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In situ Layer Characterization by Spectroscopic Ellipsometry at High Temperatures

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Abstract

The demand for increased cost-effectiveness in semiconductor manufacturing is the driving force for the development of *in situ* and in-line measurement tools. Some of the most critical manufacturing steps are high-temperature processes such as thermal oxidation and chemical layer deposition. Solutions for accessing batch furnace processes by high-temperature single wavelength and spectroscopic ellipsometry for layer thickness and composition control have been proposed and studied intensively in the past. These techniques require comprehensive knowledge of the optical parameters at high temperatures. Therefore, a systematical study has been started to determine the optical high-temperature data (refractive index, extinction coefficient) of relevant semiconductor materials. Moreover, optical data of amorphous and polycrystalline silicon at high temperature are under investigation. All measurements were performed with a spectroscopic ellipsometer integrated in a vertical LPCVD-batch furnace. Optical access is provided by a special beam-guiding system. The established data are used to develop models for ellipsometric *in situ* monitoring of layer structure such as thickness, roughness, crystallinity, or density. Accurate and reliable optical models are particularly required for *in situ* monitoring of polycrystalline silicon because the optical properties vary considerably, depending on the deposition conditions. A Bruggeman-Effective Medium Approximation (B-EMA) is used to calculate the dielectric function of the layer. This method allows to characterize multilayer structures and to obtain all layer thicknesses and optical layer characteristics from one SE measurement. These models are implemented into the measurement programs and they are already used in a commercial spectroscopic ellipsometer for use in industrial applications.

INTRODUCTION

Today, *in situ* and in-line metrology is often regarded as the key to achieve increased cost-effectiveness in semiconductor manufacturing. The main advantages of integrated metrology in processing equipment (*in situ* and in-line) are a reduced number of monitor wafers and direct process control instead of off-line monitoring (1). Especially with respect to the processing of 300 mm wafers, the demand for process control without monitor wafers wherever possible is extremely high. Moreover, integrated metrology provides higher tool utilization through optimized maintenance instead of preventive maintenance and shorter ramp-up cycles of new processes.

In semiconductor processing, the formation of thin films in high temperature processes is one of the key techniques. Thermal oxidation and chemical layer deposition comprise some of the most decisive manufacturing steps such as gate oxide formation, deposition of stacked dielectrics, and deposition of conductive layers like polysilicon or amorphous silicon. The process control (layer thickness and quality) is completely carried out off-line. *In situ* monitoring of layer growth and composition as well as end-point detection support new control techniques to provide a better process stability without the use of monitor wafers and without additional handling for process and quality control. Solutions for accessing batch furnace processes by high-temperature single wavelength and spectroscopic ellipsometry have been proposed and studied intensively in the past (1)(2). The major requirement for the use of these techniques is to be in the exact knowledge of the optical material properties at high temperatures up to 1000°C. As these data are in most cases not available, a systematical study has been started to determine the optical high-temperature data (refractive index, extinction coefficient) of relevant semiconductor materials. In the first step, the optical high-temperature data up to 900°C of crystalline silicon, silicon dioxide, and silicon nitride were established (1). Now, these data are used to develop models for ellipsometric *in situ* monitoring of layer structure such as thickness, roughness, crystallinity, or density. An important layer material in semiconductor manufacturing is polycrystalline silicon because of its application as e.g. gate material or interconnects. Especially for this material accurate and reliable optical models are a precondition for *in*

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11 December 2024 23:06:22

situ monitoring because the optical properties vary considerably depending on the process conditions. Therefore, the change of the polycrystalline silicon structure depending on the temperature is presently under investigation. In order to calculate the dielectric function of the polycrystalline layer, a Bruggeman-Effective Medium Approximation (B-EMA) is used by employing a mixture of materials with dielectric functions. These functions can be determined independently. As studies showed in the past, this method allows to characterize multilayer structures and to obtain all layer thicknesses and optical layer characteristics from one SE measurement (3)(4).

EXPERIMENTAL

The off-line reference measurements were carried out with a SOPRA ES4G spectroscopic ellipsometer. For the *in situ* measurements a spectroscopic ellipsometer (SOPRA MOSS OMA) integrated into a vertical batch furnace for thermal oxidation and low pressure chemical vapour deposition (LPCVD) was used. Spectroscopic ellipsometry was chosen because of the possibility to measure on absorbing layers like polycrystalline silicon or in multilayer structures (5). As vertical furnaces in semiconductor manufacturing do not provide any optical access for a ready-to-use integration of metrology tools, a special beam guiding system is employed. This system keeps the modifications of the furnace geometry at a minimum. In Fig. 1, the ellipsometer arrangement and the beam path with the beam guiding system are shown. The two ellipsometer heads are placed side by side and are mechanically coupled to the base plate of the furnace. Together with the base plate and the wafer carrier, they form a mechanical unit that moves vertically with the boat loader. The light beam of the ellipsometer is guided through the base plate into the furnace tube and directed onto the wafer by quartz glass prisms operating in total internal reflection (TIR) mode (1).

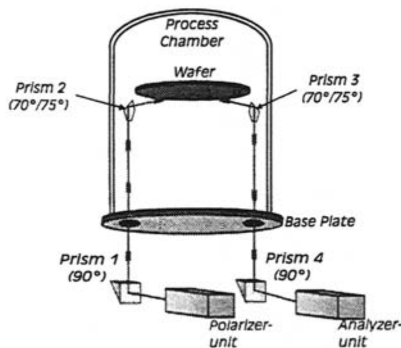

FIGURE 1: The *in situ* ellipsometer arrangement with the beam guiding system (1)

In this setup the calibration of the offsets and angle of incidence or an ellipsometric measurement can be carried out without boat insertion. In addition, measurements of the wafers´ "starting conditions" and *post process* measurements inside as well as outside the tube are possible. No modifications of the process tube and the heating cassette are necessary. In this configuration the wafers are loaded into the boat with the device side down. Conventional loading with the device side up is possible by using two additional beam deflections for the *in situ* metrology. In all measurements, the selected prisms provide an angle-of-incidence of the measuring beam of 75° onto the wafer (1).

As shown in Fig. 1, the ellipsometer beam must be deflected four times. The two reflections of 90° are only necessary if the overall height of the vertical furnace with integrated ellipsometer heads would otherwise exceed the usual cleanroom height. The TIRs lead to a well-defined additional phase shift in the polarization state of the light that can be calculated or measured and subtracted from the measured phase shift according to eq. (1).

$$\Delta=\Delta_m-(2\delta_{p90°}+2\delta_{p75°}) \quad (1)$$

Δ_m is the phase shift as measured with the *in situ* SE setup, Δ is the phase shift caused by reflection on the silicon wafer, $\delta_{p90°}$ and $\delta_{p75°}$ are the phase shifts resulting from the TIRs in the prisms. A measurable change of the amplitude ratio $\tan\Psi$ does not occur. The phase shift at a constant angle of reflection depends only on the index of refraction of the prism material. In practice, the phase shift of the prisms can be detected by measuring a completely transparent sample (quartz glass wafer) in the vertical furnace. Additionally, the angle of incidence can be calculated from the same measurement (sensitive to $\tan\Psi$). The ellipsometer software has been adapted to support measurements with the additional optical components (prisms). With this system the optical material properties of crystalline silicon, silicon dioxide, and silicon nitride as a function of temperature were determined.

In order to enable *in situ* monitoring during the deposition of complex layer structures like polycrystalline silicon, the optical models of this material and the change of the layer structure and composition at deposition condition are presently under investigation. The poly samples were prepared by using LPCVD at a pressure of 250 mTorr and a gas flow of 50 sccm at 560°C. The bulk material was single crystalline (111) oriented, 15-20 Ωcm, p-type CZ-silicon with a thermal oxide of approximately 100 nm thickness.

The spectroscopic ellipsometric monitoring of the recristallization was carried out during the annealing process in the vertical furnace with the beam guiding system as described above. This batch furnace is not designed for rapid heating (max. ramp-rate 20 K/min). Therefore, the insertion

11 December 2024 23:06:22

of the samples into the process chamber with a pure nitrogen atmosphere was performed at 530°C where no changes of the layer structure occure, as test measurements showed. After the stabilization of the temperature, the monitoring started and the furnace was ramped to 600°C. The temperature of the furnace was measured with thermocouples. A special designed fuzzy controller provides an accuracy of less than 0.5 K. It is inevitable that the temperature of the thermocouples may differ from the temperature of the samples. Parallel measurements with a thermocouple wafer were executed to determine the dependence of the thermocouple temperature and sample temperature. With this known dependence it is possible to calculate the real sample temperature out of the course of the profile temperature with an accuracy of better than 2°C.

RESULTS AND DISCUSSION

The capability of the beam guiding system in the vertical furnace was proved by measuring the optical material properties of crystalline silicon, silicon dioxide, and silicon nitride as a function of temperature. These data are now available in the wavelength range between 250 nm and 900 nm. They are already used for growth monitoring and end-point detection during thermal oxidation. Examples of the measured spectroscopic refractive indices of crystalline silicon and silicon dioxide for different temperatures from 450°C to 900°C are shown in Fig. 2 and Fig. 3 (1).

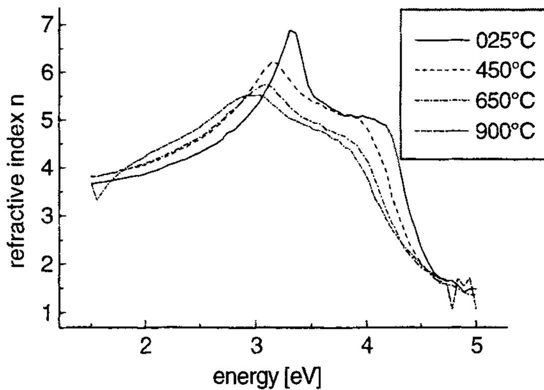

FIGURE 2: Spectra of refractive index of crystalline silicon at different temperatures

These data are used for calculating the dielectric functions of the poly layers during the anneal process. In order to get the initial conditions before the annealing, the layer structure was measured *ex situ* with a SOPRA ES4G spectroscopic ellipsometer (table device). The results are shown in table 1.

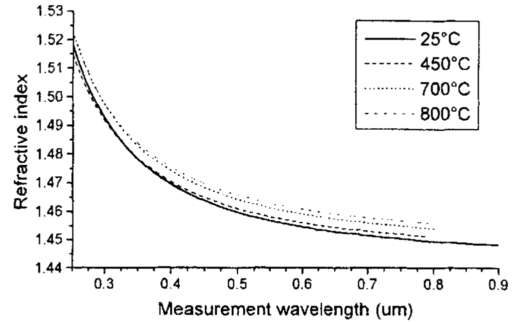

FIGURE 3: Spectra of refractive index of silicon oxide for different temperatures

TABLE 1. Poly-layer structure before annealing (correlation error: σ=0.014)

Layer-number	material 1. comp.	material 2. comp.	conc. of 2. comp. [%]	thickness [nm]
1. Layer	SiO2	--	--	2.0
2. Layer	a-Si	c-Si	5.42	477.9
3. Layer	SiO2	--	--	110.6
Bulk	c-Si	--	--	∞

The evaluation of the measurement was done by fitting theoretical spectra of the ellipsometric angles (tan Ψ and cos Δ) describing the polarization state of the reflected beam to the measured spectra. In this method appropriate models are assumed and the fit is done by varying the wavelength-independent model parameters using Linear Regression Analysis (LRA). The error in the correlation of measured and fitted spectra is given by the unbiased estimator (σ) of the mean square deviation:

$$\sigma=\sqrt{\frac{\sum\left[(\cos\Delta_{j,m}-\cos\Delta_{j,c})^2+(\tan\Psi_{j,m}-\tan\Psi_{j,c})^2\right]}{(2n-p-1)}} \quad (2)$$

where n is number of independent measurement values (corresponding to the different wavelengths), p the number of independent model parameters, m the means measured data, and c means the calculated data (6).

The Bruggeman-Effective Medium Approximation (B-EMA) is used to calculate the dielectric functions of the layers. This method allows the characterization of multi-layer structures (thickness and composition) by one measurement (3)(4). The poly layer is described by a mixture

11 December 2024 23:06:22

of amorphous silicon (a-Si) and single crystalline silicon (c-Si) as shown in Table 1.

Measurements after the annealing process (ES4G, *ex situ*) show that there remains no amorphous part in the sample. The best fit with a correlation error σ better than 0.02 could be obtained by using a mixture of c-Si and a fine-grain polycrystalline silicon (p-Si) without an amorphous component.

An example of a measured spectra (tanΦ and cosΔ) of a polycrystalline sample is given in Fig. 4. The change in the spectra depending on temperature and time is evident. Before a calculation of the dielectric functions is started, the additional phase shift caused by the prisms must be corrected.

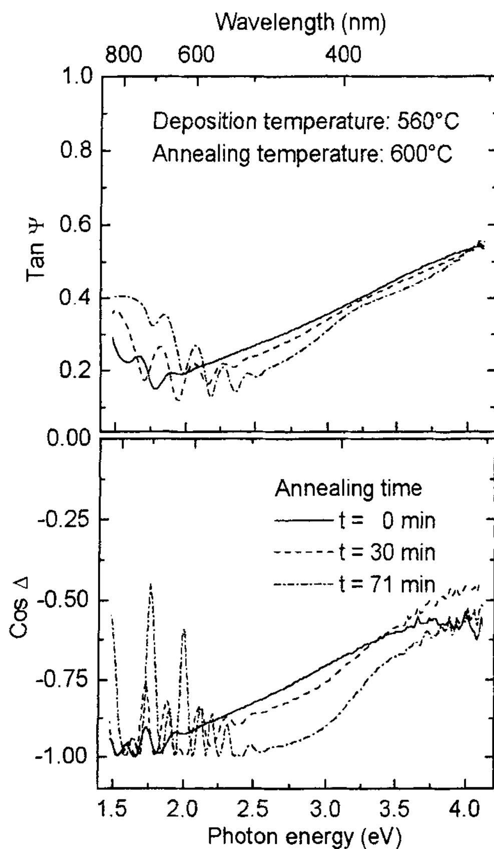

FIGURE 4: Measured spectra at different annealing times

Unfortunately, this correction is only accurate enough in the ultra violet region (2.5 eV to 3.5 eV), because there is no change in the sign of cosΔ for the correction. With thin layers it is possible to determine the points where the sign changes. But in this case there are so many changes in the sign of cosΔ that the error caused by the correction is too high for calculating the dielectric functions. The disadvantage of using only the region from 2.5 eV to 3.5 eV is a much smaller sensitivity to the structural change of the material. To circumvent this problem, in the *in situ* setup an additional compensator must be used to determine the signs of the cosΔ values in the spectrum during the measurement itself. Then, the whole spectral range of the measurement can be used for the calculation of the dielectric functions.

A first calculation of the dielectric functions in the UV-region is shown in Table 2. Here a one-layer model is used, because the polycrystalline layer is not transparent in this region. Therefore, the polycrystalline layer is assumed to be a bulk with a thin oxide layer on its top. The bulk material is assumed to be a mixture of amorphous and fine-grain polycrystalline silicon for the whole annealing time. For the oxide layer, the established data of silicon dioxide at 600°C (SiO2-600) is used. The sample temperature was calculated from the profile temperature and the measured temperature dependence of the profile thermocouple and the thermocouple wafer.

TABLE 2. Recrystallization of the polycrystalline sample

annealing time [min]	sample temp [°C]	a-Si 600°C [%]	p-Si 600°C [%]	SiO2 600°C [nm]	error (σ)
0	525	100	0	1.9	0
11	595	99.9	0.1	2.0	0.003
21	600	93.1	6.9	2.3	0.006
27	600	68	32	3.4	0.007
32	600	51	49	3.4	0.006
34	600	40	60	3.3	0.005
37	600	33	67	3.6	0.005
42	600	16	84	3.3	0.006
47	600	8	92	3.2	0.006
53	600	3	97	3.0	0.005
63	600	-1	101	2.9	0.005
71	600	-4	104	2.6	0.007

The dielectric function of amorphous silicon at 600°C (a-Si-600) is calculated from the first measurement at 530°C assuming that 70°C deviation in the temperature will cause only a small impact on the dielectric function. The data of the polycrystalline silicon is calculated from the last measurement at 600°C after 71 min annealing. To get a information about the reliability of the regression, the thickness of the top oxide was also calculated for each

11 December 2024 23:06:22

measurement.

The results show that the change in the structure of the polycrystalline silicon is very fast (Fig. 5). After 50 min the recrystallization process is finished. The proportion of polycrystalline silicon in the sample is 0% in the beginning and about 95% after 50 min. After this time the sample was for about 35 min at 600°C because approximately 15 min were needed for heating up to 600°C.

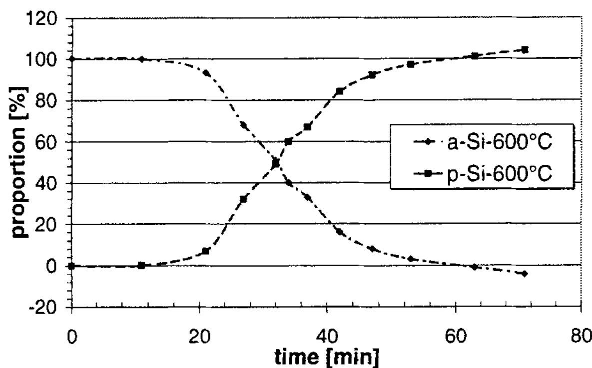

FIGURE 5: Proportion of amorphous and polycrystalline silicon during the annealing process

The quality of the fit is very well during the whole annealing time and the results of the top oxide are reproducable. This means that the model works reliable in this wavelength range (2.5 eV - 3.5 eV). But the error of the proportion values at the end of the annealing process (104% and -4%) and the little variation of the top oxide thickness results indicates that there is a small error in the calculation of the high temperature refractive indices of a-Si-600 and p-SI-600.

CONCLUSION

The capability of the beam guiding system for ellipsometric *in situ* measurement was demonstrated. With the established optical high temperature data of crystalline silicon, silicon dioxide, and silicon nitride the possibility of having *in situ* monitoring of layer growth, layer thickness, and end-point detection in production equipment like vertical batch furnaces was shown. It is very important that these data can be directly measured in the real processing environment, because the optical characteristics of some materials depend on the specific process conditions. The arrangement of the integrated ellipsometer enables *in situ* process control as well as high temperature material research. It could be proved that by the use of the B-EMA to describe the optical material properties the monitoring of layer structure and composition at high temperature is possible. The example of *in situ* recrystallization monitoring of polycrystalline silicon using the beam guiding system reveals a wide variety of applications for which *in situ* spectroscopic ellipsometry can be the key in establishing optical high temperature data. Investigations of the temperature dependence, process time, and changes in material structures can be carried out under real process conditions. Further work has to be done in order to calculate and prove the high temperature dielectric functions of amorphous and polycrystalline silicon in the whole wavelength range from 1.5 eV to 4.5 eV (280 nm to 840 nm). Adaptations of the ellipsometer software to enable measurements with the additional optics (prisms) for *in situ* monitoring of thin oxide and nitride layers are already completed and tested. Automatic measurements of multi-layers or complex layer materials will require the use of a compensator. This will enable automatic correction of the phase shift in the measured signal which is caused by the prisms operating in the total internal reflection mode. Additionally, for layer deposition processes, an especially constructed gas shielding system has been developed to prevent the prisms from coating. Coating of the prisms during a deposition process would cause an continuously increasing error which is difficult to correct. At present the capability of this prism protection system is under investigations. In addition to the established optical high-temperature data this will enable *in situ* layer deposition control during LP-CVD-processing in batch systems.

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11 December 2024 23:06:22