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ORIGINAL PAPER

Stresses in thin films: an experimental study

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Abstract: Thin film technology is a relatively young and ever-growing field in the physical and chemical sciences, which is confluence of materials science, surface science, applied physics and applied chemistry. Thin film technology has its objectives in the provision for scientific bases for the methods and materials used in thin film electronics (integrated circuits and micro-electro-mechanical system). Additionally, it provides a sufficient data in the area of applications to permit for understanding of those aspects of the subject that might still be termed an "art". Thin films of metals were probably first prepared in asystematic manner by Michael Faraday, using electrochemical methods. Thin films go through several distinct stages during growth, each affecting the resulting film microstructure and internal stress. Hence before proceeding to synthesis and characterization, the knowledge of formation, growth and stress generation in thin film is necessary. This paper explains the influence of process parameters on stress in silicon nitride (Si_3N_4) thin films with experimental results.

Keywords: Thin films; Residual stress; IC; MEMS; Microstructure

PACS Nos.: 68; 81; 81.15.Gh; 85.85. +j

1. Introduction

Residual stress (RS) in thin films and its optimisation play an important role for application in micro-electro-mechanical system (MEMS) devices. It is therefore not surprising that considerable attention around the world has been focused on these particularly as increased finite modelling (FE) power and advanced experimental facilities became available. Influences of residual stresses are well known to MEMS engineers and are used in standard techniques for enhancing the fatigue performance of a wide variety of sensors and actuators components (cantilever, beam, diaphragm, RF switch and resonator etc). The magnitude and distribution of residual stresses in a component or structure is a significant source of uncertainty in MEMS design and one that can affect subsequent micromachining as well as life prediction and assessment of structural integrity. These components or structures use various types of thin films and residual stress in these films are governed by processing parameters. Therefore, before proceeding to manufacture any MEMS device database of process parameters with characterized results of RS in particular thin film should be prepared.

Using plasma-enhanced chemical vapour deposition (PECVD) and low-pressure chemical vapour deposition (LPCVD) methods [1, 2], silicon nitride films of thickness ~ 50 Å to ~ 20 µm were deposited and used for MEMS structures [3–9]. Mostly MEMS structures are free standing, and the presence of RS may bend upward/downward (Fig. 1) [10–12] and even may break them. Therefore, characterization and the reduction in residual stress in nitride thin films are important for improving the reliability of MEMS devices. This film is also used in other various applications such as passivation, diffusion barriers, interlayer dielectric, memory fabrication, gate insulators and capacitor dielectric in MEMS and integrated circuits (IC). This film exhibits stress after deposition, and MEMS structures are sensitive to it. If the stress is too compressive, thin film may blister or buckle, whereas if the stress is too tensile, the film may fracture. It degrades the sensitivity, durability, reliability and overall performance of MEMS sensors/actuators. These consequences of RS drive process engineer to control this during deposition of film.

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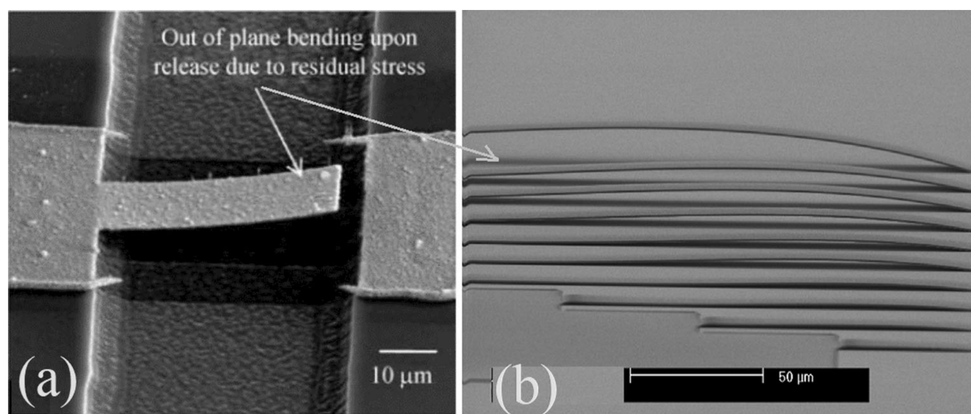


Fig. 1 **a** Out of plane cantilever and **b** bending of cantilevers due to RS [10, 11]

Thus, purpose of this paper is twofold, firstly to study the role of process parameters on residual stresses and secondly, the application of obtained results in MEMS device fabrication. The characterization results are used to optimum value in order to get minimum RS for successful realization of MEMS devices.

1.1. Formation of thin films

The substrate is exposed to the incident vapours in CVD reactor, known as adatoms which arrives on a perfectly flat surface in the very initial stages of film growth. These adatoms will diffuse randomly until they either encounter another adatoms to form a dimer or desorb from the surface. The dimer will dissolve back into two adatoms or absorb another adatoms to either grow further or shrink back due to detachment of atoms. A uniform distribution of small but highly mobile clusters or islands is observed. The next stage involves merging of islands by coalescence phenomenon at high substrate temperatures. Coalescence decreases the island density, resulting in local denuding (uncovering) of the substrate where further nucleation can then occur. Crystallographic facets and orientations are frequently preserved on islands and at interfaces between initially disoriented, coalesced particles. Coalescence continues until a connected network with unfilled channels in between develops. With further deposition, the channels fill in and shrink, leaving isolated voids behind. Finally, even the voids fill in completely, and the film is said to be continuous. This collective set of events occurs (Fig. 2) during the early stages of deposition, typically accounting for the first few hundred angstroms of film thickness.

Thus, growing thin films go through several distinct stages, each affecting the resulting film microstructure and hence its mechanical properties changes.

1.2. Intrinsic stress in thin films

"Intrinsic stress" refers to stress that are not the result of directly applied loads or thermal expansion between the film and its substrate. It is caused by numerous mechanisms such as excess vacancies, crystal dislocations, grain boundary interactions and phase transformations. Formation of voids and incorporation of foreign atoms also contribute to the intrinsic stress [13–19]. Bombardment by ions/atoms during deposition also has a striking effect on many film properties, including stress because the bombardment pinches off loosely bound atoms, resulting in a

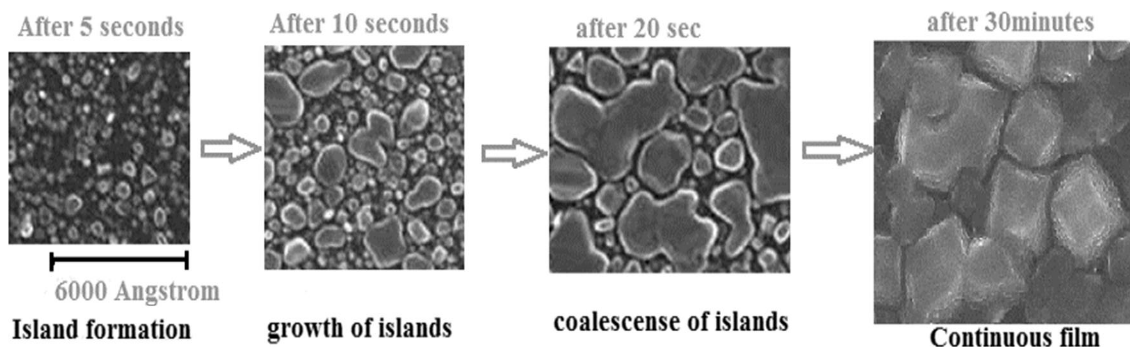


Fig. 2 Thin film formation steps

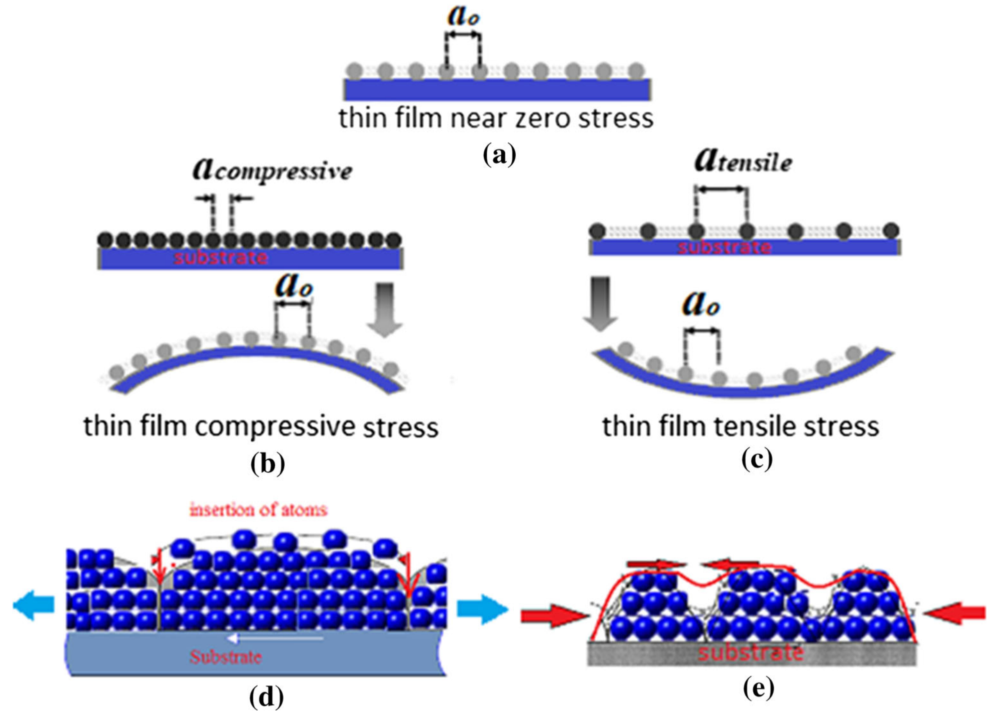


Fig. 3 Pictograph of influence of thin film stress substrate. **a** Thin film has zero residual stress. **b** Compressive stress bend downward after relaxation: rearrangement of constituent atoms in the lowest energy ($a_{compressive} < a_o$). **c** Tensile stress after relaxation of adatoms, bend upward ($a_{tensile} > a_o$). **d** Insertion of adatoms in grains (islands) boundaries causes compressive stress. **e** Tensile stress due to coalescence of islands

more uniform, less stressed film. Too high bombardment, on the other hand, implants atoms into the film in a non-equilibrium way, and compressive stresses (film tend to expand and bend the substrate convexly outward) build up (Fig. 3). Change in volume due to crystallization or phase transitions, and any other processes such as outgassing, lead to stress changes. During deposition or after deposition, polycrystalline films are not in their minimum energy. Low deposition temperature could not impart enough energy to find energetically favourable positions, and the film builds up without relaxation and will be under tensile stress (film tends to contract and bend the substrate concavely upward, Fig. 3).

1.3. Thermal stress

Thin film is deposited normally at high temperature on top of a substrate and cooled down to room temperature. At room temperature, film–film or film–substrate composite expands or contracts according to their thermal coefficient of expansion (TCE) and film is subsequently strained elastically to match the substrate, causing the substrate to bend (Fig. 3). The TCE of silicon is 2.6×10^{-6}/°C (around room temperature). Polysilicon TCE is 2.8×10^{-6}/°C and puts tensile stress on silicon substrate. We can expect compressive extrinsic stresses of silicon dioxide, silicon nitride on silicon wafer which have TCEs 0.5×10^{-6}/°C, 2.4×10^{-6}/°C, respectively, but it was found that stress can be tensile or compressive depending on deposition method and parameters used.

1.4. Total residual stress

Thermal stress along with the intrinsic film stress developed during film growth gives rise to a total RS. Theory for calculation of total stress using free body diagram was proposed by Nix's and others [14, 16], and total RS was calculated from composite curvature of the film and substrate: (here $t = t_s$ thickness of substrate, t_f is film thickness, k is radius of curvature and equal to 1/R, ϑ_s is Poisson's ratio of the substrate, E_s is Young's modulus) by:

$$\sigma_f = \left(\frac{E_s}{(1-\vartheta_s)}\right)\frac{t_s^2}{6t_f}k = \left(\frac{E_s}{(1-\vartheta_s)}\right)\frac{t_s^2}{6t_f R} \qquad (1)$$

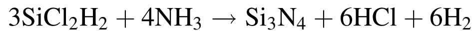
Equation (1) is used to calculate RS of single film. Here thickness of film should be very very less than substrate thickness.

2. Experimental details

2.1. LPCVD process

This deposition process is used to deposit solid film of material on heated substrate through decomposition or chemical reaction of gaseous compounds (precursors). The gaseous precursors were passed over the substrate at low pressure in closed horizontal tube (or furnace). Tempress furnace was used to deposit polysilicon and silicon nitride thin films. It consists of four independent horizontal tubes for deposition.

Silicon nitride films were deposited at two temperatures 800, 850 °C with varying mixture of dichlorosilane (SiH_2Cl_2) and ammonia (NH_3). The mixture of SiH_2Cl_2 and NH_3 was pumped into the furnace, reaction took place on the surface of substrate, and solid film of silicon nitride was formed.

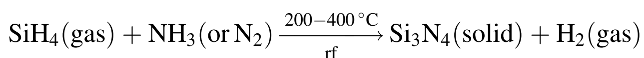


$$3SiCl_2H_2 + 4NH_3 \rightarrow Si_3N_4 + 6HCl + 6H_2$$

2.2. PECVD process

Novellus reactor was used for deposition which operates in dual radio frequency (RF) mode: low-frequency mode (LF = 380 kHz) and high-frequency mode (HF = 13.56 MHz). RF source is utilized to dissociate gases into high energy charged positive and negative ions at low temperature, but mixture is electrically neutral. This mixture of ions known as plasma reacts on substrate surface and forms solid film at low temperature. A low deposition temperature not only prevents damage to substrate material and already deposited thin films but also avoids other undesirable effects that can alter the device performance.

Process parameters used to deposit Si_3N_4 film: (1) total HF–LF power 1000 W (adding LF power 10–90%), (2)-pressure 1–4 Torr, (3) temperature 200–500 °C and (4) NH_3/SiH_4 ratio 1–10. Deposition of Si_3N_4 takes place as per the following reaction:



$$SiH_4(\text{gas}) + NH_3(\text{or } N_2) \xrightarrow[\text{rf}]{200-400\,^{\circ}C} Si_3N_4(\text{solid}) + H_2(\text{gas})$$

Advantage of this process is fast deposition at very low substrate temperatures, good conformality, step coverage's low pinhole counts and excellent adhesion to metal for oxide, nitride layers.

2.3. Measurement of RS

The curvature of each bare Si 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 silicon nitride thin films, the curvature was again measured. The change in curvature was converted into stress by inbuilt software (based on Eq. 1). Refractive indices (RI) were also measured using Opti-Probe tool. RI describes how light propagates through that medium ($n = c/v$, where c is the speed of light in vacuum and v is the phase velocity of light in the medium). RI indicates the composition of the deposited film.

3. Results and discussions

3.1. LPCVD Si_3N_4

Figure 4 shows the relation between SiH_2Cl_2/NH_3 ratio and RS, RI at deposition temperature (DT) 800 °C. With the increase in ratio, RS decreases up to ratio 6 and later on became steady. An increase in ratio can be associated with increase in availability of reactant gases for formation of stoichiometric Si_3N_4 film. Increase in RI confirms this. Initially, high RS and low RI indicate that film has high Si–H contents and so high density. After reaching to optimum value, RS and RI became constant. After ratio 3, no more reactive species are available (decrease in NH_3) for

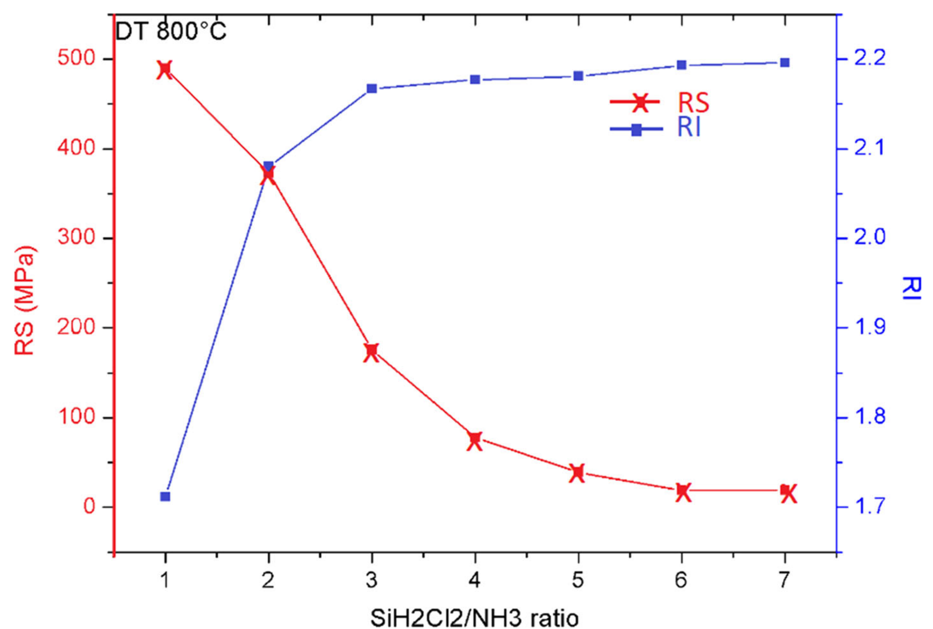


Fig. 4 Variation in RS and RI as function of SiH_2Cl_2/NH_3 ratio at DT 800 °C

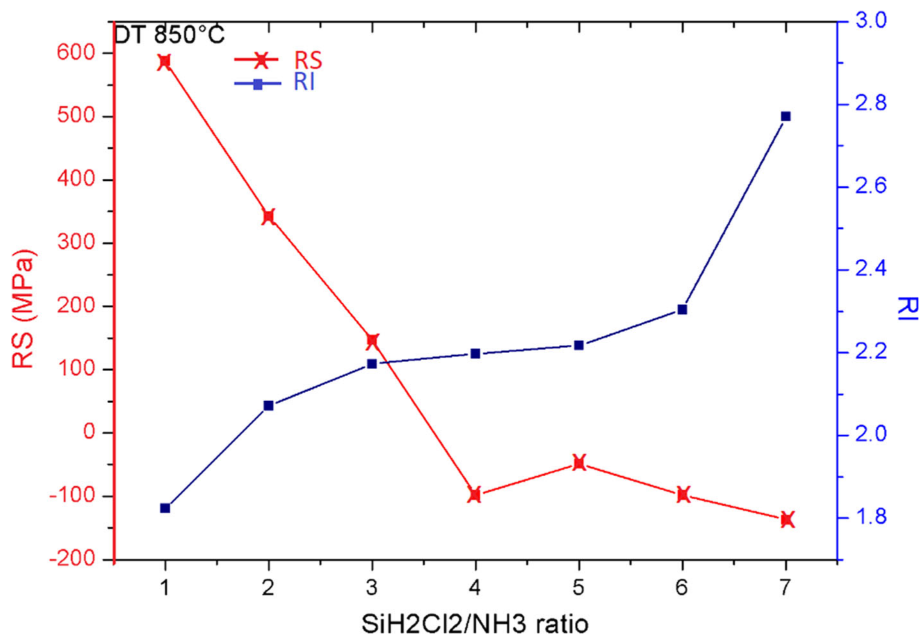


Fig. 5 Variation in RS and RI as a function of SiH_2Cl_2/NH_3 ratio at DT 850 °C

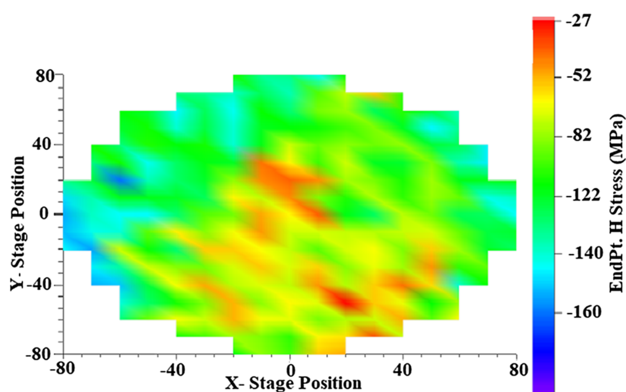


Fig. 6 2D stress map for LPCVD nitride film

diffusion in growing films which can alter the RS except increase in Si-contents and RS reduces. Increase in RI > 2.1 confirms this.

Figure 5 shows the relation between SiH_2Cl_2/NH_3 ratio and RS, RI at DT 850 °C. With the increase in ratio, tensile RS decreases up to ratio 3 and thereafter became compressive but remains almost steady. Reasons discussed above are responsible for decrease in RS and increase in RI. But here temperature is higher and incorporation of more Si-N contents takes place which changes the RS from tensile to compressive. High RI (> 2.2) confirms this.

Figure 6 shows the 2D stress map of silicon nitride film deposited at 850 °C, SiH_2Cl_2/NH_3 ratio 3. It can be seen that RS is not uniform all over the substrate (silicon wafer) and averages are taken for the thin films deposited by two methods.

3.2. PECVD Si_3N_4

Figure 7 shows the variation of RS, RI as a function of LF power for Si_3N_4 films. With a slight increase in LF power to 25%, RS reduces to minimum tensile and RI decreases. With further rise in LF power, RS became compressive and continues to decrease. RI also follows the same trend.

The fascinating behaviour of change in stress from tensile to compressive with the increase in LF% can be explained: Continuously flowing reactant gases are ionized/dissociated by RF power into Si- and N-containing radicals. These radicals react on the surface of substrate and hydrogen contents are released. A low-frequency power results in ions with high energy, while a high-frequency power could lead to a reduction in the ion's flux and energy. Here total power was constant, so increase in LF power yields higher energy electrons. The increased energy of electrons increases the dissociation of the precursor gases and as a result the deposition rate increases. Moreover, the dissociation energy of N_2 is about 9.8 eV, whereas the bond strength H–NH_2 is only about 4.6 eV, and the critical power to activate NH_3 is only about 1/5 of that needed to activate N_2. Therefore, in the high-power range, much more N_2 has been activated and dissociated, which means more N atoms are available to react with Si-radical and film approaches to stoichiometricity. Therefore, N–Si bonding release H-contents and tensile RS approaches to zero after LF power 25%. Further rise in LF power increases the compressive stress due to incorporation of more N (in other words Si/N ratio is less or density is less) contents. Decrease in RI confirms this.

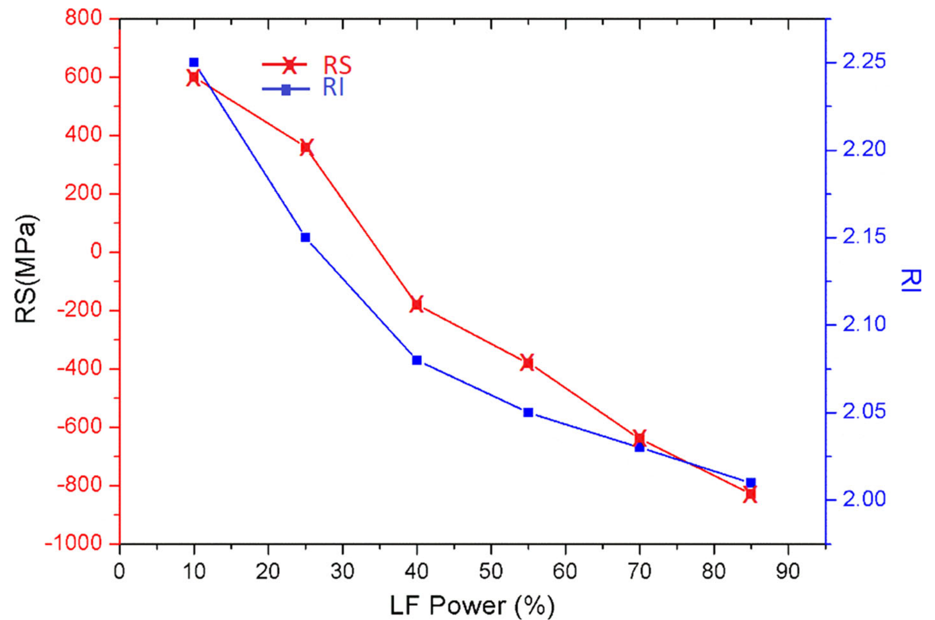


Fig. 7 Variation of RS, RI as a function of LF power for Si_3N_4 films

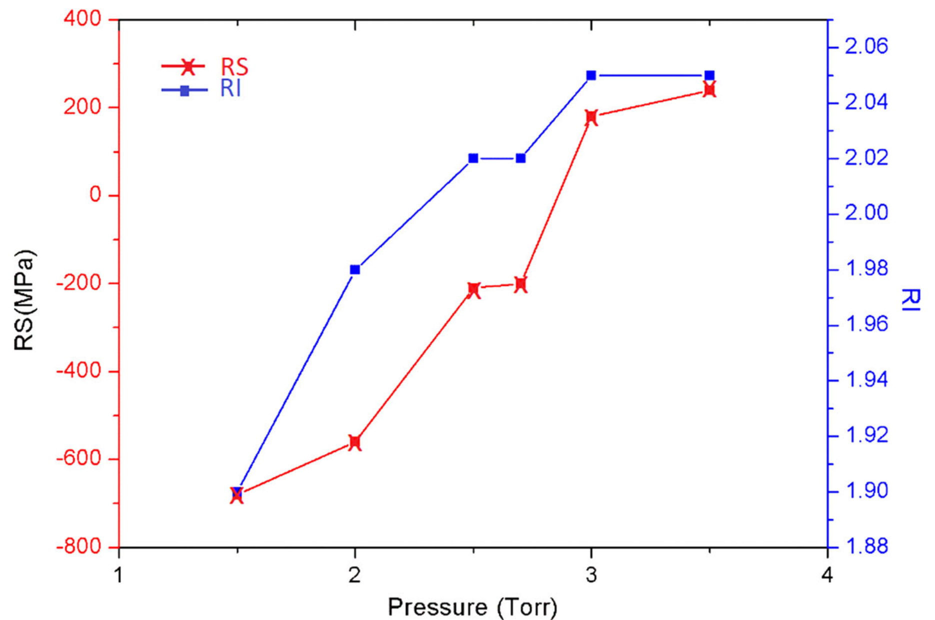


Fig. 8 Variation of RS, RI as a function of deposition pressure for Si_3N_4 films

Figure 8 shows the variation of RS, RI as function of deposition pressure for Si_3N_4 films. At low pressure, reactive species of plasma have enough energy which leads the insertion of species deep into growing films; therefore, at low-pressure RS was highly compressive. Low RI indicates the presence of high N contents, and high RS indicates more Si–N bonds in the film. Pressure helps in plasma stabilization. At high pressure, the mean free path of molecules of reactant gases, charged and energetic species is reduced and thus increases the collisions rate. This results into association and dissociation of the molecules thereby increasing the reaction rate, deposition rate increases and incorporates more Si-contents (Si/N ratio decreases) and RS changes compressive to tensile. Thus, rise in pressure increases in Si-contents and film approaches to stoichiometric (Si/N $\sim$ 0.75 or RI $\sim$ 2.1).

Figure 9 shows the variation of RS, RI as function of DT for Si_3N_4 films. It is proposed that high DT results an increase in dissociation of Si and N radicals and increases the diffusivity into growing film which results in the formation of nearly stoichiometric nitride film between DT 350 and 400 °C. RI between 2.0 and 2.05 confirms this. Increase in tensile stress with rise in DT indicates increase in density.

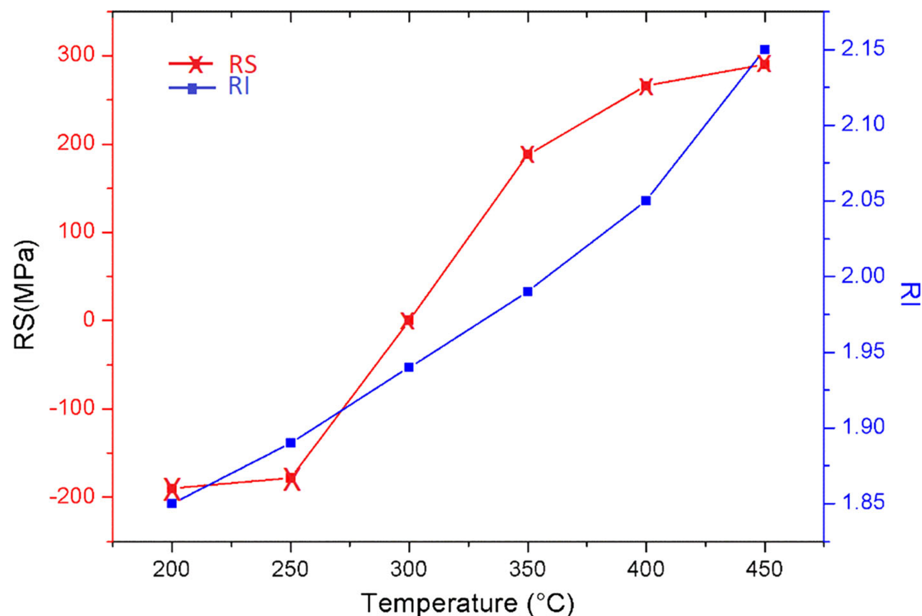


Fig. 9 Variation of RS, RI as a function of DT for Si_3N_4 films

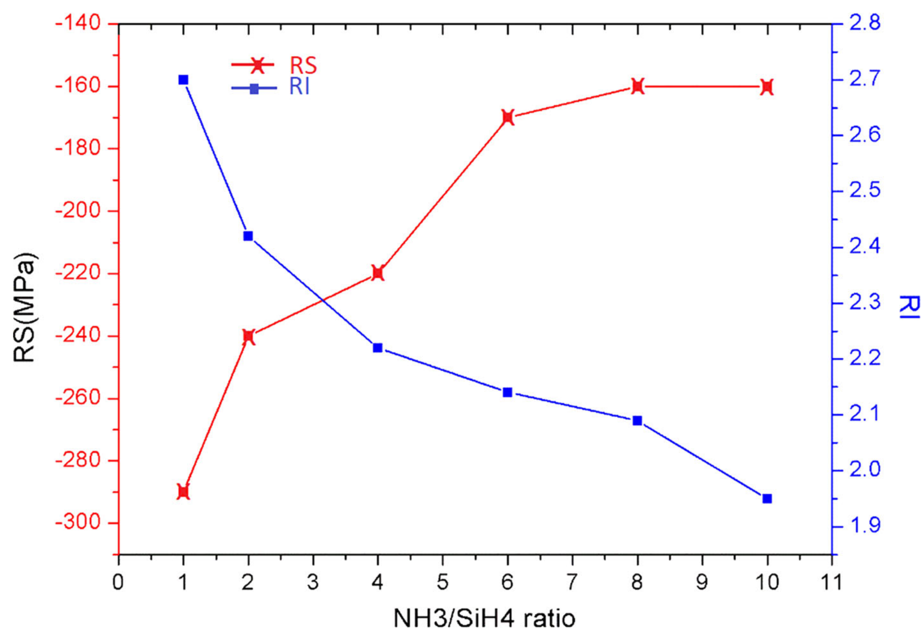


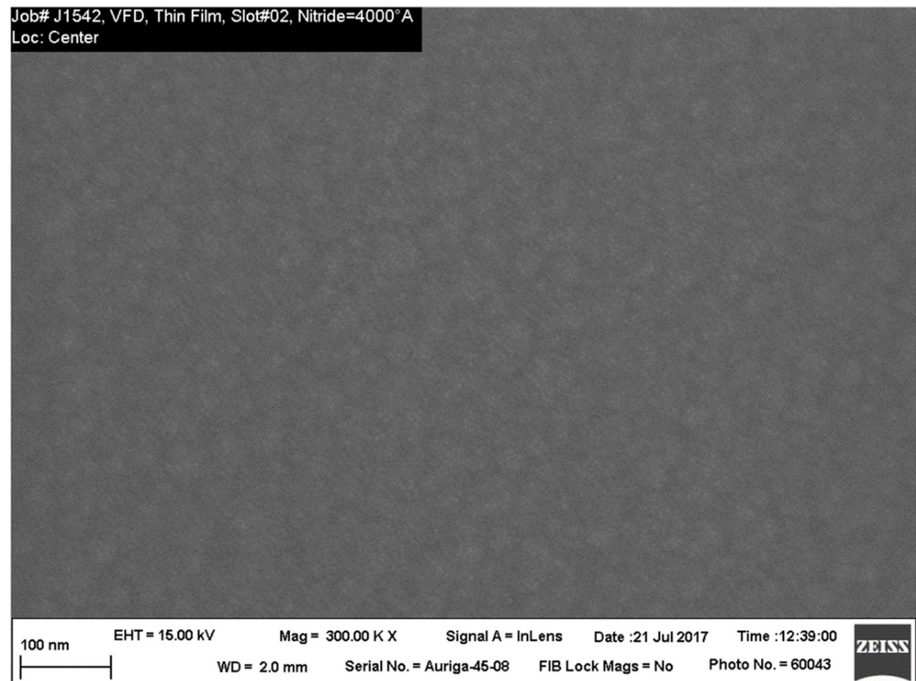
Fig. 10 Variation of RS, RI as a function of NH_3/SiH_4 ratio for Si_3N_4 films

Figure 10 shows the variation of RS, RI as a function of NH_3/SiH_4 ratio for Si_3N_4 films. With the increase in NH_3/SiH_4 ratio, Si-contents increases but Si–H bonds also increase, which results decrease in RI and compressive RS. After ratio 6, RI approaches to 2.1, which is an indication of the stoichiometry of film and RS variation was the least.

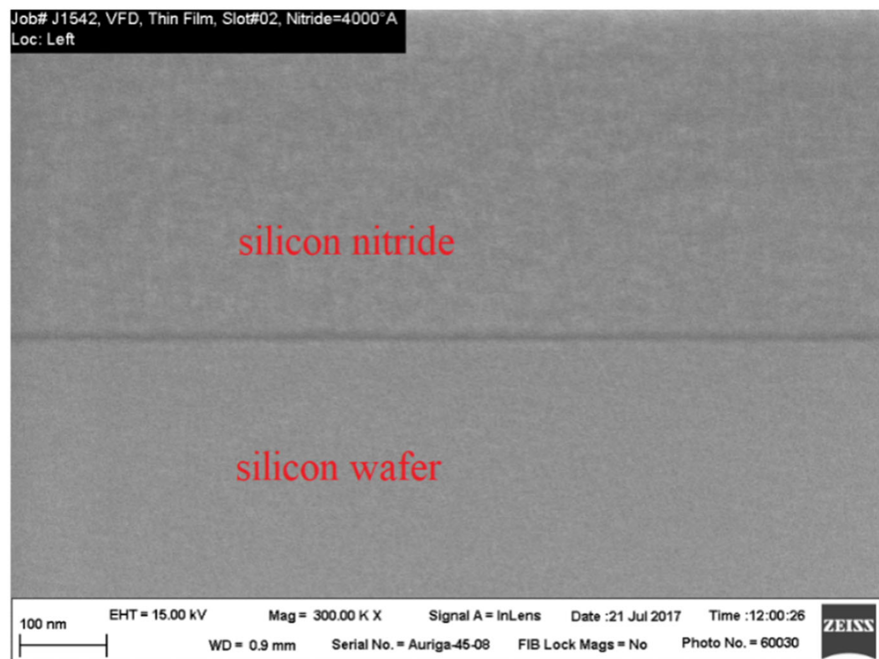
Silicon nitride deposited by LPCVD and PECVD methods is amorphous in structure, Scanning electron microscope image of PECVD nitride is shown in Fig. 11.

3.3. Applications of results

MEMS structures are mostly hanging and bear load. Therefore, films should have minimum RS but at the same time it must have high mechanical strength. Mechanical strength is directly related to density of nitride film. Huang et al. [20] demonstrated that modulus, hardness, film density and Si/N ratio increase with the increase in substrate temperature, plasma power and chamber pressure. $RI > 2.1$ is the indication of higher film density and in other words more silicon contents. Therefore, film with less RS and $RI \sim 2.1$ is always preferred. In the above



(a)



(b)

Fig. 11 **a** Top view of PECVD nitride on silicon wafer. **b** Cross-sectional view of PECVD nitride on silicon wafer

experimental results, if high temperature does not impose restrictions then LPCVD nitride films prepared at 850 °C with SiH_2Cl_2/NH_3 ratio 3.25 are most suitable for MEMS devices. For low-temperature applications, PECVD nitride films were prepared at processing parameters: temperature 400 °C, pressure 2.2 Torr, NH_3/SiH_4 ratio 8 and mixing/adding 50–60% LF power in HF power. Prepared films with these parameters have low RS and RI $\sim$ 2.1. These deposition parameters are preferable for cantilevers, diaphragms or other load-bearing structures. Figure 12 shows the output of two pressure sensors in which PS1 has negligible RS and PS2 has compressive stress − 90 MPa. Full-scale output and linearity drops due to RS.

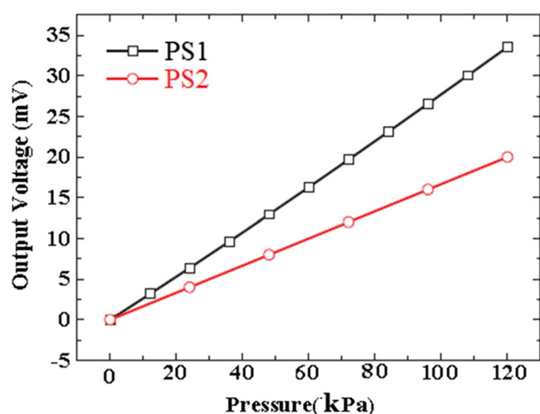


Fig. 12 Influence of residual stress on sensor output voltages

Other important application of nitrides is in passivation field: protection of entire chip (IC or MEMS), interlayer insulating layer, multi-layered wiring from water molecules, movable ions such as Na, pinholes and cracks, corrosion of Al or metal connections. Films with minimum stress but low silicon contents (higher N contents) are required [20–25]. Therefore, if high temperature does not put restrictions then LPCVD nitride can be used. Experimental results suggest process temperature 800 °C, and SiH_2Cl_2/NH_3 ratio 3 will accomplish this. Where high temperature may degrade the quality of existing film on substrate, in such a situation PECVD nitride films are used. Processing parameters pressure 3 Torr, temperature 400 °C, NH_3/SiH_4 ratio 4 and mixing 30% LF power will give nitride film of required properties. These parameters were used to deposit the nitride film on bare silicon wafer.

RI is related to Si/N ratio and RS in both types of processes. LPCVD process have three major drawbacks: It is a high-temperature process, film deposited on both sides of substrate and low deposition rate. Hence, for mass production/batch processing PECVD nitride films are always preferable.

4. Conclusion

A series of experiments were conducted on LPCVD and PECVD silicon nitride films with varying process parameters and elucidated the control mechanism of intrinsic stress. This mechanism can be used for any types of CVD films to develop engineering solutions to prevent failure in MEMS fabrication. As thin film materials have wide applications in MEMS/NEMS, it is expected that the experimental results and theoretical interpretation presented in this paper will contribute to a better understanding and characterization of not only LPCVD/PECVD films, but also to wide range of other thin films for MEMS devices.

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