Scanning probe studies of the electrical activity at interfaces formed by silicon wafer direct bonding

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In order to investigate the electrical properties at the surface of dislocation rich silicon, we conducted Electrostatic Force Microscopy on cross-sections of samples prepared by Wafer Direct Bonding. The applied methods, namely Scanning Kelvin Probe Microscopy and non-contact Scanning Capacitance Microscopy, yield a distinct contrast at the position of the dislocation area, i.e. the bonding interface, indicating strong electrical activity. For an explanation of the explicit electrostatic potential extracted from the experiment a simple model taking into account only an intrinsic charge distribution at the dislocation area appears to be insufficient. Instead, a more complex approach has to be used considering carrier generation and recombination by additional, dynamic mechanisms.

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1 Introduction

A promising approach to provide a regular, nanostructured template for the immobilization of bioactive material on a silicon substrate could be the controlled formation of regular dislocation networks in silicon, provided these translate into a surface electric field variation supporting the self-assembly of appropriate biomolecule patterns. EBIC measurements on such networks and corresponding first principles calculations [1, 2] show that the potential barrier at such dislocations should be in the range of 100 meV, and the emanating electric field around 100 V/cm.

At the surface, however, these properties are not reliably known. In particular, individual surface preparation and conditions might have a major influence on the actual values of these quantities at the silicon surface. To shed light on this problem, we investigated dislocation rich silicon samples, prepared by Wafer Direct Bonding [1], conducting Scanning-Probe based electrical measurements, namely Electrostatic Force Microscopy (EFM). Such measurements, Scanning Kelvin Probe Microscopy (SKM) and Scanning Capacitance Microscopy (SCM), allow a detailed inspection of the electrical surface properties with a lateral resolution of about 50 nm [3].

Unfortunately, due to the specific preparation technique [1], the dislocation network is always covered by a thin layer of unperturbed crystal, inhibiting a direct contact between the microscope tip and the dislocation plane. Therefore, considering the presently obtained characteristic lateral distance between the dislocations of about 50 nm, on the one hand, and the typical screening length in moderately doped silicon of around 150 nm (for a doping density of \(10^{15}\) cm\(^{-3}\)), on the other hand, the potential variation in a plane parallel to the dislocations is rather washed out. To overcome this problem, the experiments were...
done on cross-sections perpendicular to the dislocation network which, therefore, only occurs as the “interface” between the two bonded wafers.

2 Experimental

The measurements were performed on cross-sections of p-type silicon (acceptor density \( N_A \approx 10^{15} \text{ cm}^{-3} \)) samples prepared by Wafer Direct Bonding [1], making sure that no considerable contamination occurs at the contact between both wafers. Thus, the interface was only made up by misfit dislocations between the two wafers. We used conductive coated tips in a “NT-MDT Smena” microscope, working in a “two-pass” setup: in a first pass the surface topography is measured and the acquired profile then is retraced with a certain vertical offset in a second pass, allowing the quantification of electrostatic forces \( F_{\text{Tip-Sample}} \) acting on the tip at constant tip-sample spacing [4]:

\[
F_{\text{Tip-Sample}}(z,U_{\text{tip}}) = \frac{1}{2} \frac{\partial C(z,U_{\text{tip}})}{\partial z} \left( U_{\text{tip}} - \varphi_{\text{surface}} + \text{const} \right)^2.
\]  

In Eq. (1) \( U_{\text{tip}} \) is the voltage applied to the tip, \( C \) the tip-sample capacitance, \( z \) the tip-sample distance and \( \varphi_{\text{surface}} \) the electrostatic potential of the surface. The constant appearing in the last factor accounts for voltage offsets specific for the setup, e.g. the bulk work function difference between tip and sample. For the determination of the surface potential (SKM) both DC and AC \((f = 300 \text{ kHz})\) voltages are applied to the tip and a feedback system regulates the DC component for minimizing the electrostically induced oscillations of the cantilever.

For SCM the component of the cantilever oscillating at the second harmonic of the AC voltage is mapped, providing a signal proportional to the local tip-sample capacitance according to Eq. (2) [5].

\[
\text{Mag}_{\text{SCM}} = U_{\text{AC}}^2 \left\{ \frac{\partial^2 F_{\text{Tip-Sample}}(z,U_{\text{DC}})}{\partial U_{\text{DC}}^2} \right\} = \frac{U_{\text{AC}}^2}{2} \frac{\partial C(z,U_{\text{DC}})}{\partial z} + g(U_{\text{DC}} - \varphi_{\text{surface}}).
\]  

Here \( \text{Mag}_{\text{SCM}} \) is the cantilever oscillation amplitude and \( g \) a function containing derivatives of the capacitance with respect to voltage multiplied by the contact potential. A more detailed description of the measurement principles is given in [6].

To investigate the influence of an external bias on the measured EFM profiles, aluminium contacts were deposited on the sample surfaces parallel to the bonding interface. Then, during the recording of an image, the bias was changed stepwise, allowing a direct evaluation of the changes.

3 Results and discussion

3.1 Scanning Kelvin probe microscopy

An evaluation of the recorded surface potential (depicted in Fig. 1b) yields a value decreased by about 100 mV around the position of the bonding interface while there is no hint of its presence in the topography image (Fig. 1a). From the profile shown in Fig. 1c (upper trace) one can read a full width of the affected region of about 5 \( \mu \text{m} \) and, in addition, a potential difference of around 30 mV between the two wafers used. These findings are in contradiction to the simple model that SKM would measure the electrostatic potential caused by positively charged dislocations. In this case, using the depletion approximation, one could expect the following values:

\[
W = \frac{\sigma}{2N_A} \quad \text{and} \quad \Delta \varphi = \frac{q \sigma^2}{8 \varepsilon_0 \varepsilon_S N_A}.
\]  

Here, \( W \) means the full width of the depletion region, \( \sigma \) the interface charge equivalent to the line charges present, \( N_A \) the acceptor density, \( \Delta \varphi \) the potential change, \( q \) the electronic charge and \( \varepsilon_0 \varepsilon_S \) the dielectric constant of silicon.
Fig. 1 Sample topography (a), simultaneously acquired surface potential map (b), and profiles of topography (lower trace) and surface potential (upper trace) (c), extracted along the lines marked by the gray bars in (a), (b). The “steps” in the upper right of the potential map were caused by applying different DC biases. It appears that the potential at the bonding interface (dotted lines and arrows) is decreased by about 100 mV.

Assuming \( \sigma \) in the range of \( 10^{11} \) charges per cm\(^2\), Eq. (3) would yield \( W \approx 1 \) \( \mu \)m and \( \Delta \phi \approx +200 \) mV, respectively. While the estimate for the width appears to be reasonable, the sign of the calculated potential is different from the measured one. In contrast, the presence of negative charges at the bonding interface could be responsible for the recorded negative surface potential but not for the detected width, since the p-type silicon then would be in a state of majority carrier accumulation, causing a very effective screening.

Another approach to explain the observed change in \( \Delta \phi \) across the dislocation area takes into consideration the photogeneration of carriers by the detection laser of the Scanning Probe Microscope. This laser (\( \approx 1 \) mW power at a wavelength of 650 nm) could create an excess carrier concentration at the same order of magnitude as the equilibrium majority carrier concentration for this doping level. Then SKM would rather measure a Surface Photovoltage (SPV) or a Dember Voltage than the potential created by the charged dislocations. The expected voltage differences \( \Delta V \) can be expressed by [7, 8]:

\[
\Delta V = V_t \ln \left( 1 + \frac{\Phi L}{C(sL + D)} \right)
\]

with \( V_t \) being the thermal voltage (\( \approx 25 \) mV at room temperature), \( \Phi \) the photon flux, \( s \) the surface recombination velocity, \( L \) the minority carrier diffusion length, \( D \) the minority carrier diffusivity and \( C \) a constant including surface reflectivity and equilibrium carrier concentration. In a one-dimensional approximation, the potential would be increased for an illuminated position with low recombination velocity and unchanged for an illuminated position with very high recombination velocity. This could explain the measured SKM voltage drop since dislocations are expected to exhibit high recombination activity fixing the local potential according to Eq. (4). However, the recorded decay lengths are much shorter than typical diffusion lengths, indicating that a modelling of the profiles would require at least a two-dimensional computation of the drift-diffusion equations.

If a bias voltage is applied across the bonding interface, the extracted SKM profiles (Fig. 2a) show, that a large amount of the applied voltage is dropped at the position of the dislocation network. A more detailed inspection of the voltage differences within the scan range (Fig. 2b) yields an average drop of about 50% of the applied bias, illustrating a very high local lateral resistivity.
Fig. 2 SKM profiles for different sample bias (a) and dependence of the voltage drop inside the scan area on bias voltage (b).

Fig. 3 SCM map recorded from the same area as the surface potential data (a). The profile displayed in (b) reveals three regions having different capacitive properties. Expected qualitative response curves as described in the text (c).

3.2 Scanning capacitance microscopy  Comparing the SCM map in Fig. 3a with the SKM data in Fig. 1b, an important difference is recognized: While the surface potential scan may be separated into two regions showing different values (an almost constant potential everywhere but at the bonding interface, where the potential is greatly decreased) a third feature appears directly at the interface.

From Eq. (2) one can read that for a DC voltage nearly equal to the surface potential, which was the case for our measurements, the expected amplitude is proportional to the derivative of the total tip-sample capacitance with respect to the tip-sample separation. Applying a MOS capacitor model, the shape of the response is expected to be equal to typical Capacitance-Voltage (C-V) curves, perhaps changing from a more low frequency type for high excess carrier density (curve “A” in Fig. 3c) to a high frequency type (curve “C” in Fig. 3c) near the interface.

Assuming that the local potential value only shifts the capacitive response curve, one would expect an increase in capacitance when approaching the bonding interface because the p-type silicon should be in a state of majority carrier accumulation. This explains the regions with increased SCM amplitude appearing as positive peaks (“B”) in Fig. 3b, which have the same lateral extension as the voltage “dip” in Fig. 1.

A second region, appearing as darker area further away from the interface in Fig. 3a may have a similar origin since regions with slightly increased potential were found by SKM (best seen in Fig. 2a causing the surface to be in a state of carrier depletion.

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Within this concept, the abrupt drop of the signal directly at the interface ("C") can only be explained by assuming the influence of a positive charge in a region with a low density of excess carriers. This would change the response curve to the high-frequency shape and cause a strong depletion of the material. So, in our opinion, the central minimum in the SCM profile represents the depletion layer generated by dislocations.

4 Conclusions It was shown that both Scanning Kelvin Probe Microscopy and non-contact Scanning Capacitance Microscopy can be used to map surface electrostatic properties of semiconductor samples. Unfortunately for an interpretation of the recorded data, carrier generation and recombination have to be taken into account.

For the sample discussed in the present work SCM appears to deliver data that coincide with the proposed model of having positively charged dislocations surrounded by depleted silicon. SKM was not able to image this feature but gave the opportunity to quantitatively measure voltage variations caused by recombination properties.

References