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EFFECT OF PECVD CONDITIONS ON MECHANICAL STRESS OF SILICON FILMS

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Abstract. In the work, silicon films were obtained from SiH_4 by plasma-enhanced chemical vapors deposition. The influence of deposition temperature (200 – 650°C) and gas mixture pressure (500 – 2000 mTorr, Ar/SiH_4) on the mechanical stresses in the films were studied by stylus profilometry and curvature method. Mechanical stresses in films with thickness changing from 270 nm to 1.93 μm are in the range of –750 to +250 MPa. The deposition rates were 7 – 46 nm/min irrespective of temperature at 500 mTorr. Temperature dependences had inflection close to 450°C. Low-stressed (tensile and compressive) and stress-free films can be formed and qualified for solar cells fabrication, based on textured silicon, glass or flexible substrates as well as for micro- and nanomechanics, particularly relatively thick films of 1 – 2 μm, obtained at 2000 mTorr and rates about of 50 nm/min.

Keywords: mechanical stress; silicon film; SiO_x; PECVD; profilometry.

1. Introduction

Silicon is an important electronic and structural material for microelectronics, particularly solar cells, and micro-electromechanical systems [1 – 4]. In these applications, mechanical properties play a key role in determining the devices performance. Therefore, their characterization for silicon films is of great importance, especially for MEMS [2, 5] and flexible electronics and solar cells [2, 6], and has been a long-term interest for research [1 – 16]. Mechanical properties of silicon films significantly depend on fabrication conditions in both deposition or growth and post thermal processing [2 – 4, 15].

Silicon films are typically deposited by chemical vapor deposition at low pressure (LPCVD) or plasma enhanced (PECVD) as well as magnetron sputtering [3 – 5, 10]. Among them PECVD is more interesting since allows one to deposit films at low temperatures with higher rates [15]. Specifically, amorphous, nano- and polycrystalline silicon films obtained by PECVD for thin film solar cells and micromechanical structures [3, 5, 10, 17, 18]. Moreover, single-crystal silicon as well as glass and polymer flexible films can be used as a substrate at low temperatures (about 100 – 300°C) [2, 3, 5, 6, 8, 10, 14]. Presence of undesirable residual stress in the film leads to generation defects, and, as a result, decrease in efficiency, significantly changes productivity, reduces structural integrity, including cracking of the film or even substrate, durability, reliability and dynamic characteristics devices [2, 4, 7, 8, 14].

Residual stresses can be described as stresses that remain in the material after manufacturing in the absence of external forces and thermal gradients. In other words, the residual stresses depend on the parameters of the manufacturing process. Residual stresses in PECVD films depend mostly on flow and composition of gas mixture, its pressure and

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deposition temperatures [3, 8, 10]. Studies of these parameters on the residual stresses for silicon, various oxides, nitrides and carbides of silicon and other materials have been performed [2, 5 – 7, 9 – 13]. However, there exist few papers on residual (intrinsic) stresses in PECVD silicon films, deposited at low-rf power. Moreover, the stresses in films, deposited at 10 W, have not been investigated (such studies have not been identified).

Several methods are used to measure mechanical stresses: curvature measurement, bulge test, buckling and vibration method, as well as Raman spectroscopy [2 – 6, 18 – 20]. However, only curvature analysis of profiles, obtained by profilometry, allows determining the residual stresses in films require no forming additional elements or a laborious system of result interpretation.

The aim of this work is to present average mechanical stress of silicon films, deposited by plasma enhanced chemical vapor deposition from monosilane and to determine the effect of deposition temperature and total pressure by using curvature measurements.

2. Experimental technique

In our experiments, silicon films were prepared from gas mixture of silane (SiH_4) and argon (Ar) using plasma-enhanced chemical vapor deposition (PECVD, PlasmaLab 100, 13.56 MHz [17 – 19]). The *n*-type (100) silicon wafer of 60 mm diameter was used as substrate. It was covered with a layer of silicon oxide (SiO_x) of 1.145 µm thick. The SiO_x films were obtained by PECVD at temperature 350°C, pressure 1000 mTorr, power 20 W and time 16 min 46 sec. Then silicon films were deposited on the SiO_x/Si(100) structures under the conditions, presented in Table 1. Gas ratio, gas flow and RF power were constant and equal 9:1, 500 sccm and 10 W, respectively. Thickness of the deposited films was determined by the scanning electron microscopy using focused ion patterning (Nova NanoLab 600) [18].

Table 1. Silicon film deposition parameters.

Samples	Temperature, C	Pressure, mTorr	Rate, nm/min
Sample 1	650	500	7.67
Sample 2	650	2000	45.83
Sample 3	450	500	6.69
Sample 4	200	500	6.43
Sample 5	200	2000	22.62
Sample 6	450	2000	33.81

The stresses in the films were studied by the profilometry method (KLA-Tencor AlfaStep D-100) with wafer stress chuck and thin film stress options. To do this, surface profiles were obtained before and after deposition of the film along four mutually perpendicular directions by turning the stage by 90° counterclockwise (Fig. 1). Directions of stress measurement from the centre are shown by arrows in different colors: in direction to the base (BD) – black, to the right (RD) – yellow, in the opposite direction from the base (TD) – green, and to the left (LD) – red. The scanning speed was 0.1 mm/sec, the length of the track was 10 mm, the pressure was 1.0 mg, the number of dots in one scan was 10^5. Then the curvature radiuses of these profiles were determined and the average stress of deposited film was calculated according to the formula of Stoney [21]:

$$\sigma = [E_s/(1 - \upsilon_s)](t_s^2/6t_f)(1/R_b - 1/R_a), \quad (1)$$

where E_s is the Young's Modulus; υ_s is the Poisson's ratio; t_s is the substrate thickness; t_f is the film thickness; R_b, R_a are the radiuses of curvature on the substrate before and after film deposition, respectively (for silicon (100), $E/(1–\upsilon_s)$ = 180.5 GPa [1, 22]).

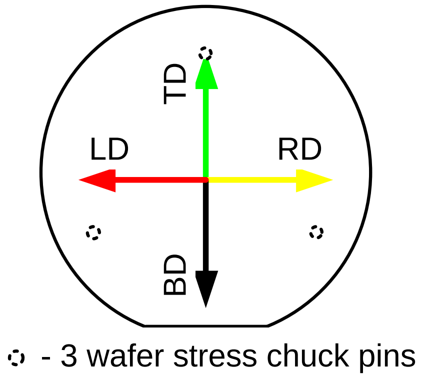

Fig. 1. Profile and stress measurement directions.

The surface profile of the substrates, SiO_x films, and silicon films were determined and the mechanical stresses of SiO_x/*n*-Si(100) and Si/SiO_x/*n*-Si (100) structures were calculated using the described procedure.

3. Results and discussion

A series of six structures Si/SiO_x/Si was manufactured. Typical surface profiles of the substrate and SiO_x films are shown in Fig. 2. Color line denotes direction of stylus motion in accordance with Fig. 1.

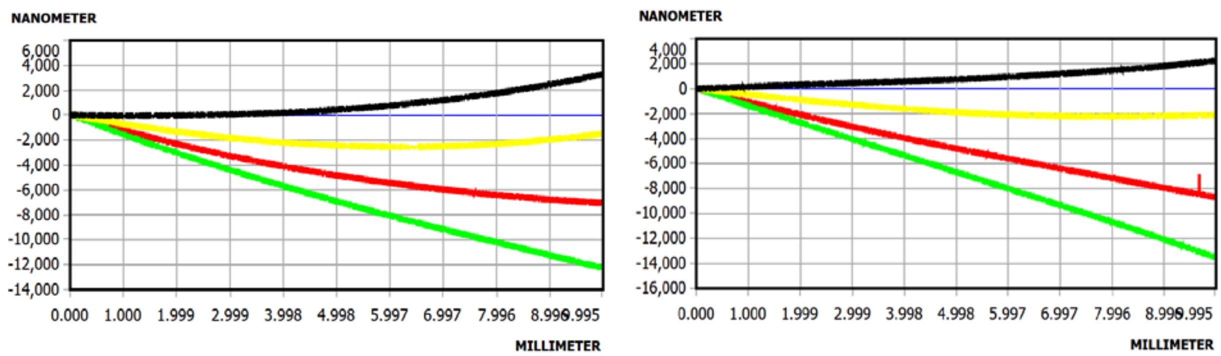

Fig. 2. Profile curves of the substrate (left) and SiO_x/Si(100) structures (right); direction of color line: black– BD, yellow – RD, green – TD, red – LD.

The mechanical stresses in the SiO_x film of 1.145 μm thickness, calculated from profilometry data, were compressive and amounted to –189.9±6.4 MPa and –196.6±10.3 MPa along BT and RL-directions, respectively, or –193.3±8.9 MPa, in average. The results are in a good agreement with [8, 9, 16]. Moreover, the stress values in our films are less.

The profilometry result of Si/SiO_x/Si(100) structures is shown in Fig. 3. The measured values of film thickness and average stresses in silicon films are present in Table 2. The stresses in the films are alternating.

Based on the obtained data, dependences of the average stress in silicon films on deposition temperature at various values of pressure are plotted in Fig. 4.

An analysis of the obtained data shows that the mechanical stresses in films with a thickness from 270 nm to 1.42 μm are in the range of –750 to +250 MPa. The maximum compressive (negative) stresses were found in films, deposited at 450°C; the minimum positive values are at 200°C and 2000 mTorr.

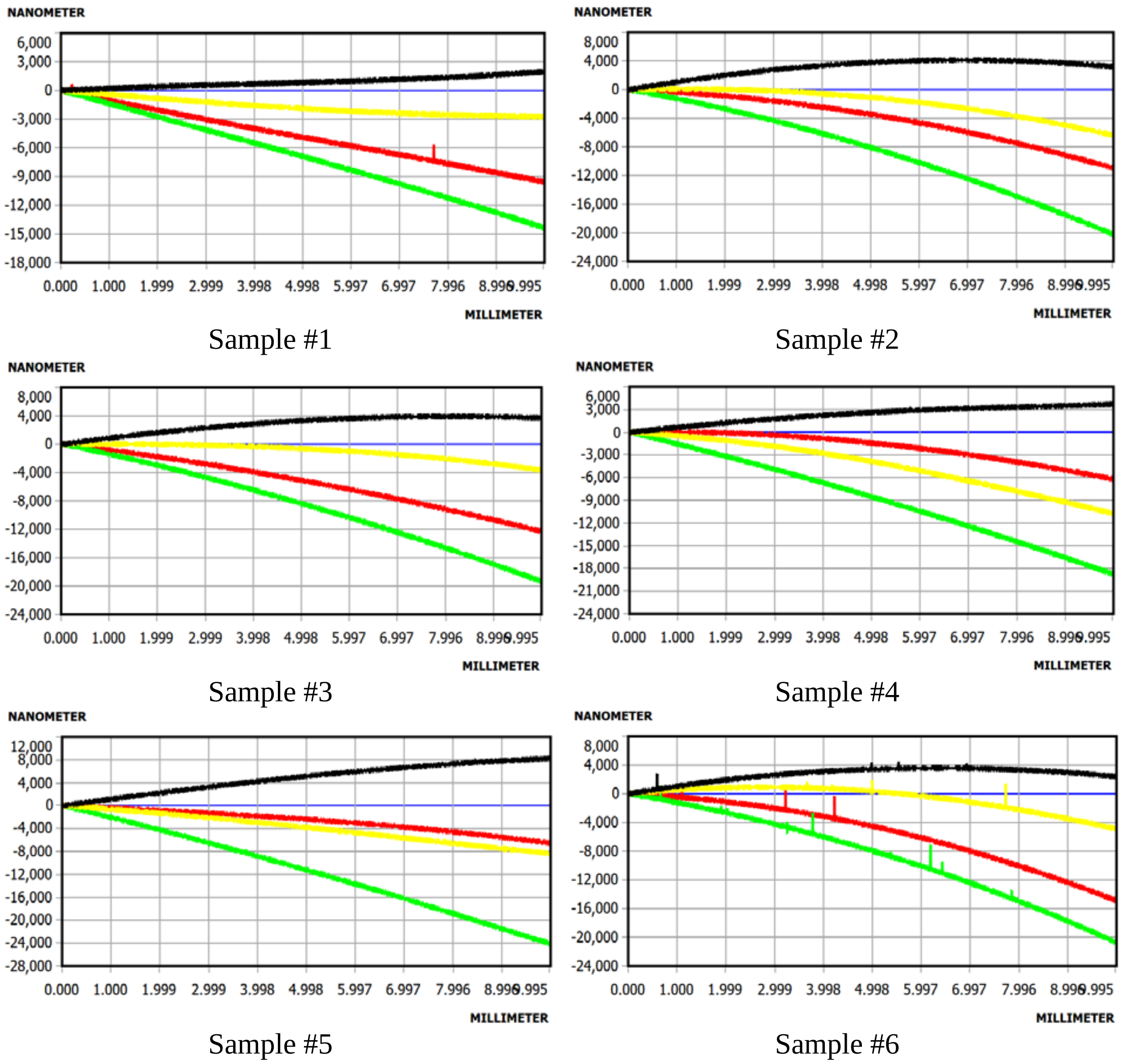

Fig. 3. Profile curves of the samples; direction of color line: black– BD, yellow – RD, green – TD, red – LD.

Table 2.Silicon film parameters.

#	Film thickness, μm	Measured stress, MPa	
		BT direction	RL direction
1	0.322	+156.35	+246.30
2	1.925	–168.15	–168.75
3	0.281	–746.90	–700.80
4	0.270	–399.20	–448.95
5	0.950	+108.95	+65.40
6	1.420	–394.95	–398.55

The films deposited at elevated pressure (2000 mTorr) are characterized by lower stresses and mainly compressive in the investigated temperature range. At temperatures of up to 500 °C, a pressure drop by four times leads to a significant reduction in the deposition rate, the formation of relatively thin and highly stressed films. At temperatures above 500 – 650°C, the behavior changes and the stresses decrease. In this case, for the films, obtained at relatively low pressure (500 mTorr), the stresses shifted to tensile region. The obtained results correlate with [3, 15], partially.

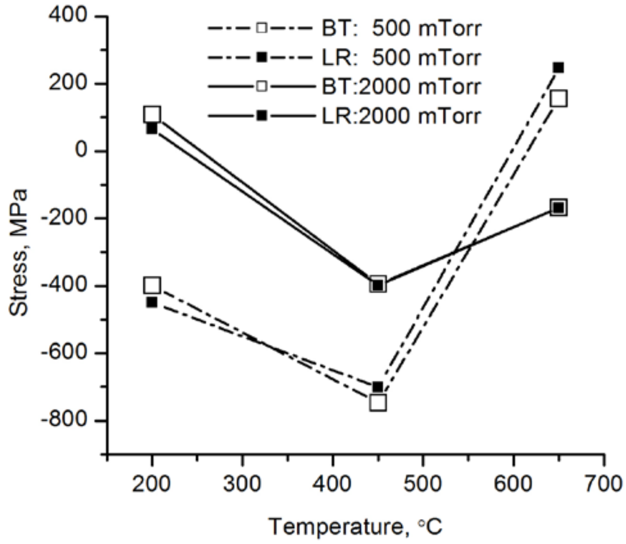

Fig. 4. Mechanical stress in silicon films with different deposition parameters.

A detailed analysis and explanation of the presented results of the dependences will be given together with data on morphology, structure and stress, measured by another method of the deposited films, for which it is proposed to study the films by atomic force microscopy and Raman spectroscopy.

The deposition rate of 22 – 46 nm/min was temperature dependent at high pressure (2000 mTorr) and irrespective of temperature was about 7 nm/min at 500 mTorr.

Low-stressed films, deposited at rates about of 5 – 10 nm/min and temperatures as less as 200°C can be used for solar cells fabrication, based on textured silicon wafer as well as glass or flexible substrates.

Higher rates (more than 40 – 50 nm/min) and deposition temperatures are acceptable for obtaining relatively thick structural layers. It is expected that micromechanical structures formed by releasing the films, i.e. removal of the sacrificial layer (SiO_x), will slightly bend downward. Decrease in pressure from 2000 mTorr to 1000 mTorr presumably will allow forming structures with significant reduced stresses. Furthermore, the good conformity and mechanical characteristics of PECVD films make it essential to MEMS/NEMS applications.

4. Conclusion

The silicon films were obtained from SiH_4 by PECVD technique. The influence of deposition parameters: temperature (200 – 650°C) and total pressure of argon-silane mixture (500 – 2000 mTorr) on mechanical stress in the films has been investigated. Stylus profilometry was performed and average residual stress was determined by measuring the curvature radiuses of silicon substrate, covered by thin films.

The deposition rates were 7 – 46 nm/min, and, particularly, irrespective of temperature at 500 mTorr. It is shown that mechanical stresses in films with a thickness from 270 nm to 1.42 μm are in the range from –750 to +250 MPa.

For mentioned temperature and pressure values, measured stresses in the films preferably were compressive. Residual stress in silicon films was found to increase with temperature up to 400 – 500 °C and decrease with further temperature rises (600°C and more).

The average stresses decrease to a relatively low values with decreasing pressure at high temperatures (650 °C) and with increasing pressure at low temperatures (200 °C); they can be shifted to tensile region (90 – 200 MPa).

According to obtained results, low tensile or compressive stressed films and stress-free films can be formed and used for solar cells and micro- and nanomechanical systems.

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