Characterization of \{111\} planar defects induced in silicon by hydrogen plasma treatments

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Microstructural characterization by transmission electron microscopy of the \{111\} planar defects induced in Si by treatment in hydrogen plasma is discussed. The \{111\} defects are analyzed by conventional (TEM) and high-resolution transmission electron microscopy (HRTEM). Quantitative image processing by the geometrical phase method is applied to the experimental high-resolution image of an edge-on oriented \{111\} defect to measure the local displacements and strain field around it. Using these data, a structural model of the defect is derived. The validity of the structural model is checked by high-resolution image simulation and comparison with experimental images.

1. Introduction

Over the years, silicon hydrogenation has been intensively studied as an important step in silicon technology. The role played by hydrogen atom insertion in a silicon matrix, with respect to control of doping atom activity, defect passivation or suppression of carrier traps, has been evidenced and characterized [1–4]. Presently, silicon hydrogenation is studied with the aim of developing and improving the technological method known as ‘smart cut’, used in the fabrication of ‘silicon-on-insulator’ (SOI) substrates [5]. The smart cut technique involves creating a high density of crystal defects at a certain depth under the surface of an Si wafer and then removing the Si layer situated above the mechanically weakened region by an appropriate method. The as-removed Si layer should be single crystalline and, ideally, free of defects for use in multilayer architecture of the ‘single crystal layer/amorphous insulating layer/single crystal substrate’ type. Ion implantation or treatment in a plasma of light atomic species, such as H or He, is a practical way of inducing crystal defects with a certain distribution profile under the Si wafer surface [5–10].

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Successful results have been reported for the smart cut procedure using hydrogen ion implantation, where layers as thin as 200 nm have been removed from Si wafers [5]. In our research, hydrogen plasma is employed to induce the necessary number of defects. Apart from the lower cost, the use of H-plasma instead of H ion implantation could, under appropriate conditions, be less harmful for the removal of the Si layer by smart cut. Also, the possibility of peeling-off even thinner layers should be taken into consideration [11]. In general, the defects induced in Si by H ion implantation or H-plasma treatment are planar defects, bubbles and dislocation loops [12, 13]. The presence of high H concentration levels in the Si lattice somehow complicates the problem, in the sense that the resulting defects are possibly decorated with hydrogen and, consequently, different from the classical crystal defects encountered in Si. To control the type of defects induced either by ion implantation or plasma treatment, identification and characterization of the defects in hydrogenated Si is necessary. A previous paper [14] reported on surface roughening and the formation of crystal defects in the H-plasma-treated Si wafers as a function of treatment parameters and electrical conductivity of the wafer. In this paper, we present qualitative and quantitative characterization of the \{111\} planar defects in H-plasma-treated Si wafers by high-resolution transmission electron microscopy (HRTEM). Based on strain field data obtained by quantitative HRTEM image processing, a possible structural model for the defect is derived and verified by HRTEM image simulations.

2. Experimental

In this study, p and n-type (001) Si wafers were used with different electrical resistivities (1, 4.5 and 5 Ω cm) submitted to a 110-MHz RF H-plasma treatment for 1, 2 and 4 h. Wafer temperature during H-plasma treatment was maintained at 250°C. Specimens for transmission electron microscopy (TEM) observations were prepared in plan-view and cross-section by mechanical polishing and ion milling using a Baltec RES 010 machine. Conventional TEM observations were performed with a Philips CM 20 electron microscope operated at 200 kV, while a JEOL 4000EX electron microscope operating at 400 kV was used for the HRTEM studies. It is worth mentioning that, during the high-resolution (HR) observations, as a result of the 400-keV electron beam irradiation, crystal defects were created in the Si sample. These are the \{113\} defects analyzed elsewhere [15, 16] and are not the subject of this paper. However, being aware of this artefact, only HR images recorded during the first minute of observation in a certain location were considered, before the \{113\} defects began forming.

3. Results and discussion

Figure 1 shows the effects of hydrogen plasma irradiation on the morphology and structure of the silicon wafer surface by conventional TEM. The main features are
surface roughening (figures 1b and c) and the presence of three types of defects: flower-like defects (type A in figure 1a) observable on plan-view specimens close to the $B = [001]$ zone axis and identified as hydrogen bubbles [12], {111} planar defects (type B) and {001} planar defects (type C), revealed in the cross-section specimens. Surface roughening is caused by a preferential etching of the Si wafer in the hydrogen plasma, leading to the formation of micropyramids with {112} crystallographic planes as lateral faces. Throughout this paper, only the {111} planar defects will be analyzed by conventional TEM, HRTEM and HRTEM image processing and simulation.

3.1. Diffraction contrast analysis

The most frequently encountered defects in hydrogenated Si wafers are disposed along the {111} planes. The slip system in Si is $\langle 110 \rangle \langle 111 \rangle$, i.e. the dislocations lie along the $\langle 110 \rangle$ directions and can slip across the dense {111} planes.
A typical example of a planar defect in materials with diamond structure is the stacking fault, resulting from the dissociation of a perfect dislocation into partials. However, the \{111\} defects in the hydrogenated Si wafers show features which make them different from the classical \{111\} defects in materials having diamond structure.

Figures 2 and 3 show typical conventional TEM images of the \{111\} defects encountered in plasma hydrogenated Si samples. The bright-field (BF) and dark-field (DF) images in figure 2 are obtained in Bragg condition with the 111 spot strongly excited. Two \{111\} defects can be observed; one, showing fringes, is buried at about 800 nm under the surface. The habit plane for this defect was identified as the (111) plane by specimen-tilting experiments. The orientation of the reflection vector $g = (1\bar{1}1)$ with respect to the bright outer fringe of the planar defect on the dark-field image indicates that the defect has an intrinsic character, i.e. has a missing silicon plane [17]. The second defect is seen in edge-on orientation: disposed in the (1\bar{1}1) plane and intercepting the wafer surface.

As a typical feature for this type of defects, the characteristic contrast fringes are not straight but slightly curved, as can be noted in comparison to the line drawn on figure 2b. It indicates that the defect is not limited to a single crystallographic plane, but affects several adjacent planes.

Information on the displacement vector characterizing the \{111\} planar defects can be extracted by imaging a defect in Bragg condition using the diffraction spots for which it becomes invisible. Two beam bright-field images of such a defect are presented in figure 3, where the (1\bar{1}1) and (2\bar{2}0) reflections have been excited. The characteristic fringes of the defect are visible for $g = (1\bar{1}1)$, but become invisible for $g = (2\bar{2}0)$, showing that the characteristic displacement vector $R$ has no components along [2\bar{2}0] and is most probably oriented perpendicular to the habit plane, as in the case of regular stacking faults [18].

Figure 2. Bright-field (a) and dark-field (b) images of a planar \{111\} defect. ‘T’ and ‘B’ denote, respectively, the top and bottom ends of the planar defect intercepting the specimen surfaces. The black line on the DF image has been added as a reference to evidence the fringe curvature.
3.2. HRTEM observations

To ascertain the structure of defects at atomic level, HRTEM observations were performed on \{111\} defects in edge-on orientation. Figure 4 shows HRTEM images of the \{111\} planar defects in the plasma-hydrogenated silicon, revealed in a very thin (figure 4a) and in a thicker part of the specimen (figure 4b).

Figure 4a shows a sharp contrast variation in the defect plane where the columns of atoms are revealed by a much brighter row of dots. In the centre of the figure, the defect migrates over two adjacent \{111\} planes leaving a jog behind. The interpretation of the HRTEM image of the \{111\} defect in the thicker part of the foil (figure 4b) is less straightforward. This is due to the high strain field accompanying the \{111\} planar defects in hydrogenated Si specimens. The strain field along the \{111\} defects is revealed, even at low magnifications, by diffraction contrast (see the edge-on \{111\} planar defect in figure 2). This high-strain field could be related to the presence of hydrogen decorating these defects. In figure 2, one can see that the strain distribution along the defect is not homogeneous for the defect seen edge-on.
This might be related to the presence of partial dislocations and jogs, which can occur within the thickness of the thin foil. The effect of this high-strain field on HRTEM images strongly alters the contrast along the defect. A variation and even inversion of the phase contrast across the defect can be observed (figure 4b). In regions of higher strain, the contrast is almost smeared out. Moreover, the defect is not strictly limited to a single {111} plane but migrates to adjacent {111} planes, forming jogs. This fact explains the curvature and deformations of the fringes on the diffraction contrast images of the {111} defects. In this situation, proposing a defect structure at atomic level is not an easy task. Moreover, by examining the contrast along the defect plane (figure 4b), a typical contrast consisting of pairs of brighter dots at adjacent rows of dots is frequently observed (arrowed in figure 4b). Several atomic models of the {111} defects in hydrogenated silicon have been proposed, all of them taking into consideration the breaking of Si–Si bonds along {111} planes and saturation of the
resulting dangling bonds with hydrogen atoms, accompanied by longitudinal and/or transversal shifts of the Si lattice on each side of the defect. Atomic models proposed by Ponce et al. [19], Deák and Snyder [20], Van de Walle et al. [21], Zhang and Jackson [22], Heyman et al. [23] were summarized and simulated as HRTEM images by Muto et al. [24].

3.3. HRTEM image processing

To decide on a structural model of the \{111\} defect in plasma-hydrogenated Si, experimental HRTEM images of \{111\} defects were processed to determine the strain field around the defect. In the next section, we present such an image processing based on the geometrical phase method (GPM) proposed by Hýtch et al. [25] and using specialized routines under the Digital Micrograph™ 3.3.0 software package for Mac Intosh computers. The geometrical phase method is briefly described in appendix A.

Figure 5a is a HRTEM image of the \{111\} defect in hydrogenated Si in the very thin part of the foil (see figure 4a), while figure 5b gives its Fourier transform. An \(Z-Y\) coordinate system was chosen, as indicated near the lower left corner of the HR image.

As in a diffraction pattern of a thin foil containing planar defects, the spots in the FFT spectrum show streaks oriented perpendicular to the planar defect in the HRTEM image. The phase images in figure 5c and d have been obtained by selecting the \(g_1/22\) and \(g_2/22\) spots in figure 5b with a Gaussian mask. The local phase across the images is calculated with respect to the average phase value corresponding to a reference area delimited by a rectangle in the lower part of the phase images. The size and position of this reference area was chosen in such a way to eliminate regions showing stray \(+\pi/\pi\) phase jumps not related to the defect. On the phase images, white/black areas correspond to regions of higher (positive)/lower (negative) phase, while grey areas are in phase (zero) with the reference area. Note in figure 5c, on the \(g_1\) phase image (\(\phi_{g1}\)), a clear phase jump of the \(\bar{1}11\) fringes across the defect with respect to the reference area.

Using the two phase images, the displacement field \(u(r)\) can be calculated according to equation (A4) in appendix A. The two real space vectors corresponding to the reciprocal \(g_1 = [111]*\) and \(g_2 = [224]*\) vectors are \(a_1 = [111]\) and \(a_2 = 1/12[\bar{1}1\bar{2}]\). In the \(X-Y\) image coordinate system, \(a_1\) is parallel to the \(OY\) axis and \(a_2\) parallel to the \(OX\) axis. The displacement field \(u(r)\) can be mapped by imaging its \(u_x(r)\) and \(u_y(r)\) components according to the equations:

\[
\begin{align*}
  u_x(r) &= (-1/2\pi)[\phi_{g1}(r)a_{1x} + \phi_{g2}(r)a_{2x}] \\
  u_y(r) &= (-1/2\pi)[\phi_{g1}(r)a_{1y} + \phi_{g2}(r)a_{2y}]
\end{align*}
\]

In our image coordinate system \(a_{1x} = 0, a_{1y} = |a_1| = \sqrt{3}/3\) and \(a_{2x} = |a_2| = \sqrt{6}/12, a_{2y} = 0\), which turns equation (1) into:

\[
\begin{align*}
  u_x(r) &= -(\sqrt{6}/24\pi)\phi_{g2}(r) \\
  u_y(r) &= -(\sqrt{3}/6\pi)\phi_{g1}(r)
\end{align*}
\]
Figure 5. (a) HRTEM image of the \{111\} defect in the thin region; the assigned $X$–$Y$ coordinate system is figured. (b) Fourier transform of the HRTEM image. (c) and (d) Phase images $\phi_{g_1}$ and $\phi_{g_2}$ obtained by $\text{FFT}^{-1}$ using a Gaussian mask around $g_1 = \{111\}$ and $g_2 = \{224\}$, respectively. (e) and (f) $u_x(r)$ and $u_y(r)$ components of the displacement field $u(r)$ around the defect. (g) Line profile of the phase across the defect in the $\phi_{g_1}$ phase image averaged over the 8-nm width of the rectangle in the left part of the image. (h) Line profile of the $u_y(r)$ component across the defect in the positive sense of the $OY$ axis. (i)–(k) Maps of the $\varepsilon_{xx}$, $\varepsilon_{yy}$ and $\varepsilon_{xy}$ components of the symmetrical strain matrix $\varepsilon_{ij}$. (l) Line profile of the strain component $\varepsilon_{yy}$ across the defect.
expressed in fractions of the lattice parameter, $a$, or:

$$u_x(r) = -0.176 \phi_{g_2}(r)$$

$$u_y(r) = -0.499 \Phi_{g_1}(r)$$

expressed in nm (the lattice parameter of Si is $a_{Si} = 0.543$ nm).

The $u_x$ and $u_y$ components of the strain field calculated with (3) are depicted in figure 5e and f where, again grey means no displacement, white means positive displacement (in the positive sense of the coordinate axis) and dark means negative displacement. In figure 5f, one can notice a uniform negative rigid body displacement.
along $OY$ (or $[\overline{1}11]$ direction) of the upper part of the image with respect to the reference area. The phase jump, as well as the displacement field, can be quantitatively expressed using line profiles across the defect. To reduce the influence of noise, the line profile has been averaged over the 8-nm width of the rectangle figured in the left hand side of the $\phi_g 1$ and $u_y$ images (figure 5c and f). Thus, the phase jump revealed by the line profile across the defect in the positive sense of the $OY$ axis (figure 5g) measures $0.72\pi \pm 0.044$ radians. Correspondingly, the value of the $u_y$ displacement measured on the line profile (figure 5h) is $0.116 \pm 0.002$ nm. It is worth noting that the phase jump is not steep, but it occurs over approximately 2 nm across the defect. Accordingly, the displacement field $u_y$ shows a finite gradient across the defect. Thus, a jump of 0.116 nm takes place over a range of about 2 nm around the planar defect.

The displacement gradient corresponds to a strain that can be calculated according to equations (A5) and (A6) in appendix A. The three components of the symmetrical strain matrix $\epsilon_{ij}$ are graphically represented in figure 5i–k, where bright and dark means tensile and compressive strain, respectively. One can notice that the defect is characterized by a well-defined $\epsilon_{yy}$ compressive strain confined to a narrow region around the defect (figure 5j). The line profile of $\epsilon_{yy}$ across the defect, averaged over the width of the rectangle (8 nm) marked in the left part of the image in figure 5j, is asymmetrical and shows two minima, one of 13% and the other of 8%. The width of the two-peaked structure, defined as the range between the positions corresponding to 10% of the minimum value, measures about 2.2 nm. By comparing the $\epsilon_{yy}$ strain image (figure 5j) with the original experimental HRTEM image (figure 5a), one can see that the two minima of the strain field actually refer to the position of the two rows of bright dots adjacent to the brighter row of dots revealing the defect.

The existence of a strain field around the $\{111\}$ planar defect in hydrogenated Si represents a major difference from the $\{111\}$ stacking faults encountered in fcc materials, which show no strain far from the limiting partial dislocations. The presence of a strain, significant through both its amplitude and range around the $\{111\}$ planar defect in hydrogenated Si wafers, should be related to the presence of hydrogen decorating the planar defects. Previous Raman measurements [14] revealed the existence of hydrogen in these specimens. In our opinion, inside the Si lattice, hydrogen acts in two ways: on one hand, due to its high chemical reactivity, it forms volatile Si–H compounds leading to the removal of Si atoms from the lattice; on the other hand, it saturates the resulting Si dangling bonds, as we will further show in the proposed models for the $\{111\}$ defect.

### 3.4. HRTEM image simulation

To derive a structural model of the defect, results provided by the quantitative image processing in the previous section are used. As shown by conventional TEM and confirmed by GPM analysis, the displacement vector characterizing the $\{111\}$ defect is oriented perpendicular to the defect plane and has no component parallel to it ($u_x = 0$). The average value of the displacement, obtained by the GPM analysis is
\[ |R| = u_y = -0.116 \pm 0.002 \text{ nm}, \ i.e. \ -1/2.7d_{111} \text{ hence larger than the displacement field for a stacking fault.} \]

We used Crystal Kit software for structural modelling of the defect. Starting from the undistorted Si crystal structure projected along [110] (figure 6a), the 0.116-nm rigid body displacement has been applied to the upper part of the matrix with respect to the fixed bottom part. Two structural models (A and B) can be derived using the same displacement value. In one case, only one layer of Si atoms adjacent to the interface has been removed (model A in figure 6b), while in the second case, both Si layers close to the interface have been removed (model B in figure 6c). In both cases, we consider that the resulting dangling bonds are saturated with hydrogen.

A unit cell has been defined in each case and simulated HR images have been calculated using the Mac Tempas software. Figure 7a and b present a series of simulated images obtained for different values for the lens defocus and specimen thickness, corresponding to the two structural models. At a visual inspection of the simulated images based on the two structural models and a qualitative comparison with the experimental images, it can be concluded that model B provides a better fit for the HR images of the defect. Thus, the defect is imaged either as a single

![Figure 6. Possible structural models of the \{111\} defect based on the displacement values obtained by the GPM analysis. (a) Undistorted Si structure projected along [110]; the horizontal line marks the interface between the bottom fixed part of the crystal and the upper part to which the displacement vector is applied. (b) Structural model A of the \{111\} defect where one Si layer has been removed. (c) Structural model B of the \{111\} defect where two Si layers have been removed. (d) and (e) Enlarged view of the atomic arrangement close to the defect plane for models A and B, respectively; interatomic distances in nm are indicated.](image-url)
row of brighter dots in the thin areas of the specimen, as in figure 4a, or as a double row of bright dots in the thicker regions, as in figure 4b.

The structural model in figure 6c corresponds to the H$_2$C$_3$ defect model described in [26], where the Si–Si chemical bonds were cut and the hydrogen atoms saturate the resulting dangling bonds. Indeed, in the case of model B, where two rows of Si atoms are removed, the defect can be chemically stabilized by saturating with H the dangling bonds of the two rows of Si atoms adjacent to the defect plane. Consequently, the planar defect could be described as a collection of H$_2$C$_3$ defects lined up on a {111} plane.

Model B seems to be favoured from the chemical and energetic point of view against model A. An enlarged structural model of the atom arrangement close to the defect plane for the two models A and B is shown in figure 6d and e, respectively, where the real distances (not the projected ones) between the Si atoms are specified in nm. The chemical bonds are also given for the atoms limiting the defect. Note that these models do not take into consideration any atomic relaxation around the defect. In the case of model A, one sees that the shortest distance between the Si atoms bordering the defect is 0.297 nm instead of 0.235 nm, as in a perfect structure. Consequently, there are two possibilities for saturating the dangling bonds:

(i) The Si atoms delimiting the defect remain unbound and the dangling bonds, one for each Si atom limiting the defect from the upper crystal and three for each Si atom limiting the defect from the bottom crystal, are saturated with H atoms (marked with (1) in figure 6d);
(ii) The formation of a stretched bond between the Si atoms delimiting the defect and saturation with H atoms of the two remaining dangling bonds belonging to the Si atoms limiting the defect from the bottom crystal (marked with (2) on figure 6d).

The two variants of the model A need either an increased number of H atoms for the saturation of the dangling bonds or the formation of a stretched chemical bond, which is energy costly. Model B (enlargement in figure 6e) seems much more plausible, since it only involves Si–Si bond-cutting and saturation of the resulting dangling bonds with H atoms, one for each Si atom neighbouring the defect. The defect could be physically formed by removing the Si atoms from the structure as a result of the etching effect of the H-plasma on the Si wafer.

4. Conclusions

Si wafer exposure to a hydrogen RF-plasma leads to the formation of specific crystal defects decorated with H atoms. In this paper, we analyzed the typical \{111\} defects induced in Si by plasma hydrogenation. We described the diffraction contrast characteristics and studied the high-resolution images of edge-on oriented \{111\} defects. The information extracted from the diffraction contrast images on the intrinsic character of the defect and the habit plane has been confirmed by HR observations. HRTEM images show that the defect is not limited to a single \{111\} plane, but it shows jogs and migrates to adjacent \{111\} planes. Quantitative image processing by the geometrical phase method of the HRTEM image of an edge-on \{111\} defect revealed the intrinsic character of the defect and provided quantitative information on the lattice strain around the \{111\} defects. The existence of a lattice strain around the defects due to decorating H atoms makes them different from simple stacking faults. Quantitative information derived by image processing has been used as input for structural modelling of the defect, resulting in two possible models. Simulated HR images based on the two structural models have been compared with experimental images of the defect. We have shown that only one of the two possible structural models provides simulated HR patterns that fit well the experimental images, both for the small and larger specimen thicknesses. This structural model corresponds to the H$_2^*$ defect described in the literature, which seems more favourable from a chemical and energetic points of view.

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Appendix A: short description of the geometrical phase method

The geometrical phase method (GPM) [25] considers the HRTEM image of the perfect crystal as a 2D periodic pattern, which can be described as a Fourier series:

\[ I(r) = \sum_g I_g(r) \exp(2\pi ig \cdot r) \]  

(A1)

where \( I_g(r) \) is the Fourier component corresponding to the reciprocal vector \( g \).

In the HRTEM image of a thin foil containing structural defects, the complex, locally varying Fourier components are characterized by an amplitude \( A_g(r) \) and a phase \( \phi_g(r) \), both depending on the position in the image.

\[ I_g(r) = A_g(r) \exp(i\phi_g(r)) \]  

(A2)

The amplitude \( A_g(r) \) gives the local contrast of the HRTEM fringes corresponding to \( g \), while the phase \( \phi_g(r) \) represents the local shift of the HRTEM fringes from their ideal position (geometrical phase), due to the presence of a lattice defect. The geometrical phase \( \phi_g(r) \) may be described either in terms of local displacement \( u(r) \) of the fringes from their undistorted position, or as a local variation of the fringe interspacing (i.e. variation of \( g \)):

\[ \phi_g(r) = -2\pi g \cdot u(r) \text{ or } \phi_g(r) = 2\pi \Delta g \cdot r \]  

(A3)

By selecting an individual spot \( g_1 \) from the fast Fourier transform (FFT) image with an appropriate mask, one can extract by inverse Fourier transformation the phase image \( \phi_{g1}(r) \), representing the local shift of the corresponding family of fringes from their ideal position in an undistorted crystal.

The local phase is calculated with respect to an undistorted reference area on the image, far from the strain field of the defect. A second phase image \( \phi_{g2}(r) \) can be obtained by moving the selecting mask on a different vector \( g_2 \), noncollinear with \( g_1 \). Using the two phase images, one can calculate and display the image showing the vectorial displacement field:

\[ u(r) = (-1/2\pi)[\phi_{g1}(r)a_1 + \phi_{g2}(r)a_2] \]  

(A4)

where \( a_1 \) and \( a_2 \) represent the real space vector basis, corresponding to the reciprocal lattice vectors \( g_1 \) and \( g_2 \). By further derivation of the local fringe displacement \( u(r) \), one can calculate and map the matrix \( \epsilon \) of the local lattice distortion:

\[ \epsilon = \begin{pmatrix} e_{xx} & e_{xy} \\ e_{yx} & e_{yy} \end{pmatrix} = \begin{pmatrix} \partial u_x/\partial x & \partial u_x/\partial y \\ \partial u_y/\partial x & \partial u_y/\partial y \end{pmatrix} \]  

(A5)

where \( r=(x,y) \) represents the local coordinates in the image. The strain field and the rigid rotation of the lattice around the defect are expressed, respectively, by a symmetrical matrix \( \epsilon \) and an asymmetrical one \( \omega \), given by:

\[ \epsilon = 1/2(e + e^T) \]  

(A6)

\[ \omega = 1/2(e - e^T) \]

where \( T \) denotes the transpose of the matrix.
References