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Oxygen diffusivity in silicon derived from dynamical X-ray diffraction

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Thickness dependent Pendellösung oscillations are highly sensitive to strain fields from defects in a host crystal. Based on this, we present a novel technique to measure the precipitation kinetics of oxygen in silicon already at its early stage of clustering at high temperatures. At 900 °C, precipitates with a radius smaller than 4 nm and with a density of $1 \pm 0.5 \times 10^{13} \text{ 1/cm}^3$ were observed. The technique was calibrated by complementary scanning transmission electron microscope and energy dispersive X-ray measurements in the range of normal diffusivity yielding a diffusion constant of $1.7 \pm 0.1 \times 10^{-12} \text{ cm}^2/\text{s}$, which is close to the literature value of $2.074 \times 10^{-12} \text{ cm}^2/\text{s}$. The measurements have been made with the characteristic $K_{\alpha 1}$ -line of a high voltage tungsten X-ray tube at 59.31 keV, which provides the opportunity to illuminate through complex sample environments like high temperature scattering furnaces. © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4792747>]

I. INTRODUCTION

Silicon crystals grown by the Czochralski method typically have an atomic oxygen concentration in the order of 10^{17} and up to 10^{18} 1/cm^3 , which is a consequence of the dissolution of the quartz crucible during crystal pulling and the high oxygen solubility of $2.5 \times 10^{18} \text{ 1/cm}^3$ at the melting point. Usual temperatures in the production line of integrated circuits are in the range of several hundred degrees and thus way below the melting temperature of silicon. At these temperatures, the solubility of oxygen is lower than the incorporated oxygen content and the dissolved oxygen represents thus a supersaturated solution. This supersaturation is the driving force for the formation of SiO_x precipitates in the silicon lattice, which gives rise to both beneficial and detrimental effects. For example the internal gettering process is a most important beneficial effect in contrary to their role as recombination centers for charge carriers as an undesired property. In order to control the density and size of these precipitates it is important to have a deep understanding of the kinetics of the agglomeration process, which is controlled by the diffusivity of oxygen in silicon at a given temperature. For low dopant concentrations $< 10^{-16} \text{ 1/cm}^3$ and in the temperature range between 700 and 1200 °C the diffusivity is well understood and given by (Ref. 1)

$$D = 0.13 \exp\left(-\frac{2.53 \text{ eV}}{kT}\right) \text{ cm}^2 \text{ s}^{-1}. \quad (1)$$

However in the temperature range below 700 °C^{2–5} as well as for highly doped materials^{6–8} the diffusivity has been found to be different from the values extrapolated with Eq. (1).

High energy X-ray diffraction as available at the Chair of Crystallography and Structural Physics at the University of Erlangen-Nürnberg⁹ provides a unique opportunity to access the precipitation process in a wide temperature range in-situ. In this paper, we show that the combination of in-situ diffraction data of thickness-depended Pendellösung oscillations with scanning transmission electron microscope (STEM) measurements gives a reliable diffusivity constant in the regime of normal diffusion at 900 °C where Eq. (1) is valid. From this starting point further investigations at lower temperatures as well as for highly doped materials can be envisaged.

II. EXPERIMENTAL METHODS

A. STEM

In order to investigate the morphology and density of oxygen precipitates a STEM FEI Tecnai Osiris equipped with energy dispersive X-ray (EDX) detectors was used. The bulk micro defect (BMD) density was determined by the investigation of $200 \pm 100 \text{ nm}$ thick films with an area of $4 \mu\text{m}^2$. The average size and thickness of the precipitates were determined based on several STEM bright field (BF) images. The morphology and the presence of oxygen in the defects were confirmed by EDX images.

B. Pendellösung oscillations

The measurement of the integrated Bragg intensity as a function of the sample thickness is a sensitive method to detect small strain fields in nearly perfect single crystals as shown in previous publications.^{10–13} The thickness dependent Bragg intensity of an ideal crystal can be described analytically¹⁴ and shows a characteristic oscillatory behavior around the average Bragg intensity. The oscillation period is called the Pendellösung length, and for X-rays it is typically

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in the order of $100\ \mu\text{m}$. The origin of the Pendellösung oscillations is in the interference of standing wave fields in the strictly periodic lattice of the ideal crystal. Anything causing a deviation from an ideal crystal periodicity like thermal vibrations or static displacements will lead to shifted electron positions and as a consequence these will no longer or only partially take part in the formation of the standing wave fields. This can be considered as an effective reduction of the interaction strength between the X-ray wave field with the electric susceptibility resulting in an elongation of the Pendellösung length.

During annealing processes the supersaturated oxygen solution in silicon becomes mobile and starts to agglomerate. These SiO_x precipitates build up strain fields, which disturb the perfect silicon host lattice. Because of the high density of the agglomerates, this can be described in a statistical approach within the statistical dynamical theory^{15–18} which predicts as the most prominent feature an incoherent increment of the average Bragg intensity originating from the enhanced phase space becoming available for Bragg reflection. In addition, the Pendellösung length becomes elongated and the amplitude of the oscillations appears damped.

C. Experimental

A silicon wedge with an opening angle of 2.44° was cut from a massive Czochralski-grown silicon boule. The sawing damage was removed by a polishing step and subsequent etching in a mixture of nitric and hydrofluoric acid. The wedge angle was determined by an X-ray absorption experiment with characteristic molybdenum $K_{\alpha 1}$ -radiation. The wedge was illuminated in the high energy X-ray laboratory of the Chair of Crystallography and Structural Physics of the University of Erlangen-Nürnberg with a divergent X-ray beam from a high voltage (225 kV) tungsten tube. The in-plane and out-of-plane divergences were limited by slits to 0.02° and 1° , respectively. This setup including the resolution of the Pendellösung oscillations is described in greater detail in Ref. 11. The small divergence in the plane of diffraction guarantees that the sample only accepts the characteristic $K_{\alpha 1}$ -line of the tungsten X-ray tube at 59.3 keV. The Bragg angle for the 400-reflection is 4.4152° , and the acceptance of the $K_{\alpha 1}$ -line was checked in a defocused setup. The characteristic $K_{\alpha 1}$ and $K_{\alpha 2}$ -lines appear at the image plate detector as intense lines and can be separated (see Figure 1). With the sample positioned 3 m from the X-ray tube, the out-of-plane divergence was chosen to illuminate the full sample height of 5 cm. This allows measuring the thickness dependence of the reflected intensity of the wedge with one exposure only. With the high energy of the tungsten X-ray tube it is possible to illuminate through complex sample environments like vacuum scattering furnaces. An exposure time of 10 min was sufficient to get good statistics while maintaining an appropriate time resolution for the in-situ measurements.

To avoid any contamination during the annealing process, the sample was placed in a vacuum furnace with a pressure of 1×10^{-6} mbar. After a nucleation step at 650°C for 141 h, the sample was heated with 3 K/min to 900°C .

FIG. 1. Detector image of a Laue exposure of the 400-reflection of the silicon wedge for a tube to sample distance of 3 m and a sample to detector distance of 1.5 m (defocused setup); the two dark lines in the Bragg reflection correspond to the characteristic tungsten $K_{\alpha 1}$ and $K_{\alpha 2}$ -lines. The illuminated sample volume and thus the divergence were afterwards decreased by a slit system in order to receive signal only from the $K_{\alpha 1}$ -line corresponding to an energy of 59.3 keV.

In this temperature range and for a low boron doping of 3×10^{15} atoms/cm³ ($8.5\ \Omega\ \text{cm}$) normal diffusion of oxygen in silicon is expected.¹ The annealing time at 900°C was 19.5 h, and the oxygen concentration in the sample amounted to 6×10^{17} atoms/cm³.

In addition, the effect of thermal vibrations on the Pendellösung length at 900°C was measured with a wedge cut from a Float zone crystal. The oxygen concentration in this material is below the solubility limit and thus no agglomeration occurs. Thus, no static displacements are expected to build up. This allows to separate between thermal and static effects in the measurement.

III. RESULTS

After annealing, the sample was investigated by STEM. STEM bright field, and EDX images are shown in

FIG. 2. STEM BF image of a plate-like oxygen precipitate in the (001) view observed in a sample annealed at 650°C for 141 h followed by an anneal at 900°C for 20 h. Note that the size of precipitate corresponds to its diagonal length.

FIG. 3. EDX images of plate-like oxygen precipitates in the (001) view observed in the sample annealed at 650 °C for 141 h followed by an anneal at 900 °C for 20 h; image of a precipitate oriented perpendicular to the plane (left) (same as in Fig. 1), and image of a precipitate oriented parallel to the plane (right).

Figures 2 and 3, respectively. The plate-like precipitates lying on {100}-planes have an average side length of 32 nm and a thickness of 3 nm, which corresponds to an aspect ratio of 0.094. The precipitate density was determined by STEM to $1 \pm 0.5 \times 10^{13} \text{ cm}^{-3}$. The EDX images of the distribution of oxygen and silicon atoms for two plate-like precipitates one oriented in-plane and the other one perpendicular to the surface shown in Fig. 3 confirm their chemical nature of a silicon oxide. The size and density values extracted for the STEM results are used to calibrate the static Debye-Waller factor measured with the Pendellösung oscillation technique.

Figure 4 shows some exemplary in-situ data during the annealing at 900 °C compared with the ideal crystal behavior at 900 °C (dashed line). The ideal crystal behavior corresponds to a Pendellösung length of 184.2 μm measured with the Float zone wedge. The increment of the average Bragg intensity with annealing time is readily appreciated. The measurement at the beginning of the annealing already shows small deviations from the ideal crystal behavior, and these deviations grow rapidly bigger with progressing annealing. This implies that the precipitation is visible from the beginning and during the entire annealing time of 19.5 h.

A comparison of the measurement after 10 h with the measurement at the beginning and the end of the

FIG. 4. Evolution of the average Bragg intensity with the annealing time at 900 °C. The black dotted line corresponds to the calculated ideal crystal behavior; from light grey to black 0 h, 1 h, 2 h, 4 h, 10 h, and 19.5 h, respectively.

experiment shows, that the evolution of the level of the average Bragg intensity is more pronounced within the first 10 h as compared to the later 9.5 h. This clearly shows a saturation of the precipitation process towards the end of annealing.

From the data taken after 4 h it can already be suspected, that the progression of the average Bragg intensity for a thickness value around 600 μm is not homogenous. This effect becomes more pronounced with annealing time and it is clearly visible in the measurements after 10 h and 19.5 h providing evidence for an inhomogeneous nucleation or precipitation originating for example from oxygen striations. However, these local variations have only little influence on the further data analysis.

IV. ANALYSIS OF DIFFRACTION DATA IN TERMS OF OXYGEN PRECIPITATION

In the subsequent section, the Pendellösung data are first evaluated within the statistical dynamical theory, which allows for a determination of the static Debye-Waller factor E with the precision of 0.1‰. This is followed by the calibration of the E value at the end of the annealing process with the STEM. This allows evaluating the misfit parameter ε within a description by spherical agglomerates.¹⁹ On these grounds, the corresponding equivalent radius of spheres during the whole precipitation process is calculated within a diffusion limited model of a growing spherical precipitate.²⁰ As a final step, the diffusion constant at 900 °C is extracted and compared with the normal diffusion constant after Mikkelsen.

A. Evaluation of the static Debye-Waller factor within the statistical dynamical theory

Figure 5 shows the theoretical curves of Pendellösung oscillations corresponding to the statistical dynamical theory compared with some exemplary measurements at the beginning, after 2 h and the end of the annealing process. The value of the static Debye-Waller factor changes from 0.9995 to 0.977 within these 19.5 h. Whereas the theory describes the data perfectly for the first 5 h the inhomogeneous strain

FIG. 5. Comparison of the experimental data with the statistical dynamical theory for the measurement at the beginning of the annealing time (light grey), for an annealing of 2 h (dark grey) and at the end of the annealing at 19.5 h (black).

TABLE I. Values of the static Debye-Waller factor E as a function of the annealing time.

Time (min)	E
5	0.9995
35	0.9983
65	0.9965
95	0.9946
125	0.9931
185	0.9905
245	0.9880
305	0.9865
425	0.984
605	0.980
965	0.979
1175	0.977

in the thickness range of $600\ \mu\text{m}$ causes some deviations between theory and experiment at later times.

B. Time resolved determination of the equivalent radius of the precipitates

The determined values of E (see Table I) are now evaluated within the theory of a spherical precipitate.¹⁹ This description is applied although it is known from the STEM measurements (Figs. 2 and 3) that the precipitates have a platelike shape at the end of the annealing. The static Debye-Waller exponent L_H within this model is a function of the equivalent precipitate radius $R_{\text{prec}}^{\text{eq}}$, the density c_{prec} , and a volume misfit parameter of the single precipitate ε , which is adjusted to the TEM data and thus compensates for the platelike morphology. Thus, spheres with the same volume as the platelets found in the STEM measurement can be assumed even for the measurement at the end of the annealing time.

$$R_{\text{prec}}^{\text{eq}} = \sqrt[3]{\frac{3}{4\pi} V_{\text{prec}}^{\text{TEM}}}. \quad (2)$$

This leads to an equivalent radius of $90.2\ \text{\AA}$ for a sphere for the determined E value of 0.977. The relation between E and the equivalent radius is given by

$$1 - E = 1 - \exp(-L_H^{\text{prec}}) \cong L_H^{\text{prec}}(R_{\text{prec}}^{\text{eq}}; c_{\text{prec}}; \varepsilon). \quad (3)$$

Hereby L_H^{prec} for spherical precipitates takes the form

$$L_H = \frac{c_{\text{prec}} V_{\text{prec}}}{c_{\text{Si}} V_0} \left(1 + 3 \left[\frac{\cos(A)}{A^2} - \frac{\sin(A)}{A^3} \right] + 3 \int_1^\infty \left[1 - \frac{x^2}{A} \sin\left(\frac{A}{x^2}\right) \right] x^2 dx \right). \quad (4)$$

In Eq. (4) V_{prec} is the volume of the spherical precipitate, c_{Si} is the silicon lattice site density of $5 \times 10^{22}\ \text{cm}^{-3}$, V_0 is the unit cell volume of silicon of $160.1\ \text{\AA}^3$, A equals $\varepsilon H R_{\text{prec}}^{\text{eq}}$, with H being the absolute value of the reciprocal lattice vector and x represents $r/R_{\text{prec}}^{\text{eq}}$.

The first three terms in Eq. (4) arise from the precipitate itself and are in most cases orders of magnitudes smaller than the integral term, which arises from the lattice displacement of the silicon atoms surrounding a BMD.

By adjusting the equivalent radius and precipitate density derived from the STEM data to the measured static Debye-Waller exponent, the value of ε was determined to 0.193. We expect the value of ε being somewhat higher for spherical precipitates of same volume, because the higher elastic constant of silicon in $\langle 100 \rangle$ - as compared to the $\langle 110 \rangle$ -direction. The ε -value was kept constant as well as the precipitate density in order to calculate the equivalent radius for all other measured E values. The result is displayed in Table II.

C. Determination of the diffusion constant at 900°C within Ham's theory

The determined values for the radius of the precipitates can be evaluated within the diffusion limited model of a supersaturated solution after Ham's theory.²⁰ Within this model the silicon lattice is assumed to be free of defects with the exception of the precipitates. In addition, the initial oxygen concentration in the as-grown sample should be uniform, and the oxygen concentration as well as the density of the precipitates remains constant during the annealing. Further, the precipitates form monodisperse spheres with a radius much smaller than the distance to neighboring precipitates, and the oxygen concentration in the precipitate is uniform and given as c_p . In this experiment, there were indications for a non-uniform oxygen concentration (see Fig. 4 at a sample thickness around $600\ \mu\text{m}$). Nevertheless, this effect is small compared to the overall enhancement of the average Bragg intensity and thus has no relevant influence on the data evaluation. The restriction to monodisperse spheres however is a more crude assumption. In spite of this it was already used during the calculation of the radius from the static Debye-Waller factor, and its influence on the determination of the volume misfit parameter is discussed in Sec. V. The oxygen concentration in the precipitate is taken from literature to $2.9 \times 10^{22}\ \text{1/cm}^3$ (Ref. 21).

TABLE II. Equivalent radius of the precipitates for different annealing times at 900°C .

Time (min)	$R\ (\text{\AA})$
5	38.5
35	50.5
65	59.3
95	65.3
125	69.0
185	74.1
245	78.1
305	80.1
425	83.2
605	87.5
965	88.5
1175	90.2

Figure 6 shows the evolution of the square of the equivalent radius against the annealing time at 900 °C. The displayed dotted curve is a fit calculated within Ham's model according to

$$R_{\text{prec}}^2(t) = 2\tau D \left(\frac{c_0 - c'}{c_p - c'} \right) \left[1 - \exp\left(-\frac{t - t_0}{\tau}\right) \right]; \quad (5)$$

$$\tau = \left(3Dc_{\text{prec}}^2 \right)^{-1} \left(\frac{c_p - c'}{c_0 - c'} \right)^{\frac{1}{3}}.$$

In above equation τ is the time constant on which the precipitation process takes place. It takes into account that the oxygen is partially concentrated in the precipitate and not available in the supersaturated solution, D is the diffusion constant at 900 °C, c_0 is the initial interstitial oxygen concentration of 6×10^{17} atoms/cm³, and c' the solubility at 900 °C of 2.65×10^{16} atoms/cm³ (Ref. 22). Moreover, t_0 is an offset time, which takes into account the fact that some precipitation has already occurred during the 3 K/min ramp from 650 °C to 900 °C.

During the fitting procedure the values of τ , D , and t_0 were determined, respectively. Whereas t_0 is a largely independent parameter with a fit value of 40.6 ± 6.6 min, the values of τ and D are more strongly correlated. For the best fit the density of the precipitates c_{prec} is chosen as a free parameter and afterwards compared to the STEM value. Within this procedure the diffusion constant is determined to $D(900\text{ °C}) = 1.7 \pm 0.1 \times 10^{-12}$ cm²/s, which is close to the Mikkelsen value of 2.074×10^{-12} cm²/s. The value for the fit density of the precipitates is 1.59×10^{13} 1/cm³ and thus 50% higher than the STEM density, which is a satisfactory agreement considering the error of STEM determination as well as the assumptions made during the evaluation of the diffraction data.

In order to adjust the equivalent radius and measured static Debye-Waller exponent at the end of the annealing time, we used the density as determined from a fit with Eq. (5). The value of ε is then determined to 0.153. A further recursive data treatment of the measured static Debye-Waller factors with the new ε value and Eq. (4) leads to radii which are again fitted by Eq. (5). This fit leads to a diffusion constant of $1.68 \pm 0.1 \times 10^{-12}$ cm²/s and a precipitate density

of 1.59×10^{13} 1/cm³. Accordingly no further recursive data treatment is needed.

V. CONCLUSION

The comparison of the determined experimental diffusion constant $1.7 \pm 0.1 \times 10^{-12}$ cm²/s with the literature value at 900 °C 2.074×10^{-12} cm²/s demonstrates that the presented experimental technique and the model for the data evaluation are valid. Within this approach the static Debye-Waller factor can be determined from in-situ experiments with a high accuracy of 0.1‰. This extraordinary sensitivity to the strain fields in crystal allows observing the precipitation process already at an early stage. In the present case we could detect precipitates with an equivalent radius of a sphere of less than 4 nm at a TEM density of $1 \pm 0.5 \times 10^{13}$ 1/cm³.

The model of spheres describes the generation of the strain field of the precipitates reasonably well. However, the strain fields may also depend to some extent on the morphology of precipitates (like octahedron or platelet), and also point defects like vacancies, interstitials or dopants may contribute to the strain inventory. Because of this the value of ε has to be confirmed for each measurement with ex-situ STEM measurements.

The high sensitivity of the Pendellösung oscillations to the strain fields as well as the positioning of the determined diffusion constant in the regime of normal diffusion provides a sound starting point for further investigations in the low temperature regime, where enhanced diffusivity may occur. This regime is only in part understood and there is a great uncertainty for the temperature dependence of the diffusion constant. In addition to providing a new access for a determination of the low temperature oxygen diffusion in Si, the present technique also holds promise to study the influence of dopants in the low and high temperature range.

In conclusion, we have established a novel technique combining in-situ measurements of thickness dependent Pendellösung oscillations with one ex-situ STEM measurement to determine reliably the diffusivity constants of oxygen in Czochralski silicon over a wide temperature range. The applicability to other point defects in a host matrix of high structural quality will be evaluated.

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FIG. 6. Fit of the square of the precipitate radius against annealing time at 900 °C within Ham's theory of precipitation.

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