Oxygen precipitates formed in Czochralski (CZ) silicon wafers in consequence of the thermal treatment were investigated since many decades (see e.g. Ref. 1). The main reason why so much effort was directed toward these defects was the impact which they have on integrated circuits and the properties of the silicon wafer itself. Oxygen precipitates can increase the resistivity of CZ silicon, change the wafer strength and cause warpage of the wafers.2–4 It was also demonstrated that metallic impurities can be effectively trapped at oxygen precipitates in the process of gettering.5,6 All these features of the oxygen precipitates require the control of precipitation in the production process of silicon devices. However, this cannot be optimally executed if the features of oxygen precipitates like their composition are not fully known. In spite of the wide knowledge about oxygen precipitation, the composition of oxygen precipitates SiOx still remains under ongoing discussion. This is due to the different x values varying from x = 1 to x = 2 which can be found in the literature.10–20

In 1986, Skiff et al. investigated plate-like precipitates by electron energy loss spectroscopy (EELS) in the near-edge and ionization edge range.10 The composition which they found was SiO0.85. At the beginning of the 90ies, Borghesi et al. modeled Fourier transform infrared spectroscopy (FTIR) spectra by an effective medium approach and concluded that the absorbance band of precipitates at 1230 cm−1 can be well reproduced if SiO1.8 is used.11 However, in 1995 Vanhellemont investigated the growth kinetics of oxygen precipitates based on the solubility and diffusivity of oxygen in silicon using precipitate sizes determined by transmission electron microscopy (TEM). He demonstrated that the precipitated phase is closer to SiO than to SiO2.12 The later works, exploiting again FTIR spectroscopy and effective medium theory for the determination of the composition of the oxygen precipitates and carried out by De Gryse et al., confirmed the thesis of Vanhellemont. In the beginning the authors obtained SiOx with x = 1.1–1.2 but in their more recent work they modified the algorithm and concluded that the precipitated phase behaves optically as a mixture of amorphous silicon and amorphous SiO2.13,14 Meduňa et al. investigated the composition of oxygen precipitates in silicon wafers characterized by different thermal history. Their x value for plate-like precipitates was in the range between 1.8 and 1.9.15 and their method used was again the analysis of FTIR spectra by means of effective medium theory. The next investigation on the stoichiometry of oxygen precipitates was conducted by Nicolai et al.16 In their approach, direct stoichiometry measurements by electron dispersive X-ray spectroscopy (EDX) in a TEM were supported by analyzing density and size of precipitates measured by laser scattering tomography (LST) using Ham’s model. Their truncated octahedral precipitates were supposed to be composed of SiOx.

The large variation of the x values encouraged us to look closer at the precipitate composition issue. First, we analyzed FTIR spectra by effective medium theory in the samples with different morphologies of oxygen precipitates.17 The important parameters for the calculations like the morphology and the size of precipitates were also measured by scanning transmission electron microscopy (STEM). In most cases, we obtained amorphous SiO2 as the phase of the oxygen precipitates. However, FTIR is an indirect method and the analysis is based on many variables. We think that more reliable results can be obtained applying a direct method like EDX and EELS as we did in our previous work.18 The EDX results showed that the composition of plate-like precipitates and transitional forms between plate-like precipitates and octahedral precipitates is close to SiO2. Moreover, the EELS measurements of the characteristic plasmon loss energy show that the oxygen precipitates consist of amorphous SiO2. We found also that plate-like precipitates contain a 2–3 nm thick SiO interfacial layer.8

The knowledge gained within thirty years on the precipitates stoichiometry was reviewed by Vanhellemont.19 He showed that the different values of x which were obtained over the years can be explained by calculating the average composition taking into account the temperature and anneal time dependent size and shape of the precipitates and the limitations and probing volumes of the various characterization techniques. The author also claimed that precipitates are a mixture of amorphous Si and amorphous SiO2. However, we found no indication of amorphous Si in the precipitates.20

In this work, we look on the current stage of the investigation of the composition of oxygen precipitates obtained with the help of different techniques. Moreover, we present our recent and new investigation of the composition of oxygen precipitates carried out by means of energy dispersive X-ray spectroscopy, electron energy loss spectroscopy, and Fourier transform infrared spectroscopy. The FTIR spectra measured at liquid helium temperature are compared with the spectra simulated on the basis of experimental results obtained by scanning transmission electron microscopy. According to the results obtained by all methods the precipitated phase of plate-like as well as octahedral precipitates is close to SiO2.
200 times lower. The EELS spectral resolution was about 1.1 eV. The FTIR investigations were carried out by the FTIR spectrometer Vertex 60v. The samples were measured at liquid helium temperature with a resolution of 0.6 cm\(^{-1}\).

**Simulation of FTIR spectra.**—In order to calculate absorption spectra of oxygen precipitates in silicon wafers, we used the effective medium theory.\(^{21}\) This theory allows calculating the optical properties of a system of two materials, where one of them is buried in the another one. The average dielectric function, which is the result of the calculations, can be used to simulate the absorption spectrum of the two components. In a silicon wafer, the system of the two components consists of the silicon matrix and the buried oxygen precipitates. However, the oxygen precipitates in the silicon crystal adopt well-defined geometric forms like plate-like or octahedral precipitates. These forms impact the position of the absorption bands. To solve this problem, the morphology of the precipitates can be simplified by oblate spheroids with depolarization factors along the three axes \(L\). In case of dendritic precipitates, a prolate spheroid can be used to imitate the branches of the dendrites. The prolate, sphere and oblate spheroids can be defined by the aspect ratio \(\beta\) as it is demonstrated in Fig. 1a.

As shown by Hu,\(^{21}\) the average dielectric function of the Si/SiO\(_2\) system can be expressed by

\[
\varepsilon_{\text{av}} = 1 + \frac{(1 - f) (\varepsilon_m - 1) + f (\varepsilon_p - 1) \left(1 - f\right) + f g}{(1 - f) + f g}
\]

where \(f\) is the volume fraction of precipitates, \(\varepsilon_m\) is the dielectric function of the silicon matrix, \(\varepsilon_p\) is the dielectric function of oxygen precipitates, and \(g\) is the polarizability which by considering three possible orientations of plate-like precipitates can be written in the scalar form as follows

\[
g = \frac{1}{3} (g_1 + g_2 + g_3)
\]

with

\[
g_i = \frac{\varepsilon_m}{L_i \varepsilon_p + (1 - L_i) \varepsilon_m}
\]

Here, \(L_i\) is the depolarization factor (shape parameter) along the \(a_i\) axis of the ellipsoid \(L_1 + L_2 + L_3 = 1\), and approximately \(L_1 = 1/a_1\), \(L_2 = 1/a_2\), \(L_3 = 1/a_3\).

If the oxygen precipitate is coated by a SiO shell as shown in Fig. 1b, the \(\varepsilon_p\) becomes complex \(\varepsilon_p^*\)\(^{22}\) and it is given by

\[
\varepsilon_p^* = \varepsilon_p \left[\frac{2 (1 - f_s) \varepsilon_s + (1 + 2 f_s) \varepsilon_c}{(2 + f_s) \varepsilon_s + (1 - f_s) \varepsilon_c}\right]
\]

where the subscripts \(s\) and \(c\) of \(\varepsilon\) denote the shell and the core of the precipitate, respectively, and \(f_s = [R/(R + d_s)]^2\) is the volume fraction of the core component and \(d_s\) is the thickness of the shell. For oxygen precipitates without a shell \(f_s = 1\) and with a shell \(f_s < 1\).

Finally, the absorption coefficient can be calculated according to

\[
\alpha = \frac{2 \pi v}{\text{Re} (\sqrt{\varepsilon_{\text{av}}})} \cdot \frac{1}{R}
\]

with \(v\) being the wavenumber.

For the calculation of the complex dielectric constant of Si \(\varepsilon_m\), we used the index of refraction \(n = 3.41626 + 1.443 \times 10^{-9} v^3\) from Ref. 23 and the extinction constant \(k = 2.5 \times 10^3 v^{-3.11}\) according to Ref. 21. For the calculation of the complex dielectric constant of oxygen precipitates, we used the optical constants of amorphous SiO\(_2\) from Refs. 24–28 or suboxides for the core and SiO from Ref. 24 for shell of the precipitates. For the calculation of the absorption spectra of oxygen precipitates with different stoichiometry, we extracted the optical constants from the absorption spectra of hydrogenated silicon suboxides from Ref. 29 using the algorithm of Ref. 30.

### Results and Discussion

**EDX investigation.**—In order to be able to investigate the composition of the oxygen precipitates in the silicon matrix by EDX, the precipitates have to be located in a thin region of the TEM lamella. The optimum configuration would be that the precipitate completely passes through the thickness of the investigated sample. In this way, one can be sure that the signal comes only from the precipitate. If

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**Table I. Resistivity, concentration of interstitial oxygen, and thermal treatment of the samples investigated.**

| Sample | Resistivity (\(\Omega\)cm) | \(O_i\) (cm\(^{-3}\)) | RTA | Annealing |
|--------|--------------------------|------------------------|-----|-----------|
| S1     | 0.0085                   | \(5.6 \times 10^{17}\) | no  | 700°C/1 h | 1000°C/11.5 h |
| S2     | 10                       | \(6.5 \times 10^{17}\) | 1250°C 30 s | 900°C 64 h |
| S3     | 40–60                    | \(8.6 \times 10^{17}\) | 1200°C 30 s | 650°C 32 h + 800°C 4 h + 1050°C 3 h + 1100°C 3 h |
| S4     | 40–60                    | \(8.6 \times 10^{17}\) | 1200°C 30 s | 650°C 32 h + 800°C 4 h + 1100°C 2 h |

---

**Figure 1.** Prolate, sphere and oblate spheroid and their aspect ratio \(\beta\), where \(\beta = a_1/a_2 = a_1/a_3\) (a), single shell model of oxygen precipitate (b).
the density of the precipitates decreases, the probability to find a precipitate at a proper location decreases as well.

The quantitative analysis of oxygen precipitates by EDX is difficult due to the limitations of EDX for light elements like oxygen. The light elements are characterized by a strong absorption of the characteristic X-rays by the specimen. Moreover, their electrons being responsible for the generation of X-rays are involved in chemical bonding which leads to a line shift and a change of the line shape. Therefore, the measurement of the oxygen concentration in the precipitates requires a calibration sample. Therefore, we used a homogenous thermal SiO$_2$ layer of 130 nm thickness as the standard. The calibration sample was measured with the same parameters as the investigated precipitates. The measurements were done at different thicknesses of the TEM foil. This allowed us to determine the thickness dependence of the sensitivity factor. The thickness of the samples was measured by the EELS log-ratio method. Moreover, the thickness measurement by the EELS method was confirmed by measuring the sample thickness in the vicinity of a stacking fault (SF) passing through the whole sample thickness. Based on the well-known crystallographic orientation of SFs in silicon, and by measuring the length of the projection of the SF on the (110) surface along the [100] direction the exact thickness of the sample was calculated.

The ratio between silicon and oxygen atoms in the oxygen precipitates was determined according to the Cliff-Lorimer method. According to this method, the concentration can be calculated from the equations:

$$\frac{C_A}{C_B} = k_{AB} \frac{I_A}{I_B} \quad \text{and} \quad C_A + C_B = 1$$  \[6\]

where $C_A$ and $C_B$ are the weight fractions of two elements, $I_A$ and $I_B$ are the measured characteristic X-ray intensities and $k_{AB}$ is the sensitivity factor.

Figures 2a and 2b show a bright field (BF) STEM image and an EDX map of an oxygen precipitate in the S1 sample, respectively. Due to the small thickness of the foil (37 nm at the analyzed location), the observed precipitate is partially cut. This hinders the exact identification of the morphology of the precipitate. However, the morphologies which can be considered in this case are plate-like, octahedral or an intermediate form between these two mentioned forms because all these morphologies were found in this sample. Across the oxygen precipitate an EDX line scan was measured as indicated in Fig. 2a. As a reference we investigated the SiO$_2$ layer with a lamella thickness of 34 nm which is shown in Fig. 2c. In the plot in Fig. 2d, the concentration of oxygen and silicon atoms in the oxygen precipitates is compared with both concentrations in the oxide layer. It can be clearly seen that the concentrations of silicon and oxygen atoms in the investigated precipitate are similar to those in the SiO$_2$ layer.

A long nucleation anneal at 650°C was used to form a high density of precipitates in the range of about $4 \times 10^{12} \text{ cm}^{-3}$ in the S3 and S4 samples. A stabilization step at 800°C enabled the oxygen precipitates to survive at the high temperature of the growth step which is crucial to form different morphologies. The density of precipitates was high enough to find plate-like precipitates and octahedral precipitates in very thin locations of the TEM lamellae. Two examples of precipitates very close to the edge of the lamellae are shown in Fig. 3. Because the oxygen atoms are lighter than the silicon atoms more electrons can get through the precipitate to the bright field detector (BF) and the signal in the precipitate is more intense or bright compared to the dark silicon background.

Figure 3a shows a BF image of a thick plate-like precipitate observed in Sample S3 and an octahedral precipitate in sample S4 (Fig. 3b). The EDX map in Fig. 4 shows the distribution of the oxygen in the precipitates. The composition of the precipitates was determined in the center of the precipitates as shown in Fig. 4b. The concentration of oxygen and silicon was calculated measuring the intensity of the
characteristic X-rays in the EDX spectrum according to Equations 6. The thickness of the TEM lamella of the investigated precipitates was always below 40 nm. In case of plate-like precipitates, the ratio between Si and O was in the range between 1.9 and 2 with sigma 3 error of 0.4 for most of the investigated precipitates. The measurement of the octahedral precipitates is much more difficult because of the small size of the octahedral precipitates. Another problem of octahedrons is their geometric form limiting the area suitable for the measurements to the center of precipitate. In case of octahedral precipitates, measured $x$ values were in the range between 1.34 and 1.9. The lower $x$ value is mainly due to the remaining Si layer above or below the precipitate increasing the Si signal. Therefore, when measuring the composition of oxygen precipitates we should consider the maximal values only.

**EELS investigation.**—Plate-like and octahedral oxygen precipitates were also investigated by EELS. EELS is a complementary method for EDX.\(^\text{34}\) The problems which appeared by measuring the composition of oxygen precipitates in the TEM lamellae can be avoided. EEL spectra are characteristic for the various phases. Investigating the composition of oxygen precipitates, even if the precipitate is partially coated by a silicon rest, we can still separate the Si phase from the phase of the precipitate, what cannot be done for EDX. However,
the sample should still be relatively thin to avoid plural scattering and to keep a good signal to background ratio.

In Ref. 10, the authors analyzed EEL spectra of precipitates in the ionization range. However, the low-loss range can be more efficient. The lower beam current does not influence the investigated area and the signal is stronger.

In this work, we analyzed the plasmon peak in the low-loss range of the EEL spectra. In Refs. 35, 36, the authors demonstrate the dependence of the stoichiometry of an amorphous oxide layer on the maximum position of the plasmon peaks. The characteristic Si plasmon peak is located at the energy loss of 16.7 eV.37 This can be observed for the low-loss spectra taken in the vicinity of the oxygen precipitate as shown in Fig. 5 (location 1). The material change from Si to SiO₂ leads to a plasmon energy shift to about 23 eV.37,38 The spectra taken on the plate-like precipitates (location 2) show the characteristic form of amorphous SiO₂ layers with the plasmon loss energy at 23 eV.

The typical EEL spectrum of amorphous SiO₂ can be observed in the center of the octahedral precipitate as it is shown in Fig. 6. At the top of the octahedron the Si plasmon becomes much more significant but still the shoulder of the SiO₂ plasmon can be seen.

By means of EEL spectra using the deconvolution method with reference spectra of different pure phases, we analyzed the interface between the silicon matrix and oxygen precipitates.

**FTIR investigation.**—In order to see absorption bands of oxygen precipitates, a marked amount of interstitial oxygen atoms should precipitate. According to Borghesi et al., the difference of the O_i concentration between as-grown and annealed samples should be at least $1.16 \times 10^{17} \text{ cm}^{-3}$.40 This means that we need a high density of small precipitates or a lower density of large precipitates. Because the absorption bands of interstitial oxygen and precipitates at room temperature are overlapping, the FTIR measurement should be carried out at liquid helium temperature. Another limitation for the FTIR measurement is the dopant concentration of the sample. If it is too high, IR light will be absorbed by free carriers.

The absorption bands of oxygen precipitates measured at liquid helium are broad and less intensive compared to the highest O_i band at 1106 cm⁻¹. They are located in the vicinity of the O_i band. According to literature the band located at about 1230 cm⁻¹ belongs to plate-like precipitates and the band at about 1090 cm⁻¹ is originated by spherical precipitates.11,12,14–21 In fact, this can be very well explained based on the effective medium theory as it is shown in many works.11,13–15,17,21

The simulated absorption bands of the oxygen precipitates show a strong dependence on the morphology and stoichiometry of oxygen precipitates as it is shown in Fig. 7. For thin plate-like precipitates (oblate), the absorption band is shifted toward higher wavenumbers compared to the bands of a sphere and prolate spheroids as can be clearly seen on the example of SiO₂ in Figs. 7a, 7b and 7c. The bands of the sphere and prolate spheroids do not differ much between each other. The stoichiometry of the precipitates has a strong influence on the band position, especially for plate-like precipitates. In suboxides with decreasing content of oxygen, the band position moves toward lower wavenumbers as it is shown in Fig. 7a.

As we learned from Ref. 8, amorphous SiO₂ oxygen precipitates are surrounded by a 2–3 nm suboxide shell. Such a suboxide region could also influence the absorption band of oxygen precipitates. Fig. 8 demonstrates the impact of the volume fraction of the core of the precipitates $f_c$ on the absorption band. If the shell becomes thicker or the core becomes smaller, the absorption band is more affected by the dielectric function of the shell. In this case, the absorption band...
moves from higher wave numbers being characteristic for an SiO$_2$ phase toward lower wavenumbers. Moreover, the absorption bands become broader, as they are typical for an SiO phase.

The samples investigated in this work exhibit clear absorption bands of oxygen precipitates as shown in Figs. 9a, 9b, and 9c. The differences between the shapes of the spectra are caused by different forms of oxygen precipitates. The precipitates observed in the samples are presented in Fig. 9 on the right hand side. In order to calculate the spectra, we used spheroids if possible with similar aspect ratios to the oxygen precipitates being characteristic for the measured samples. The dielectric functions of the different phases and volumes fractions $f$ and $f_c$ were the only variables of the calculation. All the parameters used to calculate absorption bands are included in Table II.

In the sample S2, the dominating morphology of the precipitates is plate-like but octahedral precipitates also appear as shown in Fig. 9a1. The absorption spectra of this sample can be fitted by four bands as shown in Fig. 9a. The bands 1, 3 and 4 simulate plate-like precipitates with different thickness while the band 2 corresponds to octahedral precipitates. It should be mentioned, that the oxide phase is of great importance. The best fit is obtained only with SiO$_2$ especially for thin plate-like precipitates.

Although the absorption bands located at about 1090 cm$^{-1}$ are mainly associated with spherical precipitates, an example of sample S3 proves that a precipitate band located at this side of the highest O$_i$ band is strongly affected by thick plate-like precipitates. The absorption spectrum of the sample can be fitted by thick oblate spheroids. The reason why the fit is not perfect can be explained by the variety of forms of the precipitates in this sample. Besides many regular plate-like precipitates as can be seen in Fig. 4a and Fig. 9b3, other forms are also present (Figs. 9b1, 9b2). This and the fact that the edges of plate-like precipitates are sharp and confined by (111) planes could explain the imperfection of the fit obtained by using the spheroid simplification. Nevertheless, the bands of thick plate-like precipitates clearly contribute to the experimental bands at about 1090 cm$^{-1}$. Moreover, the composition of the oxygen precipitates is SiO$_2$ or close to SiO$_2$. The contribution of thin plate-like precipitates to the spectra is almost negligible.

In the sample S4, the main defects are perfect octahedral precipitates as shown in Figs. 4c and 6a. There is also one broad band in the absorption spectrum located at about 1100 cm$^{-1}$. This band can be fitted by prolate spheroids with an aspect ratio which is close to the ratio between the height and the length of an octahedron. In order to get a better fit, we used an additional band of thick plate-like precipitates. The usage of this band can be explained by the presence of octahedral precipitates located in the center of stacking faults. Such precipitates grow along the stacking fault (111) plane forming quasi plate-like defects as shown in Fig. 9c1–9c4. In this case, to fit the bands the dielectric function of amorphous SiO$_2$ was used.

Figure 8. Simulated absorption spectra of spheres (a), thick oblate spheroids (b), thin oblate spheroids (c) with different volume fraction between a SiO$_2$ core and a SiO$_2$ shell and without shell $f_c = 1$.
As we could see in this section, the composition of the oxygen precipitates in silicon can be determined by deconvolution of absorption spectra. The change of the stoichiometry of the precipitates influences the shape and position of the absorption band. However, for the presented examples we were not able to see significant changes caused by the change of the composition of oxygen precipitates. The consideration of a SiO shell, although reasonable, just leads to a cosmetic improvement of the fits. The main influence on the absorption band position and shape is due to the change of the precipitate morphology. The whole process for determination of the stoichiometry or the morphology of oxygen precipitates by this method is complicated because of the fact that at least one of the unknowns should be known before the deconvolution.

Table II. Parameters used to fit experimental spectra.

| Sample | Band no. | $\beta$  | $f$    | $f_c$ | $x$  | Ref. |
|--------|----------|---------|--------|-------|------|------|
| S2     | 1        | 0.011   | $9.2 \times 10^{-7}$ | 1     | 2    | 26   |
|        | 2        | 1       | $2.1 \times 10^{-6}$ | 1     | 2    | 25   |
|        | 3        | 0.28    | $2.1 \times 10^{-6}$ | 1     | 2    | 25   |
|        | 4        | 0.1     | $3 \times 10^{-7}$   | 1     | 2    | 25   |
| S3     | 1        | 0.026   | $5 \times 10^{-7}$   | 0.9   | 2    | 26   |
|        | 2        | 0.35    | $2 \times 10^{-5}$   | 0.9   | 2    | 26   |
|        | 3        | 0.11    | $2 \times 10^{-6}$   | 0.9   | 1.86 | 29   |
| S4     | 1        | 0.25    | $6.4 \times 10^{-6}$ | 0.9   | 2    | 26   |
|        | 2        | 1.4     | $6.4 \times 10^{-6}$ | 0.95  | 2    | 26   |
In this work we presented an overview of the methods which can be used for the determination of the composition of oxygen precipitates. The direct methods like EDX and EELS available during TEM investigation and the deconvolution of FTIR spectra as an indirect method were summarized.

EDX gives good results but the investigated precipitate should be significantly large to completely penetrate the TEM lamella. If the precipitate is too small, the signal from the residual silicon coating the precipitate affects the results by decreasing the x value. This method requires determination of the depth dependent sensitivity factor for the Si/O system.

EELS allows avoiding the problem of residual silicon which disturbs the EDX method. However, quantitative evaluation requires reference spectra of the pure materials. The TEM lamella should not be too thick.

The absorption bands observed by FTIR can be well fitted by varying parameters attributed to the composition of oxygen precipitates and their morphology. However, it is difficult to obtain reliable results without the support of TEM results.

In spite of some imperfection of the methods discussed, the measured phase of oxygen precipitates, plate-like as well as octahedral, was amorphous SiO$_2$. Detailed EELS measurements have shown that the precipitates are surrounded by a 2–3 nm suboxide region which was also considered in the calculation of the absorption spectra.

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