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Approximation of PE-MOCVD to ALD for TiN Concerning Resistivity and Chemical Composition

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Abstract**—**The miniaturization of circuits is advancing. During chip manufacturing, structures are filled for example by metal organic chemical vapor deposition (MOCVD). Since this process reaches its limits in case of very high aspect ratios, the use of alternatives such as the atomic layer deposition (ALD) is possible, requiring the extension of existing coating systems. However, it is an unsolved question to what extent MOCVD can achieve results similar as an ALD process. In this context, this work addresses the characterization of a metal organic vapor deposition of titanium nitride. Based on the current state of the art, the film properties coating thickness, sheet resistance, resistivity, stress and chemical composition are considered. The used setting parameters are temperature, plasma gas ratio, plasma power, plasma treatment time, deposition time, deposition pressure, number of cycles and TDMAT flow. The derived process instructions for unstructured wafers and inside a structure with high aspect ratio include lowering the process temperature and increasing the number of cycles, the deposition and the plasma treatment time as well as the plasma gas ratio of hydrogen to nitrogen (H_2:N_2). In contrast to the current process configuration, the deposited titanium nitride (TiN) layer is more uniform inside the entire test structure. Consequently, this paper provides approaches to employ the MOCVD for structures with increasing aspect ratios.

Keywords**—**ALD, high aspect ratio, PE-MOCVD, TiN

I. Introduction

THE demands on processes for the semiconductor industry are becoming more and more complex. In this context, high aspect ratios are of particular interest.

Applied technologies like the plasma enhanced MOCVD (PE-MOCVD) with the precursor TDMAT already provide uniformly deposited TiN layers with sufficient electrical properties for small aspect ratios. The PE-MOCVD TiN process consists of two major steps: one is the MOCVD process itself, which is followed by a plasma treatment. Purpose of the plasma treatment is the layer densification and removal of contaminations. Due to its directness, plasma treatments are efficient for planar films, but less for films at vertical structures. However, the throughput and the step coverage decrease for increasing aspect ratios. In order to avoid even more disadvantages, e.g. the rising concentration of contamination, this paper introduces the new technology of cyclic PE-MOCVD. It is investigated whether this adaptation of MOCVD can be applied for structures with high aspect ratios and to what extent MOCVD can provide similar results as ALD. So far the advantages of ALD are uniform and pure deposition with low resistivity values for both unstructured wafers and in structures with high and small aspect ratios [1].

ALD is a CVD process for the deposition of very thin layers. The starting materials are sequentially and cyclically injected into the reaction chamber [1]. The starting material of a reaction step does not react in ALD process with itself or with its ligands. During the plasma assisted atomic layer deposition (PAALD) radicals are formed within of the plasma gas. Advantages are the increased reaction rates, improved process efficiency, the increased fragmentation of the starting material and the improved removal of product molecules. The production of pure TiN layers with the precursor TDMAT can be achieved with impurity levels of carbon and oxygen below 6 at.% by PAALD with long plasma treatment times [2]. An in-situ X-ray diffraction measurement proves non-porous ALD films thus having excellent diffusion barrier properties. Further advantages of ALD processes compared to CVD and PVD methods are presented in [3]. These include, for example, uniform thickness concerning large area, good step coverage, small hole density and reduction of carbon contamination caused by the gas phase reaction. Compared to other CVD processes TiN films produced by metal organic atomic layer deposition (MOALD) need only low deposition temperatures between 175 °C and 210 °C and have a uniform deposition with very good resistivity values below 500 µΩ·cm. Reasons are the separate supply of the precursor and reactant gases as well as prolonged argon flushing times with an increased argon flow. By means of MOALD, a step coverage of almost 100% can be achieved in 800 nm deep and 400 nm wide contact holes [3].

According to the current state of the art there are no adequate studies for implementation of ALD properties using a MOCVD method on 200 mm wafers, in order to achieve the desired TiN film properties. Thus, this paper provides approaches to employ the PE-MOCVD for structures with increasing aspect ratios. The documentation and evaluation of TiN layer thickness, sheet resistance and resistivity, chemical composition and stress are performed in order to characterize a selected PE-MOCVD process. ALD with the desired properties is the comparative method.

II. Experimental

A. Process Conditions

The test of the gradual parameter adaptation of a PE-MOCVD is performed on unstructured and structured wafers. The objective is the deposition of very thin layers with a few atomic layers per cycle to approximate the results of an ALD to a high extent. The introduced cyclic PE-MOCVD is referred to as "ALD*" in the following. Fig. 1 illustrates a

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comparative summary of the processes covered in this paper.

An advantage of ALD* compared to the reference process of the current PE-MOCVD is a better leaching of impurities from the deposited layer in the plasma step. Uniform separation behaviour and a reduced surface roughness of the individually deposited layer per cycle are intended to allow a more even growing of further layers.

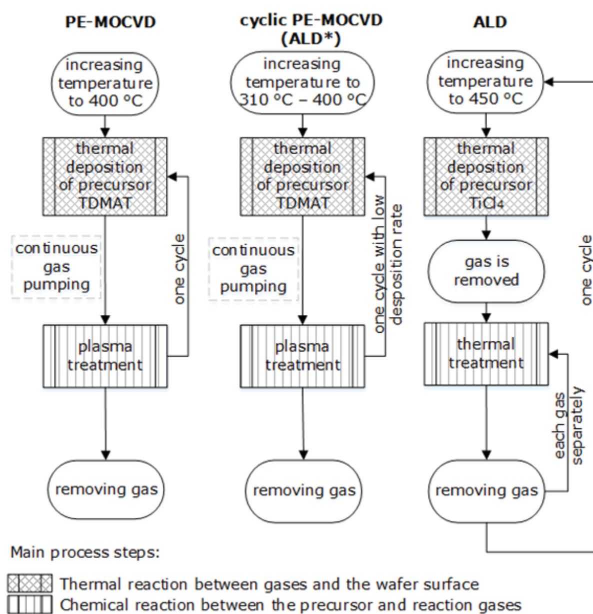

Fig. 1 Process schemes of PE-MOCVD, cyclic PE-MOCVD and ALD

The varied parameters are process temperature T, number of process cycles n_c, deposition pressure p_D, deposition time t_D, plasma treatment time t_{PT}, plasma gas ratio $R_{H2:N2}$ of hydrogen to nitrogen and TDMAT flow $\dot{V}_{TDMAT}$.

The used test structure consists of a silicon dioxide layer, which is about 100 nm thick and applied to the silicon substrate. To save space, the trench is kept as narrow as possible. The electrical contact of the component is performed via the silicon in the lower trench region. A TiN layer is required as a diffusion barrier and adhesive layer for the subsequently applied tungsten layer.

B. Film Deposition Parameters

Depositions of thin TiN films from TDMAT precursor are performed using a TxZ chamber on an AMAT Centura in a PE-MOCVD mode [4]. A reference for the tests on unstructured wafers is the PE-MOCVD process for a 10 nm TiN layer. To achieve very thin layers with similar properties, the parameters of the reference PE-MOCVD process are reduced by approximately 25% to 30%. They are summarized in Table I and form the basis for the ALD* process which is the proposed approximation to ALD. The parameters power and pressure of the plasma treatment are maintained.

After individually performed parameter variations, the properties of the generated TiN layers are analyzed. A parameter reduction of more than 25% could lead to unstable deposition, since the difference between chamber and back side pressure could no longer be sufficient, so that the wafer could slip in the chamber during the process.

TABLE I
PROCESS PARAMETER OF PE-MOCVD AND ALD FOR A TIN-LAYER THICKNESS OF 10 NM

Parameter	PE-MOCVD	ALD*
T [°C]	400	310; 330; 360; 380; 400
p_D [kPa]	0.7	0.5
$\dot{V}_{TDMAT}$ [sccm]	375	263
$R_{H2:N2}$	1,5	2
t_{PT} [s]	33	23

ALD* = PE-MOCVD approximated to ALD

C. Film Characterization

Optical properties of the TiN deposited on silicon wafers as well as their thickness are determined by means of *Bruker D8 Discover* X-ray reflectometry (XRR). The structure of the films is investigated using *Hitachi S5200* scanning electron microscopy (SEM) and *FEI Tecnai F20G2* transmission electron microscopy (TEM) equipped with energy dispersive X-ray (EDX) to determine the chemical composition of the film. The chemical composition of films on unstructured wafers is performed by *PHI 670 Auger Nanoprobe* Auger electron spectroscopy. Other properties like stress and resistance are measured with *MX 208 E&H Metrology* and *KLA-TENCOR RS100*.

In the following sections, the individual tests are evaluated on unstructured and structured wafers.

III. UNSTRUCTURED SI-WAFERS

A. Comparison of TiN Deposited by PE-MOCVD and ALD

The following film properties of the deposited TiN by PE-MOCVD and ALD were examined: thickness d of TiN, sheet resistance R, resistivity ρ, stress σ and density ρ_D. The results are summarized in Table II and are intended for comparison with the following experiments.

TABLE II
LAYER PROPERTIES DEPOSITED BY PE-MOCVD AND ALD

	PE-MOCVD	ALD
d [nm]	9	9
R [Ω]	254	124
ρ [µΩ cm]	230	113
σ [MPa]	-265	325
ρ_D [g/cm³]	3.1	5.5

B. Properties of the PE-MOCVD TiN Layer at Temperature Variation

For an approximation to ALD the influence of temperature on the layer characteristics was observed initially for five temperature ranges. A number of 20 process cycles was chosen for each temperature to deposit the minimal layer thickness per cycle, so that even at the lowest temperature a closed TiN layer can be expected. Due to chamber technical

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constraints the thermal limits of the created basic process ALD* are 310 °C and 400 °C. The parameters are set according to Table I.

The measured data of layer thickness in Fig. 2 indicate that the precursor TDMAT decomposes nonlinearly concerning the temperature, but exponential. The quantitative dependence between the reaction rate constant and the temperature results from the evaluation of the natural logarithm of the reaction rate constant concerning the reciprocal of the temperature. The basis of this consideration is the Arrhenius relationship.

Fig. 3 shows that the resistivity decreases up to a temperature of about 360 °C and increases above this temperature. Low resistivity of TiN films can be achieved below 380 °C. The decrease in resistivity can be attributed to the growing grain size with increasing layer thickness. The stress curve in Fig. 3 declines at about 330 °C. With increasing temperature up to 360 °C, the stress amount increases and decreases again with further increase in temperature.

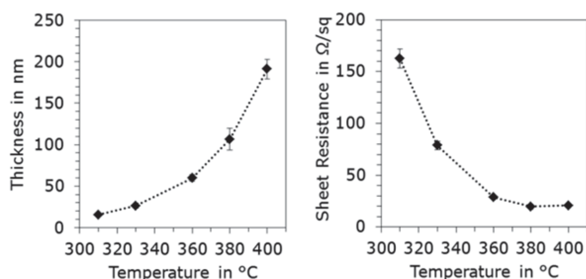

Fig. 2 Thickness and sheet resistance of TiN films deposited at 750W and 23 s/cycle deposition time and different deposition temperatures

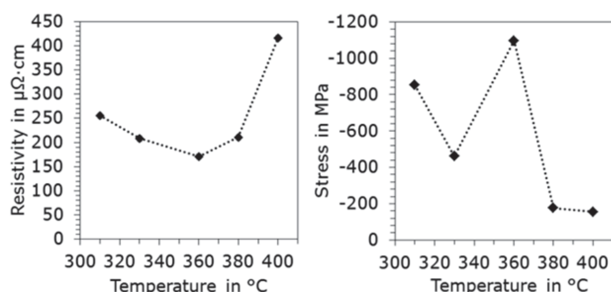

Fig. 3 Resistivity and stress of TiN films deposited at 750 W and 23 s/cycle deposition time and different deposition temperatures

C. Crystal Structure and Morphology of the Films on Unstructured Si-Wafers

XRD patterns of titanium nitride films deposited at 330 °C to 400 °C for 33 s/cycle and power of 750 W are shown in Fig. 4. The diffraction pattern of TiN Powder Diffraction File (PDF) [5] is displayed for comparison. The diffraction peaks corresponding to (111), (200), (220), (311) and (222) planes of TiN are clearly visible. The corresponding 2θ values are 36.7°, 42.6°, 61.8°, 74.1° and 78.0°, respectively.

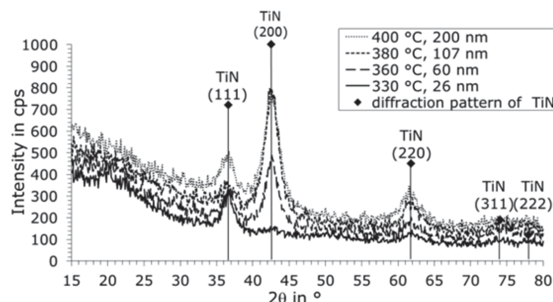

Fig. 4 XRD patterns of TiN films deposited at 750 W and 23 s/cycle deposition time and different deposition temperatures

Noticeable is the curve at a process temperature of 330 °C because it does not contain a peak for the lattice plane TiN (200). The peak is formed only from a starting temperature of 360 °C. The intensity profile increases with increasing temperature. However, the lattice plane TiN (200) is almost identical for 380 °C and 400 °C. Thus it can be assumed that temperatures greater than 330 °C lead to structural changes in the layer, which could explain the increasing stress behaviour at 360 °C in Fig. 3. The amount of stress decreases above and below this temperature. One reason for this behaviour might be an incipient lattice relaxation.

D. Varying Number of Process Cycles on Unstructured Si-Wafers

Further characterization is executed regarding the number of process cycles. The variation comprises 7, 15, and 20 cycles at a process temperature of 310 °C. Further basic process parameters are unchanged in these experiments. The results concerning film thickness, sheet resistance, resistivity and stress are illustrated in the graphs of Fig. 5.

The layer thickness is continuously growing with increasing number of cycles, whereby the sheet resistance decreases. An increasing number of cycles and film thickness causes a stress transition from tensile to compressive stresses in the TiN layer. Furthermore, a decrease in resistivity with increasing number of cycles can be observed. A thin layer thickness leads to a higher resistivity. The course of the resistivity could thus be caused by the fluctuating deposition rate of about 0.5 nm, 1 nm, and 0.8 nm per cycle. The small particle sizes with a large grain boundary share can increase the resistivity and the increased surface roughness also contributes to rising resistivity. In subsequent tests, a total number of 20 cycles is used initially because of the favorable film properties at higher cycles. After a satisfying adaption, the number of cycles should be reduced in order to achieve a low deposition rate at a low total process time.

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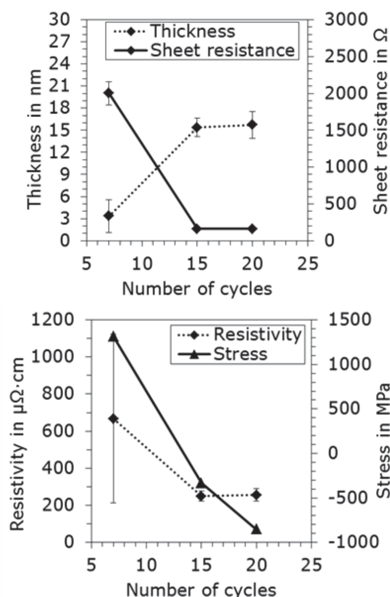

Fig. 5 Variable number of process cycles at a temperature of 310 °C

E. Chemical Composition of TiN on Unstructured Si-Wafers

In order to investigate the impact of the plasma gas ratio $R_{H2:N2}$ to the deposition of TiN, a temperature T = 310 °C was chosen. The film and filling properties are initially examined at a plasma gas ratio $R_{H2:N2}$ = 1.5 and a plasma treatment time t_{PT} = 23 s. This process is called "ALD*310_1.5". Under consideration of preliminary examinations, the plasma gas ratio is set to $R_{H2:N2}$ = 2 in the subsequent experimental "ALD*310_2". For this experiment, the plasma treatment time is extended to t_{PT} = 30 s to achieve desired layer properties.

The determined layer properties and chemical compositions for both performed experiments are shown in Table III and Fig. 6. The film properties of the approximation to ALD with a small plasma gas ratio show a significant stress increase with respect to the preliminary tests. The effect of increasing the plasma gas ratio and the plasma processing time is in the experiment ALD*310 °C_2 is noticeable. The resistivity decreases by about 13%, which could be due to the increased hydrogen supply [6]. The small layer thickness reduction and the increasing compressive stresses may be caused by the improved efficiency of the plasma treatment and the associated layer compression.

The chemical composition shown in Fig. 6 provides further findings for the different resistivity values in Table III.

TABLE III
TIN-LAYER PROPERTIES OF PE-MOCVD APPROXIMATED TO ALD

	PE-MOCVD	ALD	ALD*310°C_1.5	ALD*310°C_2
d [nm]	9	9	16	15
R [Ω]	254	124	163	152
ρ [µΩ cm]	230	113	256	223
σ [MPa]	-265	325	-852	-888

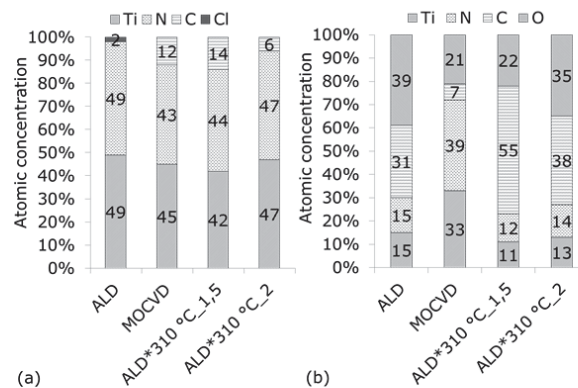

Fig. 6 Comparison of chemical composition at (a) layer centre and (b) layer surface of TiN films deposited by different processes

The increased parameters of the plasma treatment of the experiment ALD*310_2 result in significantly reduced impurities of only about 6 at.% in the TiN layer centre compared to Experiment ALD*310_1.5. A layer composition as in ALD is almost achieved because titanium and nitrogen were determined with about 47 at.% each which corresponds to the ratio of 1:1. A similar approximation is reflected in the surface composition. The presumption of the addition of air constituents on rough surfaces is confirmed.

The PE-MOCVD process caused a more than twice as high titanium and nitrogen concentration and the lowest carbon and oxygen concentration at the film surface compared to all other selected experiments and thus shows the best surface composition. Another explanation is the average diffusion distance of the diffusing substance in the layer according to

$$x = \sqrt{2 \cdot D \cdot t} \tag{1}$$

with a diffusion time t and the diffusion coefficient D [7].

During a given time, impurities can be removed from the entire layer thickness if the diffusion distance is at least as large as the current layer thickness. Since the deposited layer thickness during a particular time interval results from the deposition rate, the efficiency of the removal of impurities is affected by the deposition rate per cycle. Existing impurities can thus be removed only up to a certain layer depth.

F. Properties of the PE-MOCVD TiN Layer at TDMAT Flow Variation

For a further characterization of the PE-MOCVD process, the TDMAT flow is varied at different temperatures. First, the layer thickness is increased in order to obtain a closed TiN layer for the tests at 310 °C. In this context, the deposition time is extended by 7 s to a total duration of 19 s. The desired layer thickness for a number of seven cycles should not be less than 6 nm. Since Section III. A shows a highly temperature dependent deposition, different TDMAT flows are selected for target layer thicknesses of about 6 nm to 10 nm to subsequently enable a sufficient TiN deposition inside the test structure.

It is assumed that, with increasing temperature a smaller

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amount of TDMAT is needed because the TDMAT decomposition and thus the deposition rate increase. The upper limit of 500 sccm and the stable lower limit of 50 sccm result is limited by the used mass flow controller *Horiba STEC 4400*. The measured TiN film properties are shown in Fig. 7.

The extrapolated curves allow a presumption of the layer thickness profile at higher TDMAT flow rates. For 310 °C a stagnant deposition rate of 0.4 nm per cycle for a TDMAT flow of 400 sccm to 500 sccm is visible. In medium and high temperature range, at 360 °C and 400 °C, an increase in the film thickness can be observed with increasing TDMAT amount. For the deposition at 400 °C, the layer growth increases at the beginning by about 1.4 nm for 100 sccm and sinks to about 0.7 nm with increasing TDMAT flow of 50 sccm. In Figs. 7 and 8, the associated sheet resistances and resistivities for the lowest temperature show remarkably high values. The stress measurements illustrate a tensile-strained TiN layer, while higher temperatures produce compressive stresses. Possible reasons are discussed in [8]–[12].

A minor change of the TDMAT flow at a given temperature range only slightly affects the layer properties and, therefore, seems to be of minor importance for filling structures.

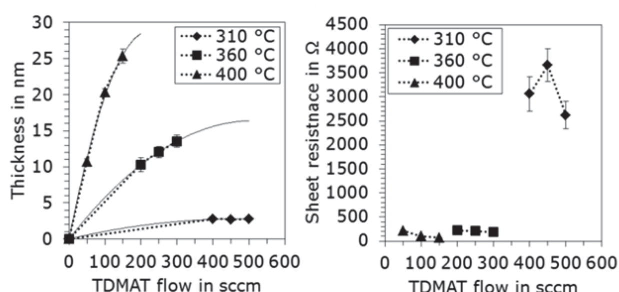

Fig. 7 Thickness and sheet resistance of TiN films deposited at different temperatures and different TDMAT flows with seven cycles

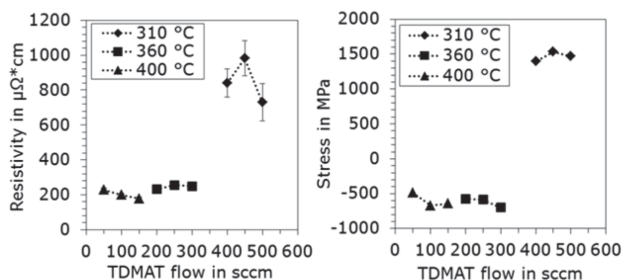

Fig. 8 Resistivity and stress of TiN films deposited at different temperatures and different TDMAT flows with seven cycles

IV. Application to Structured Si-Wafers

In this section results of the approximation of PE-MOCVD to ALD for unstructured wafer to a test structure with a high aspect ratio are described. In this context, the basic process ALD*310°C_1.5 with 20 cycles is analyzed after deposition in the test structure. Then, the experiment ALD*310°C_2 is reduced from 20 to 8 process cycles (ALD*310°C_2a) to decrease process time and performed on the test structure for comparison.

To evaluate properties of the deposited TiN layer in the structure and to be able to compare the deposited layer thickness to planar wafers, the measures filling level *FL* and deposition level *DL* are introduced. They can be used as quality criteria. The filling level

$$FL = \frac{2 \cdot d_s}{b} \tag{2}$$

is the ratio of twice the thickness d_s and the structure width b = 60 nm in the area of the sidewalls. For all trench heights, the deposition level is the proportion of the deposited layer thickness d_s in the structure with respect to the layer thickness d_{us} which could be achieved in an unstructured wafer according to

$$DL = \frac{d_s}{d_{us}}. \tag{3}$$

The layer thickness d_s represents the arithmetic mean of the corresponding measured values. For the calculation of both quality criteria, an exemplary trench is evaluated for each test. Fig. 9 shows the results for the deposited TiN layer by PE-MOCVD at 400 °C, two process cycles and 7 nm thickness. With increasing structure depth, a decrease in the fill level and deposition level up to 0% at the trench bottom can be seen.

By changing the number of cycles of the PE-MOCVD process to five cycles and with a layer thickness of about 17 nm, deposition can be achieved down to the bottom. However, the deposited layer is irregular and its thickness of approximately 5 nm on the sidewall coverage is too thick, which hinders subsequent W deposition.

The application on the test structure of the approximations to an ALD at 310 ° C and a varying number of cycles can also be seen in Fig. 9. The approximately equal target layer thicknesses on planar wafers of about 18 nm of the PE-MOCVD process at 400 °C and about 16 nm for the approximation of ALD at 310 °C with four times the number of cycles of 20 show significant variations in the filling and deposition levels. The filling level on the trench entrance increases with decreasing temperature from 53% to 61% and has more than twice the thickness at the sidewalls. The comparison of the structure bottoms for the two tests shows a strongly increasing deposition level of 11% to 109%. Thus deposition thicknesses of about 8.5 nm to 25 nm at the trench bottom are reached. To minimize the target layer thickness to about 5 nm, the number of cycles is reduced to 8.

For the experiment with the reduced number of cycles from 20 to 8, the filling level and deposition level decreased in almost all areas by about one half. The initially pronounced edge overhangs have been reduced and thus offer better preconditions for filling with tungsten.

In comparison with the PE-MOCVD processes it is clear that the approximations to ALD processes provide favorable structure fillings. A strong elemental analysis signal of

titanium is observed, suggesting an increased proportion. Furthermore, contamination by carbon at the lowest bottom point is barely recognizable at a plasma gas ratio $R_{\mathrm{H2:N2}} = 2$.

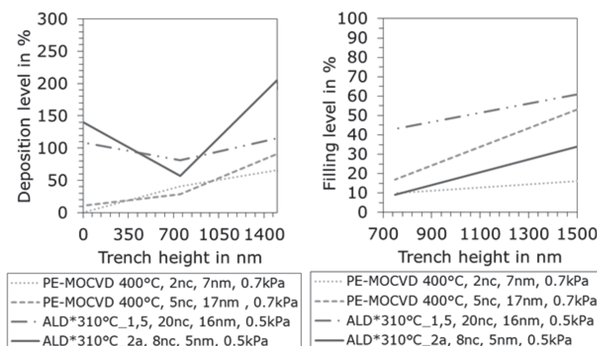

Fig. 9 Quality criteria: DL (deposition level) for the whole trench from upper section to bottom at 0 nm-1500 nm and the FL (filling level) for the lateral wall at 750 nm-1500 nm

V. Conclusion

The Approximation of PE-MOCVD to ALD for TiN concerning the resistivity and chemical composition was investigated. For this purpose, a PE-MOCVD was characterized. Due to insufficient deposition properties for structured wafers, a new type of MOCVD was introduced and investigated with respect to specific features. This cyclic PE-MOCVD (ALD *) provides advantages such as better filling properties of structures with very high aspect ratios.

A minor change in the TDMAT flow affects the layer properties only very slightly within a given temperature range and is therefore of low significance for filling structures. In addition, it can be stated that a uniform layer growth with declining deposition in the approximated ALD* processes is possible.

It could be shown that, for a fixed plasma time period, impurities may be removed within the total layer thickness, when the diffusion distance is at least as large as the current layer thickness. Since the deposited layer thickness during a particular time interval results from the deposition rate, the efficiency of the removal of impurities is affected by the deposition rate per cycle. Developed ALD* processes could therefore be observed to minimize the existing carbon contamination. Lower temperatures with advanced deposition and plasma treatment times favor not only the deposition level in deep structure regions by sufficient TDMAT adsorption, but can also allow an improved plasma gas treatment.

From a throughput point of view plasma enhanced MOCVD of TiN is the fastest process, followed by the cyclic MOCVD process, reviewed in this paper. Lowest throughputs are reported for conventional ALD processes. However, fast ALD processes (spatial ALD) have recently gained momentum and can show comparable throughputs with the cyclic MOCVD process [3], [13]–[15].

The results and determined parameters contribute to expand the fields of application of PE-MOCVD with regard to ALD for demanding shapes and sizes of structures, trenches and vias.

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