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# CHARACTERIZATION OF TEOS THIN FILM DEPOSITIONS ON PECVD REACTORS

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**Abstract.** *Abstract. The deposition of silicon dioxide layers on Silicon substrate using tetraethyl-orthosilicate (TEOS) by Plasma Enhanced Chemical Vapor Deposition (PECVD) method was characterized in this work. Deposition rate, film thickness uniformity, refractive index uniformity and film stress were analyzed in relation to variation of process parameters such as: chamber pressure, substrate temperature, RF Power and mass flow rate (of oxygen and TEOS) had been investigated. The challenge is to optimize the film deposition for (a) a high deposition rate with low film stress which is significant for Microelectromechanical Systems (MEMS) and (b) a high deposition rate at a low temperature (200°C) which is relevant aspect for microelectronics packaging applications.*

**Keywords:** Keywords: PECVD, TEOS, silicon oxide, residual stress, thin film deposition

## 1. Introduction

Deposition methods at lower temperatures are desired for most of MEMS applications. [1] Plasma enhanced chemical vapor deposition (PECVD) can be a solution in this direction.[2] A number of studies investigated the structural, optical and electronic properties of the PECVD thin layer such as amorphous silicon, [3-5] silicon oxide, [6, 7] silicon nitride,[8-10] or silicon carbide.[11-13] In other applications, the PECVD this films were used as masking layers for the bulk wet and dry etching.[1,14-16]. Moreover BioMEMS applications of PECVD thin film has been reported. [18-21] A challenge for MEMS and NEMS applications is achieving a low residual stress in thin layer correlated, if possible with a high deposition rate and good uniformity. [22]

For the above mentioned applications, a critical temperature is 200°C. This value is required in processing of thin silicon wafers (using polymeric temporary bonding on dummy Si wafers carrier). The deposition of passivation layers ($SiO_2$ or $Si_3N_4$), for the manufacturing of infrared detectors based on InSb and HgCdTe, needs processing temperatures below 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 the area of wafer level packaging, for creating chip scaled packages are exploring the use of adhesives bonding.

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Use of these adhesives that allow wafer bonding to take place at low temperatures of 100÷200 °C also demand that the deposition of thin films (such as $SiO_2$) at temperatures as low as 200 °C. For 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 200 MPa), while the insulating properties are quite poor. For this point of view the deposition of TEOS layer can be an alternative.

Previous works of Manhajan *et al.* [23, 24] and Raupp *et al.* [25] reported deposition rates of the TEOS thin film  up to 80 nm/min. Another important 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). Stress tuning in $SiO_2$ passivation layer correlated with a reasonable deposition rate, good uniformity as well as a good quality of the deposited layer is the main desired for most of the above-mentioned MEMS applications.

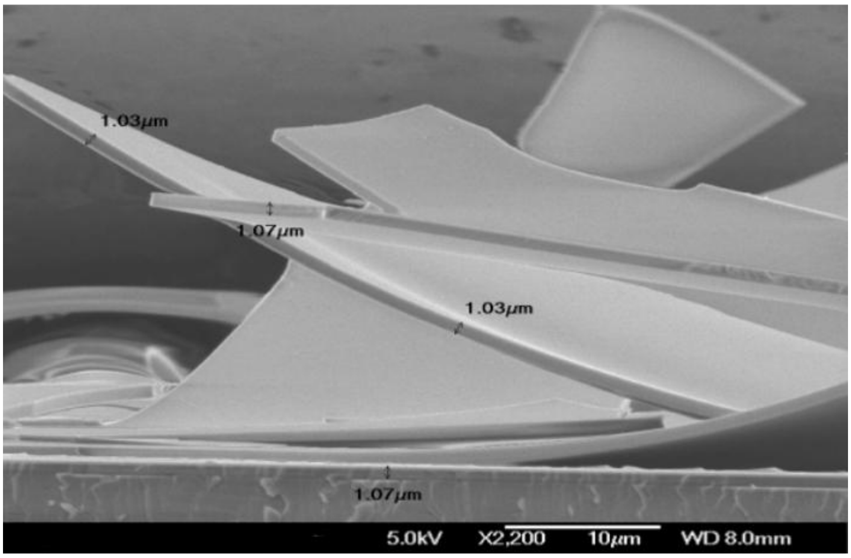



**Fig. 1.** SEP image showing the main defect that characterizes the TEOS deposition: peeled-off TEOS thin film due mainly to the adhesion and residual stress.

Here we report deposition o low stress TEOS layers on PECVD reactors at low temperature (200 °C) having high deposition rate (over 200 nm/min). The step coverage of the TEOS layer presents also good step coverage of 0.7 (thickness of the layer on the wall of vertical trench/thickness on the top or bottom surface). The process parameters were analyzed an optimized. We concluded that the main causes of these defects are related to extreme high pressure, low temperature or high TEOS/$O_2$ ratio.

## 2. Materials and methods

TEOS layers can be deposited at low temperatures in Plasma Enhanced Chemical Vapor Deposition (PECVD) reactors. In such reactors plasma is generally created between two electrodes is generally created by radio frequency (or DC discharge) in the presence of reacting gas, the substrate being connected at one of this electrodes.

The deposition of TEOS layers was performed using a STS Multiplex Pro-CVD PECVD system. A schematic diagram of this equipment can be found elsewhere and a detailed description is presented in , where it is also reported that low stress SiNx layers were deposited with a high deposition rate in similar reactor. This system can generate plasma in two RF modes: low frequency (LF) at 380 kHz and/or 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. The depositions of our TEOS layers were always performed using a liquid delivery system (LDS) incorporated to the system and $O_2$. The specific values of deposition parameters will be given later when the various experiments are presented in detail in the following sub-sections.

The average stress characterization of the TEOS films was performed with a stress measurement system (KLA Tencor FLX-2320, USA). The thickness and thickness uniformity as well as the refractive index and its uniformity across the wafer for the deposited TEOS films were measured with a refractometer (Filmetrics F50, USA).

For the experiments 4”, p type, 500 m-thick silicon wafers with a (100) crystallographic orientation were used. The wafers were initially cleaned in piranha ($H_2SO_4$:$H_2O_2$ in the ratio of 2:1) at 120$^{O}$C for 20 minutes and rinsed in DI water. The native silicon oxide layer was then removed by immersing the wafers in a classical BOE solution for 30 seconds. In the PECVD chamber, the silicon wafers are then deposited with silicon dioxide for 5 minutes based on the recipes bearing variations of process conditions to investigate the effects of these conditions on the silicon dioxide deposition. The process conditions varied and their working range are:

a) Chamber pressure (500 mTorr÷1400 mTorr)

b) RF power at high frequency (HF); 13.56 MHz (0 W÷500 W)

c) RF power at low frequency (LW); 380 kHz (LF) (0W÷500W)

d) TEOS flow rate (0.2v 1 sccm/min)

e) Oxygen flow rate (900÷2000 sccm/min)

f) Substrate temperature (200 °C – 350 °C)

## 3. Influence of the deposion parameters

### 3.1. Influence of chamber pressure

Pressure was varied between 500÷950 mTorr. It was found that the refractive index remained fairly constant, showing that pressure had slight effect, but the deposition rate improved with increasing pressure.

Both uniformity of refractive index and film thickness decreases quickly at the lower pressures. It can be concluded that in order to achieve a uniformity of the refractive index and thickness below 2%, the optimal range for pressure must be between 700 mTorr and 950mTorr – (Figure 2a).

At higher pressures of above 950 mTorr the film was “damaged” showing exfoliations as the illustrated in Figure 1.

The variation of the residual stress with the chamber pressure was not very relevant showing slow increases from -15 MPa to +15 MPa for a variation of the pressure between 500 MPa and 950 MPa.

The deposition rate is strongly influenced by the pressure in the reactor. An increased pressure in the reactor improves the dissociation rate.

Figure 2b presents the variation of the deposition rate with the pressure showing an almost linear variation.

The value achieved for the deposition rate (from 100 nm/min up to 250nm/min) being suitable for most of industrial applications.

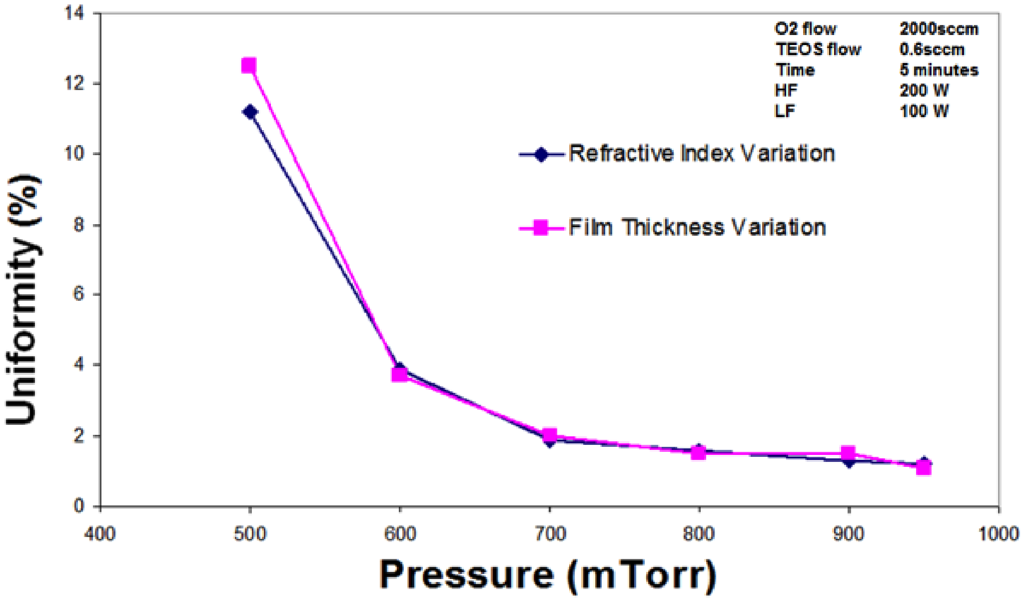
### 3.2. Influence of chamber temperature

Temperature seemed to have no effects on the refractive index. However, the deposition rate decreases with increasing temperature. Vianna et al attributed this to the possible incorporation of non-dissociated TEOS sub-products at lower temperatures although the constant refractive index at the different temperatures is not indicating this aspect.

The experiment performed with TEOS flow rate at 0.6 sccm and temperature at 200 °C, the film was damaged. TEOS flow rate was reduced to 0.3 sccm and the deposition was successful. Deposition rate decreases with increasing temperature (Figure 3a). The uniformity worsened slightly with increasing temperature although it may not be too significant with variations of less than 2% (1.5÷1.8%).

The residual stress in the silicon oxide is found to be tensile at lower temperatures and more compressive at the higher temperatures (Figure 3b). This may partly be due to the difference in the coefficient of thermal expansion **(**CTE) between silicon dioxide (0.5 ppm/°C) and the silicon wafer (3 ppm/°C).

**a)**




**b)**

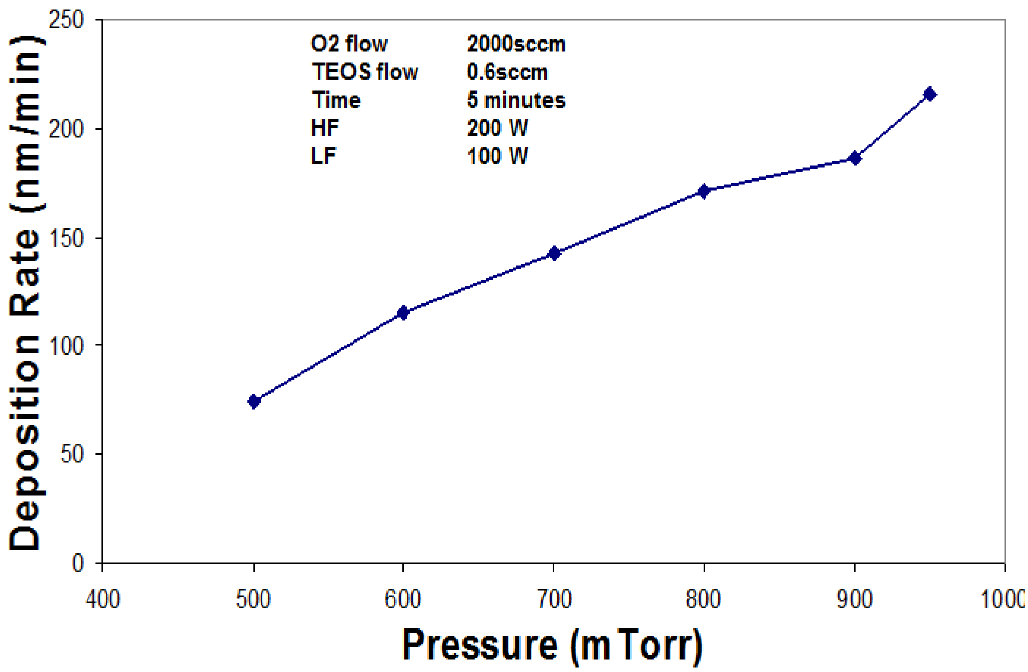
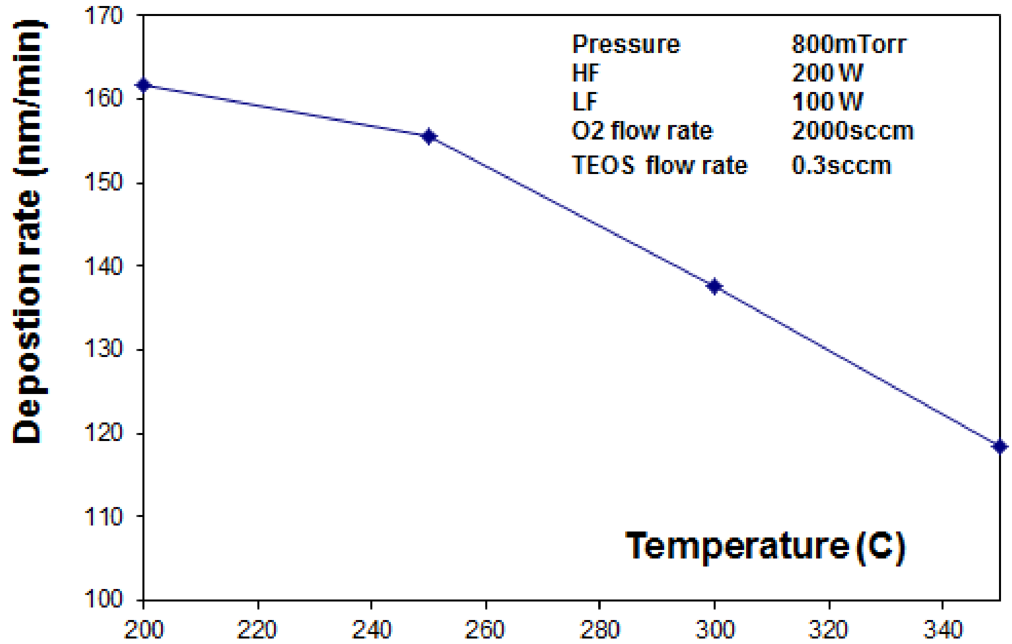



**Fig. 2.** Variation of the uniformity of the refractive index and film thickness with the pressure in the PECVD reactor (a); variation of the deposition rate with the pressure (b).

**a)**

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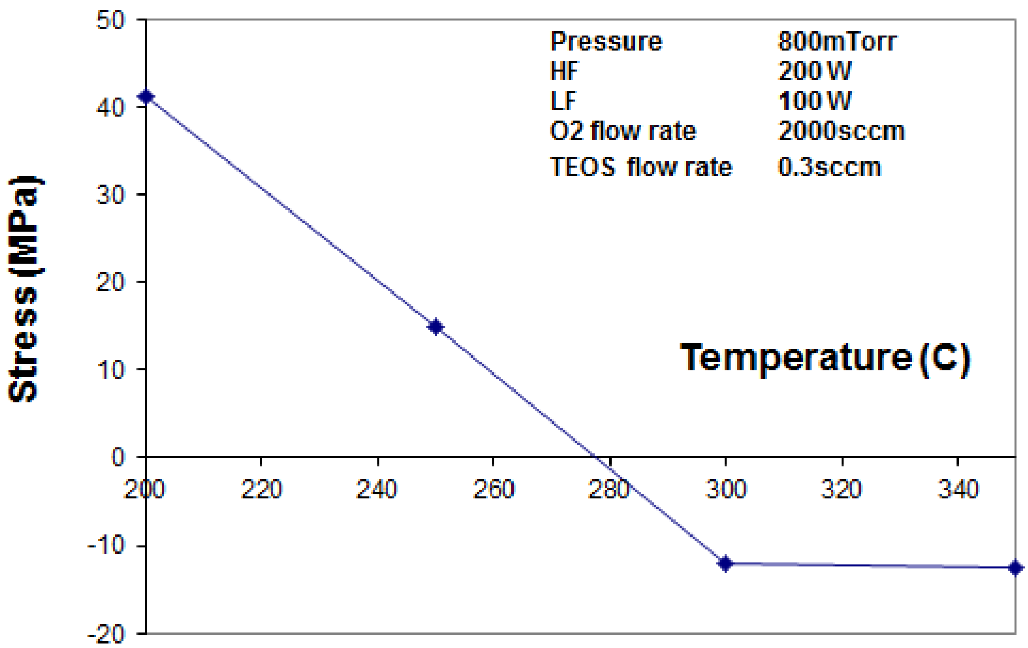



**Fig. 3.** Variation of the deposition rate (a) and residual stress (b) with the temperature.

### 3.3. Influence of power in high frequency mode

With low frequency disabled, the influence of high frequency (HF) power variation was investigated.

The main role of HF is to aid in breaking the bonds within the precursors in the deposition process. This can explain why the deposition rate rises with increasing HF power.

The increased RF power leads to higher electron density and therefore there is a relatively larger population of high-energy electrons.

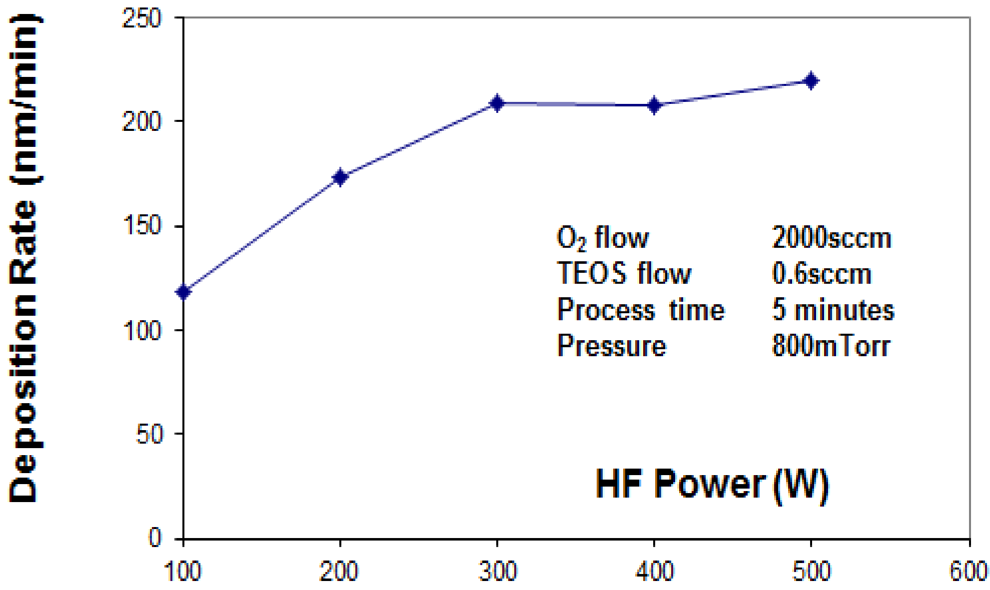
These high-energy electrons yield a higher ionization and dissociation rate, which consequently results in a higher deposition rate.

This increase begins to taper at the higher powers, possibly indicating a saturation of the rate of bond breaking.

The refractive index dips slightly at higher HF powers, which may indicate that the higher rate of bond-breakage may result in silicon dioxide film that is less dense.

The uniformity worsens exponentially with an increase in HF power which warrants that there be a maximum of 300 W to achieve uniformity suitable for industrial applications.

**a)**



**b)**

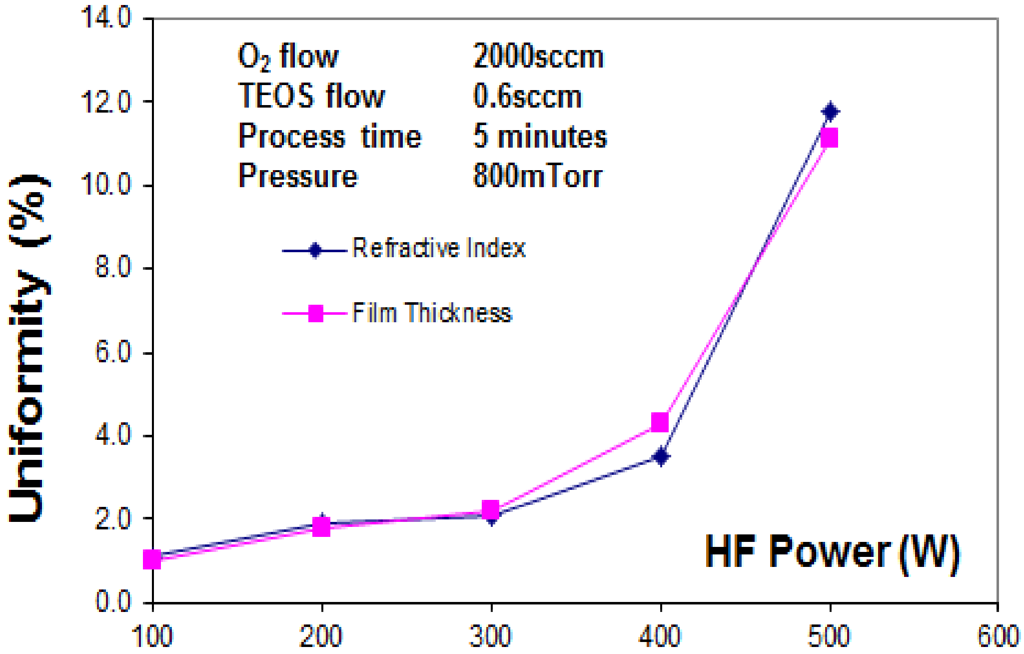



**Fig. 4.** Variation of the deposition rate (a) and residual stress (b) in HF mode.

### 3.4. Influence of power in low frequency mode

In order to investigate the effects of low frequency (LF) power, HF power mode was disabled whilst LF power was varied from 100 W to 500 W.

However, even up till 500 W, there was no deposition on the wafers.

This would indicate the necessity of HF to break the bonds of the precursors in TEOS PECVD and that LF alone is not able to break the bonds of the TEOS precursors.

Characteristic to the LF mode is the "ion bombardment" phenomenon.

At high frequency (13.56 MHz) only the electrons are able to follow the RF field while the ions are "frozen" in place by their heavier mass.

The crossover frequency at which the ions start following the electric field is between 1 MHz and 5 MHz depending upon the mass of ions.

Consequently, below 1 MHz, the ion bombardment is significantly higher.

As a result, a combination of HF and LF mode can lead to a densification of the thin $SiO_2$ layer due to the ion bombardment.

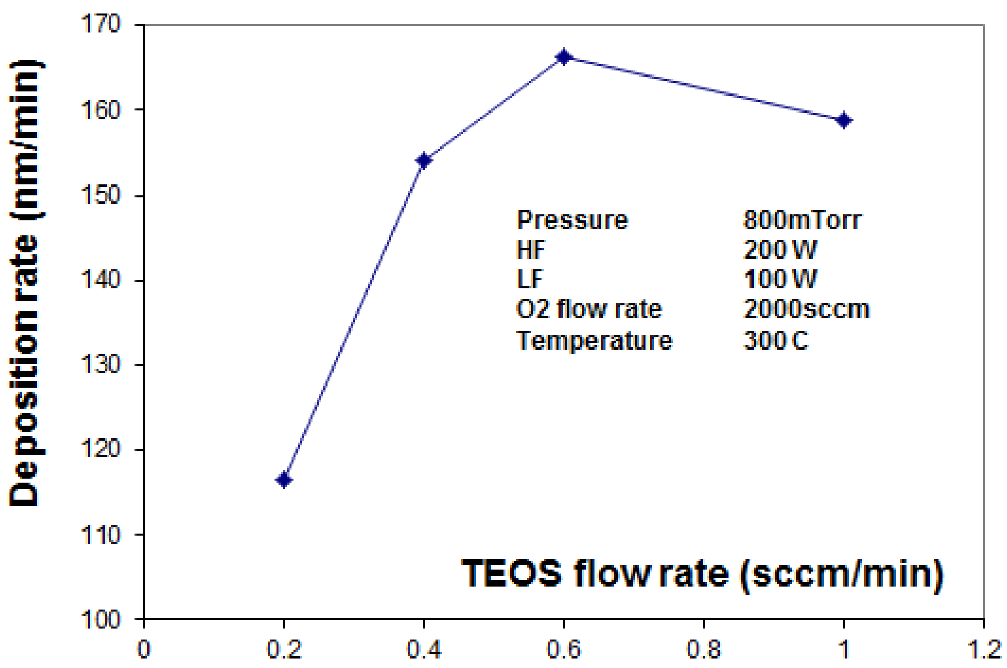
### 3.5. Influence of $O_2$ flow rate

The results showed that there was a minimal decrease in the $SiO_2$ deposition rate with respect to changes to the $O_2$ flow rate. One possible contribution is that the $O_2$ flow rate even at 1500sccm is already highly saturated such that further increase in the $O_2$ flow rate would not result in a noticeable increase of the deposition rate. However this little explains the gradual decrease in the deposition rate from 1500sccm to 2000sccm. Another possible explanation, as found by Granier et al [ ], is that the $O_2$ itself contributes to the chemical etching of the film. It was demonstrated the oxygen atoms are responsible for the chemical etching of $SiO_xC_yH_z$ films. Whilst an increase in $O_2$ density may increase the rate at which TEOS is oxidized, fragmented and deposited as $SiO_2$, this competes with the increase in etching brought about by a higher $O_2$ flow rate**.** There was no relevant variation on stress and refractive index with the $O_2$ flow rate variation.

### 3.6. Influence of TEOS flow rate

With $O_2$ flow rate constant at 2000sccm, deposition rate is found to be increasing with TEOS (Figure 5a). There is little effect when the TEOS flow rate is above 0.6sccm probably due to saturation of TEOS. At lower TOES flow rate, the film stress is compressive as compared to at higher levels where the film stress is tensile (Figure 5b). The changes on TEOS flow rate had no effect on the refractive index and the film uniformity (less than 3%).

**a)**



**b)**

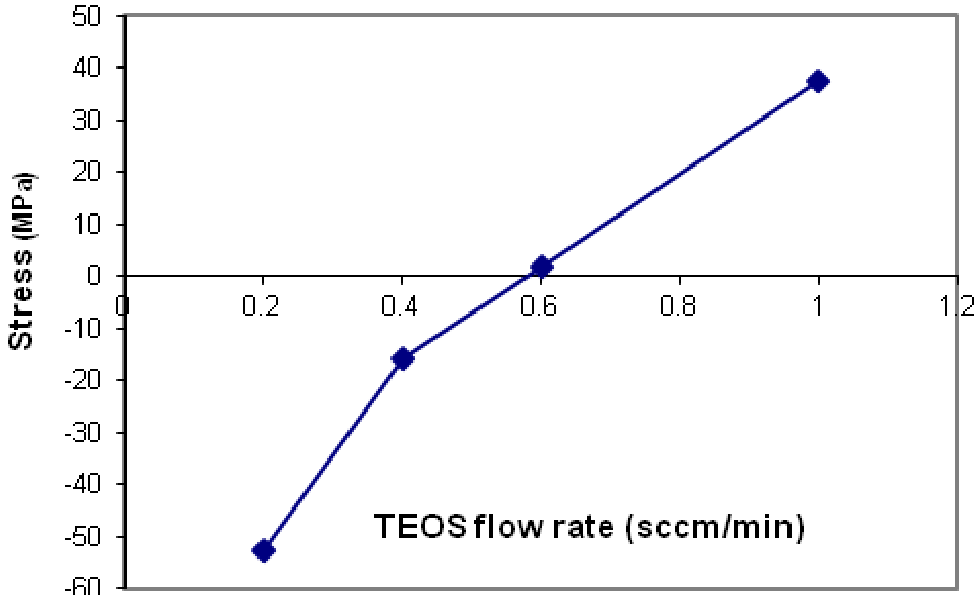



**Fig. 5.** Variation of the deposition rate (a) and residual stress (b) with the TEOS flow rate.

## 4. Discussions

Based on the results, pressure is best maximized to take advantage of the better deposition rate and Uniformity at higher pressure. The upper limit for chamber pressure for the TEOS PECVD process at 300 °C is 1000 mTorr over which the film will have defects.

The lower limit for optimization of pressure is > 700 mTorr below which its uniformity is below industrial applications standard (above 3% non-uniformity). HF Power has a tradeoff between deposition rate and uniformity. An HF Power of 200-300W provides the best compromise. LF Power has insignificant effects on the deposition rate, refractive index and uniformity, affecting most prominently on the stress of thin films, but can play an important role in film densification in the mixt frequency deposition (HF+LF modes). $O_2$ flow rate is taken to be at 2000sccm which is the highest permissible by the PECVD machine coupled by the TEOS flow rate taken at 0.6sccm to produce a high deposition rate. Above 0.6sccm TEOS flow rate for a 2000 sccm $O_2$ flow rate, there is little increase in the $SiO_2$ deposition rate. At a high TEOS/$O_2$ ratio, the thin film is damaged. A lower temperature increases the deposition rate, low temperatures also tend to cause the film to be damaged. It is noted that the film tends to have structural defects under these 3 extreme conditions; high pressure, low temperature and low $O_2$/TEOS mass flow ratio.

Figure 6 illustrates a SEM picture with a PECVD TEOS deposition over a 18μm-depth trench with vertical walls. Can be noticed a very good step coverage obtain for a PVD system.

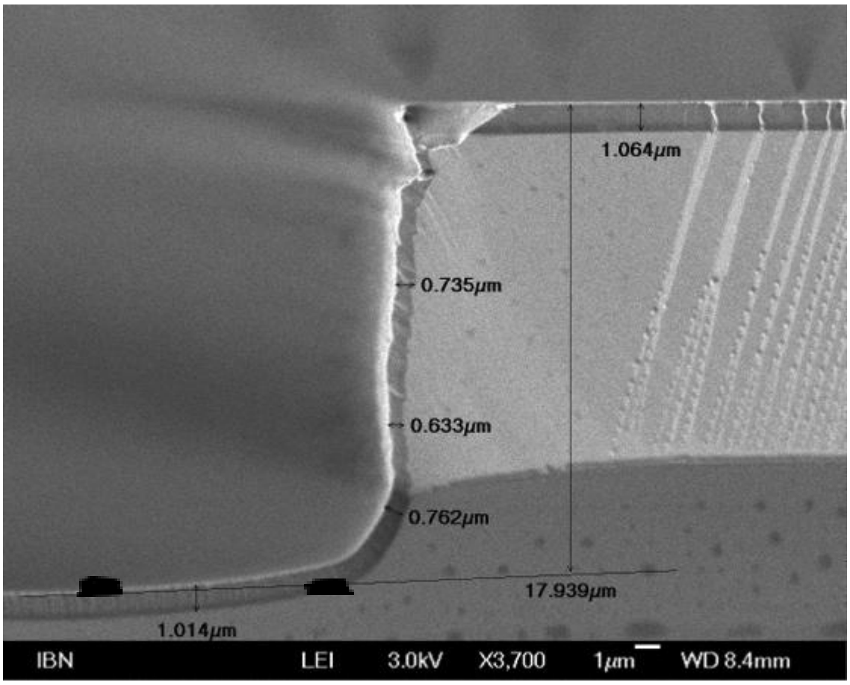



**Fig. 6.** SEM image with step-coverage of a TEOS deposition using a PECVD reactor

## 5. Conclusions

The deposition of TEOS on PECVD reactors are a viable solution for MEMS and IC applications. [27-33] We demonstrate the controlling the deposition process deposition rates above 200nm/min can be achieved. Based on the results, we are able to recommend the following parameters for the TEOS PECVD process.

- For a ***low stress TEOS*** with deposition rate of 210 nm/min (nonuniformity 1.3%), a residual stress -9.5 MPa and refractive index 1.456, the process parameters were: process temperature of 285 °C, pressure of 875 mTorr, HF Power of 200 W, LF Power of 100 W, $O_2$ flow rate 2000 sccm/min, TEOS flow rate 0.6 sccm/min

- For ***low temperature*** (200 °C) TEOS deposition process with a deposition rate of 220 nm/min (nonuniformity of 1.2%), a residual stress of -127 MPa and a refractive index of 1.457, the process parameters were: Pressure of 850 mTorr HF Power of 200 W, LF Power of 50 W, $O_2$ flow rate of 2000 sccm/min TEOS flow rate of 0.3sccm/min.

The results of our investigations related to the parameter influence on TEOS deposition are summarized in the table 1.

**Table 1.** Summary table of results

| **Process parameters** | **Dep. rate** | **Uniformity** | **Stress** |
|---|---|---|---|
| ↑ Pressure | ↑ | ↑ | ~ |
| ↑ Temp | ↑ | ~ | ↑ compressive |
| ↑ HF Power | ↑ | ↓ | ↑ compressive |
| ↑ LF Power | ~ | ~ | ↑ compressive |
| ↑ O2 | ↓(min) | ~ | ↑ tensile |
| ↑ TEOS | ↑ | ~ | ↑ compressive |

*Legend:* ~ (insignificant),
*The changes in refractive index (not shown) were in the range between 1.44-1.47

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