A COMPREHENSIVE REVIEW ON THIN FILM DEPOSITIONS ON PECVD REACTORS

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Abstract. *The deposition of thin films by Plasma Enhanced Chemical Vapor Deposition (PECVD) method is a critical process in the fabrication of MEMS or semiconductor devices. The current paper presents an comprehensive overview of PECVD process. After a short description of the PECVD reactors main layers and their application such as silicon oxide, TEOS, silicon nitride, silicon oxynitride, silicon carbide, amorphous silicon, diamond like carbon are presented. The influence of the process parameters such as: chamber pressure, substrate temperature, mass flow rate, RF Power and RF Power mode on deposition rate, film thickness uniformity, refractive index uniformity and film stress were analysed. The main challenge of thin films PECVD deposition for Microelectromechanical Systems (MEMS)and semiconductor devices is to optimize the deposition parameters for high deposition rate with low film stress which and if is possible at low deposition temperature.*

Keywords: PECVD, TEOS, silicon oxide, residual stree, thin film deposition
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1. Introduction

Microelectromechanical System (MEMS) technology evolved from silicon process fabrication and is involved in various applications that require miniaturisation, such as mechanical sensors, [1-3] optical microdevices, [4, 5] microactuators [6], chemical synthesis [7, 8] or even biomedical devices [9-12].]. Plasma-enhanced chemical vapour deposition (PECVD) is one of the techniques frequently used in MEMS manufacturing. A relevant number of reports reveal the structural, optical and electronic properties of the classical PECVD thin layers such as amorphous silicon, [13-15] doped amorphous/polysilicon, [16] silicon oxide, [17-19] or TEOS [20, 21], silicon nitride, [22-24], silicon carbide [25-27] or diamond like carbon [28, 29]

Besides their role as a "passivation layer", the thin film PECVD layers were also used as a structural layer for surface micromachined MEMS devices [14],] and as a masking layer for deep wet or dry etching (bulk micromachining) [14, 30, 31], electrodes [32, 33] structural layer in solar cells, [34, 35] or in applications related cell cultures. [23, 36-38]. The main challenge of the PECVD for MEMS and

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NEMS applications is achieving low residual stress, if possible, with a high deposition rate and good uniformity. [39]

The current work reviews the main dielectric layers deposited using the PECVD technique, giving an overview of the influence of process parameters on achieving of main characteristics of the thin layer.

2. Reactors

PECVD is a vacuum-based deposition process operating at pressures typically <1 Torr allowing the deposition of films at relatively low substrate temperatures, usually up to 300°C. The deposition is achieved by introducing reactant gases between a grounded electrode and an Rf-energised electrode. The capacitive coupling between the electrodes excites the reactant gases into plasma, which induces a chemical reaction and results in the reaction product being deposited on the substrate. An example is the SPTS Multiplex Pro-CVD PECVD system. the system can generate plasma in two RF modes: low frequency (LF) at 380 kHz and high frequency (HF) at 13.56 MHz. The RF power for both these modes can be tuned within a broad range: between 0 to 1 kW for the LF mode and 0 to 600 W for the HF mode. Some reactors are also equipped with a liquid delivery system (LDS). Other versions of reactors make use of an Inductively Coupled Plasma (ICP) source. The creation of high-density plasmas in the ICP source means this technique delivers the deposition of high-quality dielectric films at low temperatures with low damage. Low-temperature deposition means that temperature-sensitive films and devices can be processed successfully. Some of the advantages of ICP PECVD rely on independent control of ion energy and ion current density, typical process pressure: 1- 10 mtorr, plasma density: $>10^{11}$ cm^{-3}; plasma in contact with the substrate, low energy ion current during deposition' Ion Current (Plasma Density) dependent on ICP power, ESS (electrostatic screen) for a purely inductive plasma.

3. Thin layers deposition on PECVD reactors

3.1 Silicon oxide.

The SiO_2 thin films are hardly used to fabricate Si, III-V, MEMS and GaN semiconductor devices. One of the applications that drove the development of the PECVD SiO_2 process was optical waveguides, were insulating the dielectric optical medium from the substrate is necessary due to its light absorption characteristics.[40] In this application, the main advantage of the PECVD technique relies on effectively controlling the optical properties (refractive index) by tuning the deposition parameters. The SiO_2 films were prepared on PECVD reactors using SiH_4; N_2O chemistry with N_2 or Ar as a dilution gas. The residual

stress is the main element that limits the thickness of the deposition, required in optical waveguide applications, a method to increase the deposition thickness. At the same time, maintaining the quality of deposition (low surface roughness and uniform stoichiometry along with the silicon oxide layers) is the deposition of a multilayer process, each deposition being followed by an RTA (Rapid Thermal Annealing) process at a temperature of 900 DC. Using this method, up to SiO_2 layers up to 4 μm were deposited in [40]. The phenomenon of stress release can be attributed to an increase of the Si-O-Si bond angle at the bridging oxygen atom site as a consequence of the reordering of the oxide network during the annealing in this almost stoichiometric oxide film.[41] Thick SiO_2 layers deposited using careful control of the process parameters were frequently used as masking layers for deep RIE processes. [42-44]

3.2 TEOS.

For the manufacturing of infrared detectors, the processing of thin silicon wafers using temporary polymeric bonding on dummy Si wafers carrier or deposition of passivation layers (silicon oxide or silicon nitride) InSb and HgCdTe require deposition temperature of 200°C in order to avoid the substrate damaging. Optical/display applications, which may use polymeric and glass substrates, also require lower deposition temperatures. Research into wafer bonding techniques, especially in wafer-level packaging, for creating chip-scale packages explores the use of adhesive bonding. These adhesives that allow wafer bonding to take place at low temperatures of 100-200°C also demand the deposition of thin films (such as SiO_2) at temperatures as low as 200°C. From this point of view, the deposition of classical SiO_2 using PECVD reactors is not a practical solution. The residual stress in such layers is compressive (with values around 200MPa), while the insulating properties are pretty poor. From this point of view, the deposition of the TEOS layer can be an alternative. Manhajan *et al.* [45, 46] and Raupp *et al.* [47] reported TEOS thin film deposition rates up to 80nm/min. Another critical aspect of TEOS deposition in PECVD reactors is related to the exfoliation of the layer, mainly due to the poor adhesion of the thin film on the substrate and residual stress (Figure 1). [21] Stress tuning in the SiO_2 passivation layer correlated with a reasonable deposition rate, good uniformity, and good quality of the deposited layer is the primary desire for most of the MEMS mentioned above.[20] Low-stress TEOS deposition and low temperature can be achieved by controlling the deposition process. In [20], Carp et al reported thin films TEOS deposited on PECVD reactors with a deposition rate of 210nm/min (nonuniformity 1.3%), residual stress -9.5Mpa and refractive index 1.456.

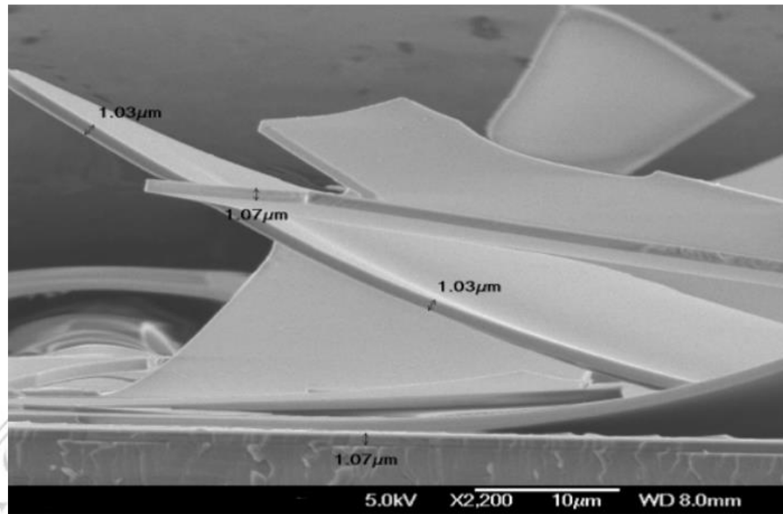
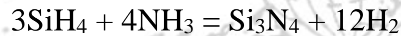


Figure 1. Peel-off TEOS thin film due to the poor adhesion and residual stress. [21]

3.3 Silicon nitride

ECVD silicon nitride is a heavily used material in MEMS applications and has been well studied. Deposition of low-stress silicon nitride layers was required for free-standing MEMS structures such as beams and cantilevers.[22] It can also be used as a masking layer in bulk processing silicon. [48] or even in cell culture applications [23, 49, 50]. With PECVD, silicon nitride can be formed by the reaction:

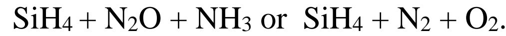


$$3SiH_4 + 4NH_3 = Si_3N_4 + 12H_2$$

Pure silane gives the advantage of a higher deposition rate, but for safety reasons, silane is diluted with Ar or He (especially in research labs). In all cases, hydrogen is built into the deposited layer. By varying the process parameters, deviations from stoichiometry are easily caused. For PECVD nitrides, the hydrogen content can reach 40 at.%. [51] The H content drastically depends on deposition temperature, gas composition, and stoichiometry. It was reported that for the SiH_4–NH_3 reaction, the total H content increases drastically for N–Si ratios above 0.7 and that the H atom is mainly bonded to N in the composition range above 0.7.[52] This is not the case when N2 replaces NH3. The result is then a substantially reduced total H content.

3.4 Silicon oxynitride.

Silicon oxynitride ($SiO_xN_yH_z$) is a material that can be prepared with properties ranging from those of nitride and oxide. It can be formed using the following gases:



$$SiH_4 + N_2O + NH_3 \text{ or } SiH_4 + N_2 + O_2.$$

The properties of $SiO_xN_yH_z$ such as residual stress, etching selectivity to Si or refractive index, can be fine-tuned by adjusting the process parameters. This leads to applications where both SiO_2 films and Si_3N_4 films are inferior. An example is the use of $SiO_xN_yH_z$ as passivation layers in ISFETs. With a variation of the gas flow ratio of N_2O/NH_3 in a 300°C PECVD process, it is possible to control the concentration of the reactive species such as SiNH, $SiNH_2$, and SiOH and, therefore, to optimise the grafting of this layer by silane components.[53] The diffusion barrier is better than that of SiO_2 and sensors using $SiO_xN_yH_z$ exhibit good long-term stability. The possibility of adjusting the refractive index of SiOxNyHz between 1.46 and 2.0 by varying the ratio of O_2/N_2O in the gas mixture in the PECVD process makes this material suitable for optical sensor applications such as couplers, y-junctions, interferometers or dielectric mirrors [54]

3.5 Silicon carbide.

Silicon carbide is a frequently used material for micromechanical applications, especially for high-temperature sensors or MEMS applications. [55] It is highly transparent, can be p- or n-doped in the crystalline phase has a high breakdown field and is piezoresistive. Its high mechanical strength, low friction coefficient and thermal conductivity, and its extreme chemical inertness— while still being RIE etchable—makes it a primary micromechanical material. It can be deposited in the amorphous phase from:

$$SiH_4 + CH_4 + H_2$$

gas mixtures [55]] at temperatures ranging from 200 to 400˚. C6H18Si2 (well-known as HMDS), as a liquid precursor, can be used, which is non-toxic and non-hazardous. [56] The films can be deposited stress-free by proper choice of deposition parameters. The index of refraction varied between 2.2 and 2.9, and the observed step coverage over aluminium layers (top to flank thickness) ranged between 1 and 1.6, which are good values. The high etch resistance against concentrated HF makes it suitable to be used as a sacrificial layer or in deep wet etching of glasses.[26] An example of SiC as a functional layer combined with amorphous silicon as a sacrificial layer is the fabrication of a MEMS free-standing microstructure.[14] Another example is the free-standing mechanical structures fabricated from amorphous SiC through bulk processing of Si substrate in alkaline solution (KOH). Cantilevers fabricated from low-stress amorphous SiC (PECVD) are presented in Figure 2. Components for micromotors from amorphous SiC have already been built and reported. [57]

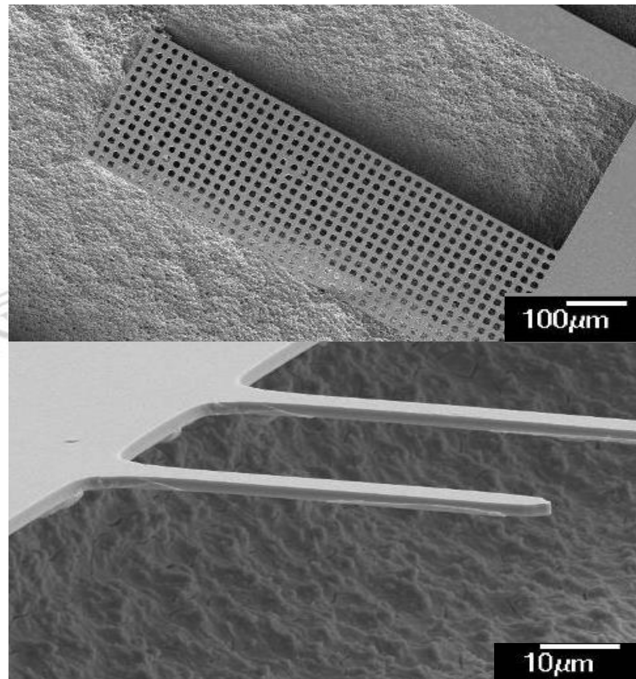


Figure 2: SEM images with cantilevers fabricated from amorphous SiC through wet etching process in KOH.

3.6 Diamond-like carbon films.

Many properties of diamond make it an attractive material for various micromechanical device applications. Diamond is a semiconductor with an even larger bandgap (5.48 eV) that presents high transparency, a high breakdown field, and high thermal conductivity; it can be p-doped and is piezoresistive. In add-on to its extreme mechanical strength and hardness, it has a small coefficient of dry friction (0.05–0.15), comparable to the value of Teflon. [58] T This and its high wear resistance makes a diamond a superior material when used in dynamic microdevices (micromotors). Diamond is inert against aggressive environmental conditions (it oxidises above 500 ◦C) and resistant to the usually concentrated acids. PECVD was successfully employed to deposit amorphous diamond-like carbon (DLC) films using different hydro-carbon process gases such as CH_4 and H_2. CH4 provides the carbon, while H_2 suppresses the formation of graphite. [59] With microwave plasma excitation, it was possible to grow poly-diamond films [59] or even heteroepitaxial [60] on silicon or silicon carbide single-crystal substrates. DLC films can be patterned through an sRF sputter-etching process in an Ar (etch rate ~ 100 A/min).

4. INFLUENCE OF THE DEPOSION PARAMETERS

4.1 Influence of chamber pressure

Pressure is a critical parameter which will significantly influence the characteristics of the thin films deposited in the PECVD reactors. It is usually varied between 500-1100 mTorr. Increasing the pressure chamber will increase the deposition and deposition rate uniformity. The "doughnut" shape of the uniformity's map observed for the low-pressure values (with smaller values in the centre of the wafer and larger values near the edges) suggests that there is not enough time for reaction and deposition of the molecules and radicals on the substrate surface with the gas being sucked to fast by the vacuum pump. Increased pressure in the reactor improves the dissociation rate. However, over the critical value of the pressure, the mean free path distance between the molecules of reactant gases, charged and energetic species, is reduced, and the number of effective collisions increases significantly, leading to other associations and dissociation. As a result, in the first phase, the number of increased molecular associations aids the deposition and, hence, the deposition rate increases. In the second phase, however, if the pressure continues to increase, although the number of collisions continues to increase, the most significant majority will be less and less related to reaction(s) with the substrate as the deposition rate decreases after reaching a local maximum. For SiNx layers, the uniformity of the layer at 700 mTorr is typically up to 10%, more significant than those of 900 and 1100 mTorr, which are 2% and 1%, respectively. [23] TEOS deposition pressure greater than 700mTorr assures a good uniformity of the thin layer and the refractive index (below 2%), but above 950mTorr, exfoliation of the film can be noticed. (Figure 3).

For SiNx layers, the pressure of the chamber significantly affects the SiN_x residual stress. The decreasing pressure results in an increase in electron energy, which subsequently leads to an increase in the N to SiH_3 radical ratio, namely the decrease in the Si/N ratio. This interpretation is consistent with the enhanced concentration of N-H bonds resulting from a pressure decrease.

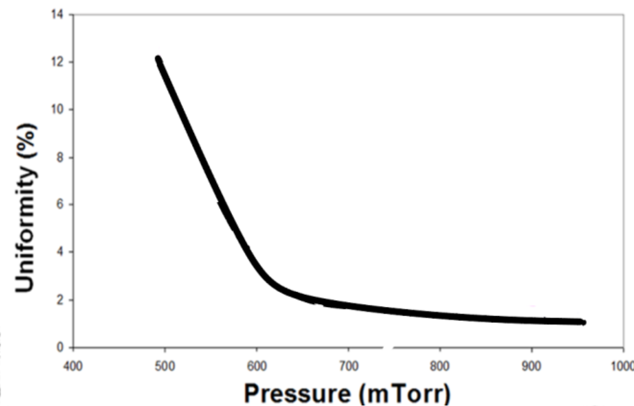


Figure 3: Variation of the thickness uniformity of TEOS-PECVD deposition with the pressure in the reactor chamber

4.2 Influence of the temperature

Temperature is another parameter that can affect the quality of the deposition. In most cases, it must be associated with the processing RF mode (High frequency-13.56 MH or Low-frequency 100-400kHz). Generally, the processing temperature has a low influence on the deposition rate but can strongly influence the residual stress. [26]. As presented in Figure 4, with the increasing temperature, the stress becomes tensile. The temperature seemed not to affect the refractive index. [20, 26] However, the deposition rate decreases with increasing temperature.

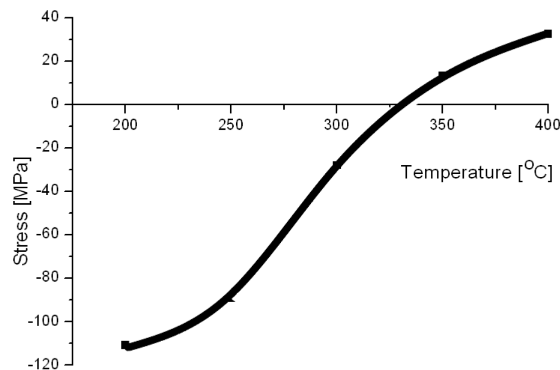


Figure 4. Variation of the residual stress with the temperature for PECVD SiC depositions

The residual stress in the silicon oxide is found to be tensile at lower temperatures and more compressive at higher temperatures (Figure 3b). This may partly be due

to the difference in the coefficient of thermal expansion (CTE) between silicon dioxide (0.5ppm/°C) and the silicon wafer (3ppm/°C).

4.3 Influence of power in high frequency and low frequency mode

The increased power of the RF signal can be associated with a high rate of gas dissociation, which leads to more reactive species in the plasma with a direct effect on increasing the deposition rate. The main difference between high frequency (HF) and low frequency (LF) modes relies on the ion bombardment that characterises plasma in LF mode. The ions cannot follow the frequency at HF (more than 1MHz) due to their inertial mass. For this reason, deposition at LF is associated with a bombardment of the thin layer, affecting densification and stress tunning. From Figure 5, it can be observed that for the amorphous Si layers, the deposition rate and stress (compressive) values increase with the power of the HF mode.

Meanwhile, for the other frequency mode, Figure 6 shows a linear proportionality of the deposition rate with the LF power. The refractive index dips slightly at higher HF powers, indicating that the higher bond breakage rate may result in a less dense film. The applied power can also influence uniformity.

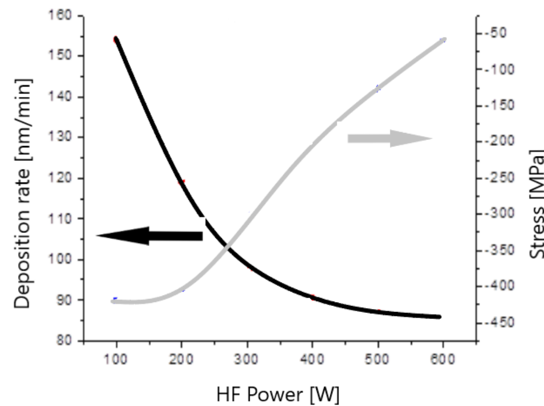


Figure 5. Deposition rate and stress variation for PECVD amorphous silicon in HF mode

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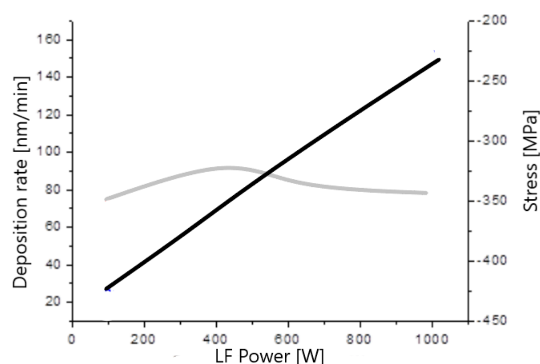

Figure 6. Deposition rate and stress variation for PECVD amorphous silicon in HF mode

4.4 Influence of gas composition

In order to achieve the desired thin films, a different combination of gases is used. Ar and He (sometimes even N_2) are frequently used for dilution. The flow rate ratio between these gases plays an essential role in defining the physical and chemical properties of the fabricated thin layers. A simple example is the deposition of SiC thin films using SiH_4/CH_4 chemistry. The results presented in [26] reveal a linear stress-dependent dependence on SiH_4/CH_4 ratio. If the content of SiH_4 increases, the deposited α-SiC film becomes more “Si-rich", leading to a decrease in compressive stress value and an increase in refraction index.

5. CONCLUSIONS

PECVD is a versatile tool for fabricating thin films with applications on MEMS or semiconductor devices. It allows stress-tuning by selecting the correct process without the necessity of using a collateral process (such as annealing or implantation). The presence of LF mode power brings more flexibility to the PECVD process through better control of the density of the film (due to the ion bombardment) and, from here, the opportunity to fine-tune the residual stress and refractive index.

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