equivalent length scale. Later on piezoresistor of polysilicon film was integrated on silicon nitride cantilever's surface for trace explosive detection after proper functionalization.

Keywords: Microcantilever, Micromachining, Sensitivity, Piezoresistor, Functionalization

INTRODUCTION

Integration of microcantilever sensors and readout with microprocessing technology is an important area of research in microelectronic industries. Detection and quantifying of harmful chemical present in water or air [1, 2], explosive [3, 4], biological detection of virus [5-8], antigen, DNA [9-12] or other protein [12-18] is possible using micro- and nano- scale sensors with appropriate chemical functionalization. Various static deflection detection techniques such as piezoresistive, piezoelectric, capacitive, magnetic or shift in resonance frequency measurement are used. It is possible to demonstrate the functioning of many devices such as nano-mechanical resonator working in microwave frequency range [19], single electron spin detection [20], measurement of mass in zeptogram scale [21] and detection of mechanical motion near the quantum limit [22] using microelectronic fabrication techniques. These achievements demonstrate the unique characteristics of nano-electro-mechanical systems (NEMS) due to nanoscale dimensions. This miniaturization of MEMS to NEMS is still an emerging research field and promise for detection of gases, chemicals or biological entities with superior performance.

Fabrication of microcantilevers with optimized length and thickness are primary issue in microelectronic industries. The chemical functionalization of fabricated cantilevers is another important issue. Therefore, the studies on mechanical properties of deposited films as well as cantilevers are performed in this present work. To sense cantilever deflection during detection polysilicon piezoresistors were fabricated on top surface of cantilevers made from LPCVD silicon nitride film only. These microcantilevers can be used to detect any harmful chemical, virus, antigen, DNA after proper chemical functionalization [5-18, 23]. In the present work, surface of cantilevers was modified by functional layer of 4-mercaptobenzoic acid (4-MBA) to detect a specific analyte 2,4,6-trinitrotoluene (TNT) vapors.

EXPERIMENTAL

Both LPCVD and PECVD techniques were used to deposit films of polysilicon, silicon nitride and silicon dioxide on silicon wafers. The details of methods are reported in previous work of authors [24-25]. Process parameters were optimized for achieving the low residual stress in deposited films. Mechanical properties of deposited films were studied by Hysitron T1-950 Tribo Indenter at IIT Ropar.

The curvature of each wafer used was first measured using non-destructive optical technique called k-Space MOS (Multi-beam Optical Sensor) and saved as reference data. After deposition of thin films, the curvature was again measured. The change in curvature was converted into stress by in-built software.

METHODS

A Berkovich indentation test was performed on deposited films before patterning or etching. A nanoindenter tip was pressed onto the film and withdrawn. The slope of load versus penetration depth curves under reducing load condition was used to estimate the reduced modulus (E_r) which is related to Young's modulus (E) as given in equation 1 and results are given in Table 1:

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

In the above equation ν represents the Poisson's ratio of the deposited film which were taken from literature, E_i is Young's modulus of the diamond indenter tip (1100 GPa) and ν_i is Poisson's ratio of diamond (0.07).

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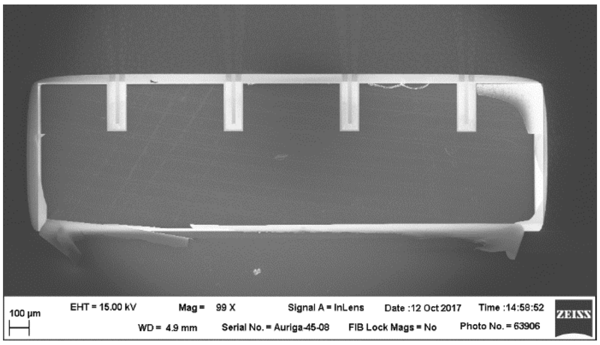

Figure 1.SEM image of LPCVD silicon nitride cantilevers

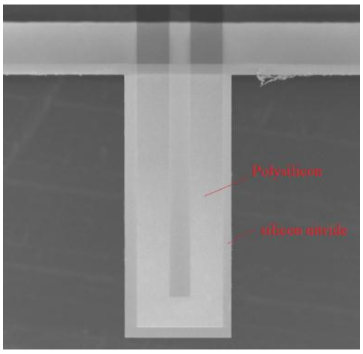

Figure 2. Enlarge SEM image of single cantilever

Surface micromachining as well as bulk micromachining techniques were used to fabricate the microcantilevers. For comparison purpose four cantilevers of same dimensions were fabricated using silicon nitride (LPCVD & PECVD), silicon dioxide (PECVD) and polysilicon (LPCVD) films. Only LPCVD silicon nitride based cantilever was embedded with polysilicon piezoresistors on top surface for sensing application in standard clean room environment. SEM image of these microcantilevers is shown in figure 1. The enlarge image of single cantilever is shown in figure 2. These images confirm that cantilevers are straight with no roughness on the surface. The surface of this cantilever was further modified by functional layer of 4-mercaptobenzoic acid (4-MBA).

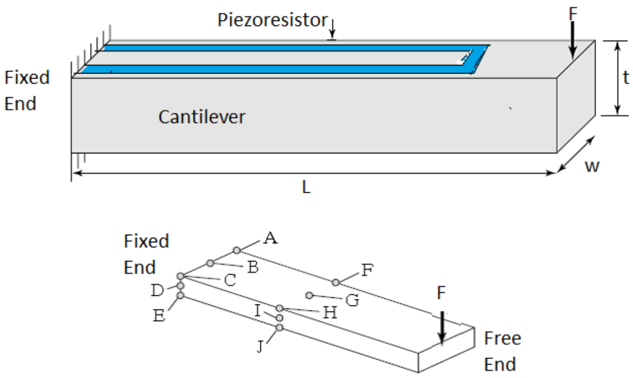

Figure 3. Cantilever dimension with piezoresistor on top surface and stress points

Figure 3 shows the maximum stress point when a concentrated transverse load is applied at free end of microcantilever by the nanoindenter. The torque (M_x) will be zero at the free end and maximum at fixed end. Due to this piezoresistors are universally found on the surface of a cantilever and near the fixed end. The deflection (d) due to point load can be found from Eq. (2) and maximum strain (ε_{max}) due to this load can be calculated from equation (3):

$$d = \frac{FL^3}{3EI}(1-\upsilon) \quad (2)$$

$$\varepsilon_{max} = \frac{M_x t}{2EI} = \frac{FLt}{2EI}(1-\upsilon) \quad (3)$$

Here, I is the moment of inertia. In the above figure, the points A, B, C and E have highest stress. Top surface points A, B, and C are under tension while bottom surface point E is under compression.

RESULTS AND DISCUSSION

Measurement of mechanical properties of films

Table 1 shows Young's moduli and residual stresses of various thin films at optimized process parameters. It was found that LPCVD silicon nitride have highest Young's modulus and PECVD silicon dioxide have least.

Table 1. Mechanical properties of various films at optimized process parameters

Properties	**Silicon nitride (LPCVD)**	**Silicon nitride (PECVD)**	**Silicon dioxide (PECVD)**	**Polysilicon (LPCVD)**
Thickness (µm)	1.0	1.0	1.0	1.0
Deposition temp. (°C)	800	400	400	625
Residual stress (MPa)	-24	18	-28	-24
Young's modulus (GPa)	235	132	56	156

Figures 4 and 5 show the nanoindenter tip force versus penetration depth of PECVD and LPCVD nitride films. It was found that LPCVD nitride film has higher stiffness and Young's modulus compared to PECVD nitride film.

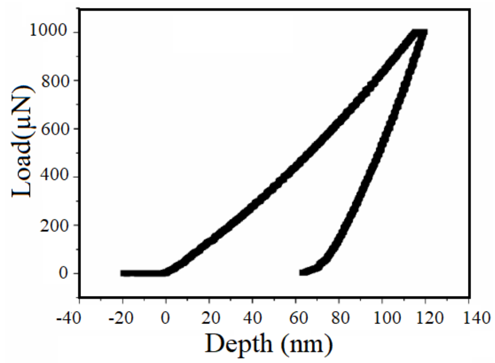

Figure 4. Plot of load versus depth for PECVD nitride film

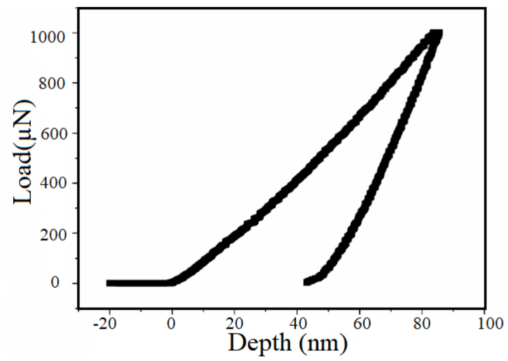

Figure 5. Plot of load versus depth for LPCVD nitride film

Influence of residual stress on cantilevers

Figure 6 shows the deflection of fabricated cantilevers as a function of their length which are due to presence of residual stress or self-loading. It can be concluded from this figure that LPCVD silicon nitride film is the best candidate with length below 200 µm for getting minimum deflection under free standing condition. Therefore, this film was chosen for fabrication of sensor. Other advantages of nitride films are its moisture and chemical resistant. On the other hand, polysilicon film based resistors require thermal treatment to remove defects occurred due to implantation.

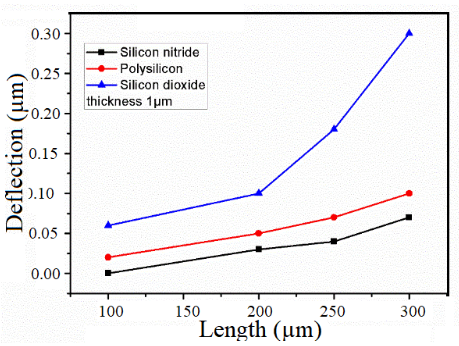

Figure 6. Deflection of cantilevers as a function of length

Measurement of mechanical properties of cantilevers

Load versus deflection curves of various cantilevers having same geometrical dimensions were measured by nanoindenter. These results were compared with calculated deflection using Eq. (2) and fitted in figure 7; here Young's modulus (E), value was taken from Table 1.

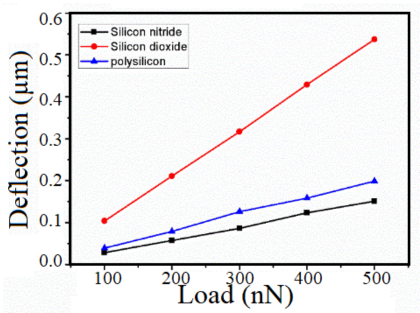

Figure 7. Deflection of various cantilevers as a function of applied load

From figure 7 it is clear that LPCVD nitride cantilever have least deflection, and hence the fracture strength will be high. This will encounter stiction problem arises during etching and developing processes. These cantilevers (150x100x1 µm^3) were integrated with piezoresistors on surface and tested for load (F) upto 4.7 µN. A deflection (d) of 2.2 µm was measured as shown in figure 8. Its stiffness (k) is 2.13 µN/µm (maximum load/deflection) and Young's modulus (E) as calculated from Eq. (4) is 288 GPa.

$$k = \frac{F}{d} = \frac{Ewt^3}{4L^3} \tag{4}$$

The maximum strain (ε_{max}) and stress (σ_{max}) as calculated from Eq. (3) and (5) are found 0.00019 and 56.4 MN/m^2 respectively.

$$\sigma_{max} = \frac{Mt}{2I} = \frac{FLt}{2I} = \frac{FLt}{2\frac{wt^3}{12}} = \frac{6FL}{wt^2} \tag{5}$$

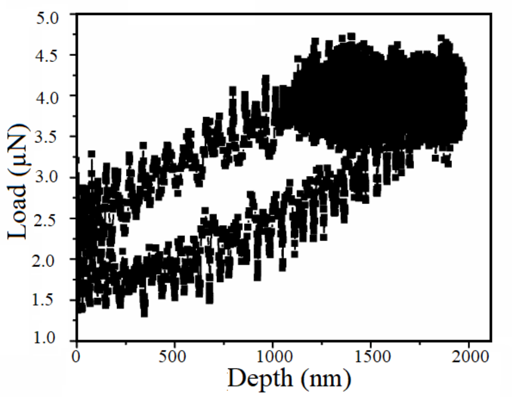

Figure 8. Load vs deflection curve of LPCVD nitride cantilever integrated with piezoresistor

Measurement of resistivity

The resistivity (ρ) of integrated piezoresistors on cantilevers with known length-width was calculated from following equation [26]:

$$\rho = \frac{t_R W_R R_A}{L_R} = \frac{W_R \ \ R_S}{L_R} \tag{6}$$

where R_A, L_R, W_R, t_R and R_S are the average resistance, length, width, thickness and resistance of piezoresistors respectively. Higher the length and resistance of piezoresistor, higher will be the change in resistance on application of stress/pressure/mass. Therefore, U-type polysilicon piezoresistor was fabricated for gaining maximum length and its resistance (R_S) was calculated as 0.5 MΩ.

Measurement of sensitivity:

Figure 9 shows the packaged piezoresistor embedded cantilevers for testing and figure 10 shows the characterization results, from which cantilever sensitivity (S) can be expressed according to below equation:

$$\text{Sensitivity} = \frac{\text{Output change}}{\text{Load}} = \frac{\frac{\Delta R}{R}}{F}$$

In the present case, change in resistance and change in voltage were measured as 120 Ω and 580 μV respectively after application of load. Sensitivity of designed cantilever against change in resistance is calculated as 5.1 x 10^{-5}/μN.

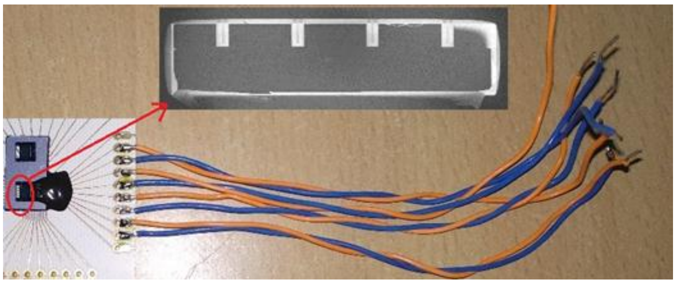

Figure 9. Fabricated device for testing

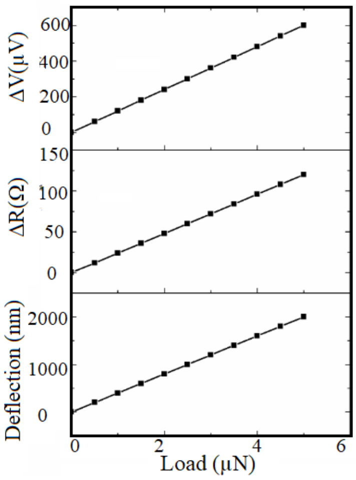

Figure 10. Characterization result of piezoresistor embedded cantilever

Detection of TNT vapors

Time response is an important parameter of cantilever sensors for trace detection of explosive [23, 27-32]. Figure 11 shows the test results of piezoresistive cantilever coated with a 4-MBA functional layer. Maximum output voltage measured was 480 μV and corresponding change in resistance was 96 Ω when exposed to TNT vapors in chamber. Output voltage saturates after ~27 seconds and hence this is considered as response time. Further, sensor chamber was flushed with dry nitrogen and TNT desorb within 140 seconds. Change in voltage to zero after a flush in time of 140 seconds indicates that TNT is completely desorb and ready for further sensing as shown in figure 11.

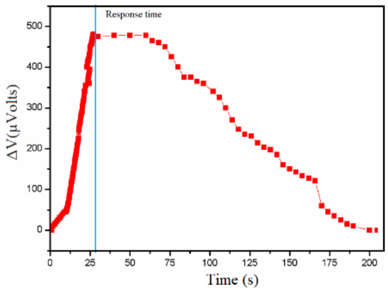

Figure 11 Time response curve of functionalized cantilever sensor

CONCLUSIONS

In the present work, it is concluded that LPCVD nitride film is the most suitable candidate for fabrication of microcantilever sensor as it has higher stiffness and Young's modulus compared to other films. These piezoresistor incorporated microcantilever sensors are simple to detect, easy to fabricate, compact, portable, cheap and reliable. Their surface can be modified for any specific analyte detection. Array of cantilevers may be connected in series to increase surface contact area and sensitivity. Output of the sensors can be amplified, integrated with readout display using microelectronic fabrication technology.

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CONFLICTS OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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