SSRM Characterisation of FIB Induced Damage in Silicon

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Abstract. Scanning spreading resistance microscopy (SSRM) has been applied to study focused ion beam (FIB) induced damage in silicon in dependence on ion irradiation doses from $10^{12}$ cm$^{-2}$ to $2 \times 10^{16}$ cm$^{-2}$. Starting from the lowest dose, SSRM detects increasing spreading resistance (SR) with increasing dose. For doses from $2 \times 10^{13}$ cm$^{-2}$ to $4 \times 10^{14}$ cm$^{-2}$, a slight decrease of SR is measured whereas for higher doses SR again slightly increases. The results are explained by physical effects like decreased carrier mobility due to increased scattering, amorphisation of silicon and precipitation of implanted Ga ions. The results clearly prove that SSRM is well suited for the fast detection of ion beam induced damage with high lateral resolution.

1. Introduction

Physical sputtering with focused ion beams (FIB) is an important patterning technique in semiconductor processing down to nanoscale sized structures [1] (i.e., device modification, optimisation of tips for scanning probe microscopy (SPM) or nanolithography). The highly energetic ions, however, induce severe damage in the target material. For silicon, it usually leads to an amorphisation of the processed area and eventually of its surrounding. Because the focused ion beam is usually Gaussian-shaped, not only the purposely exposed area has to be considered but also the tails of the beam may lead to serious damage outside of the processed region, where already a small amount of damage strongly influences electrical properties such as charge carrier mobility or charge carrier lifetime. Usually no high temperature anneal is performed after FIB processing: Thus, damage remains and may lead to a low performance or even failure of affected devices. Even if annealing occurs, the resulting Ga doping could affect the device performance. An accurate knowledge of the degree and the distribution of the FIB induced electrical damage, therefore, is of vital interest but a fast quantitative detection on a micro- or even nanoscale without any complex sample preparation using conventional techniques is not possible and only limited data on this matter has been published (e.g. [2]).

In this work, we successfully demonstrate that Scanning Spreading Resistance Microscopy (SSRM) can be applied as a highly sensitive technique with high spatial resolution for the electrical characterisation of the FIB induced damage in silicon by energetic gallium ions (30 keV). Because no sample preparation is needed for SSRM characterisation, the irradiated sample can be characterised immediately after FIB processing and a systematic study of the induced damage can be performed with reasonable effort. In this work, the dependence of the damage on the irradiated dose has been performed focusing on very low doses which already occur during FIB inspection mode.
2. Experimental

A FEI SB 800 single-beam focused ion beam system was used for the irradiation of n-type silicon samples with a specific resistance of about 0.01 Ω cm. In this FIB system, the primary Ga⁺ ion beam is accelerated to 30 kV. For all irradiation experiments similar ion beams of 1 to 3 pA were used in order to realise very low doses and to avoid additional effects due to significantly different beam currents. For each different dose, a circular pattern with a diameter of 4 µm was irradiated. Special care was taken to avoid any irradiation due to focussing, alignment or inspection. To verify the reproducibility and accuracy of the experiments 5 different samples were processed with different dose ranges which partly overlap for the different samples (referred to as A-E with increasing maximum dose). The analysed ion doses for all samples range from 10¹² cm⁻² to 2·10¹⁶ cm⁻². For standard processing, much higher doses are used. However, such doses are applied during FIB inspection and adjustment for single beam systems (reaching from 10¹² cm⁻² to 10¹⁶ cm⁻² depending on magnification and beam current for beam currents up to 70 pA), in the surroundings of purposely processed areas due to the tail of the ion beam or bombardment by neutral Ga atoms during FIB operation [3]. The aim of the study, therefore, is to investigate effects due to unintentional but unavoidable ion irradiation which might deteriorate affected devices or functional layers.

All SSRM measurements were performed with a Veeco DI5000 system. In SSRM mode, a conductive tip scans the sample surface in contact mode with a high load (10-12 GPa for silicon samples) and a DC bias \( V_{\text{bias}} \) is applied between the tip and the sample with the tip being grounded. The resulting current through the sample is measured using a logarithmic current amplifier in the range of 10 pA to 0.1 mA with an output voltage signal \( V_{\text{SSRM}} \) which is proportional to the overall resistance. Due to the small contact area and the high conductivity of the tip, the dominant resistance term is the spreading resistance in the sample. To avoid possible anodic oxidation on Si surfaces, negative \( V_{\text{bias}} \) was applied to the sample (up to -1.4 V). In this case, \( V_{\text{SSRM}} \) is more negative for higher resistances. For all SSRM measurements, standard boron doped diamond coated Si probes with a nominal tip radius of about 35 nm were used. Both, spreading resistance and topography were detected simultaneously in SSRM mode. The topography results were verified with the same SPM system operating in tapping mode using standard tips with a nominal tip radius of less than 10 nm.

Before FIB processing, the native oxide was removed for all samples using a HF dip. After FIB processing, the samples were kept under vacuum conditions in the FIB processing chamber until the SSRM measurement. This procedure is not absolutely necessary because it is possible to use SSRM even with native oxide applying high pressure and using tips with high stiffness. This might, however, lead to distortions and reduced reproducibility of the results.

3. Results and Discussion

SSRM measurements clearly identify the irradiated circular regions within the unaffected substrate for all samples. In contrast to scanning capacitance microscopy (SCM) [2], significant differences in SSRM signal can be observed for different ion doses over the whole analysed dose range. As an example, figures 1 and 2 show the SSRM and topography map of sample B. The quantitative evaluation for each irradiated structure is summarised in figure 3 where both, the SSRM signal and the corresponding topography measurement results are given (as an average over the centre region of each processed area). For the SSRM signal, negative values correspond to increased spreading resistance, positive topography values correspond to an elevation, both compared to the unaffected substrate. Since the results for the different samples and for slightly different ion beams show good agreement in the overlapping dose ranges, the reproducibility and consistency of the experiments is given.

For the SSRM signal, three regimes can be distinguished (i.e., I-III in figure 3). The first is characterised by a strong increase in spreading resistance (SR) up to a dose of about 1.5·10¹³ cm⁻², whereas the second exhibits a less pronounced decrease in SR up to a dose of about 5·10¹⁴ cm⁻², while the third regime again shows an increase in SR with a SR value at the maximum dose of 2·10¹⁶ cm⁻² which is similar to the maximum of the first regime. From the presented results, a saturation of the SR value for
doses above $1 \times 10^{16}$ cm$^{-2}$ might be deduced. The topography measurements reveal a smooth increase up to a value of about 1.7 nm for a dose of about $5 \times 10^{14}$ cm$^{-2}$ which corresponds to the transition from the second SSRM regime to the third. For higher doses, the topography decreases until the maximum investigated dose.

**Figure 1.** SSRM image of sample B (structures 1-9 correspond to Ga doses from $4 \times 10^{13}$ cm$^{-2}$ to $1 \times 10^{15}$ cm$^{-2}$).

**Figure 2.** Topography image of sample B. The maximum height is about 1.7 nm.

**Figure 3.** SSRM output voltage $V_{SSRM}$ and topography results for samples A-E. Closed symbols: SSRM signal (RMS is about 75 mV), open symbols: topography results (max. RMS is about 0.25 nm). I, II, III refer to the different SSRM regimes as described in the text.

The increase in volume for silicon samples irradiated by highly energetic ions is a well known phenomenon called swelling. The implantation of energetic ions into crystalline silicon leads to an increased number of displaced silicon atoms with increasing dose resulting in a fully amorphised silicon layer of 50 to 70 nm thickness for Ga$^+$ doses of about $2 \times 10^{14}$ cm$^{-2}$ (at 30 keV) [3,4]. Since the density of amorphous silicon (a-Si) is smaller than that of crystalline silicon (by 1.7 to 7% [5,6]), this results in an increase of the affected volume. Superimposed to this effect, material removal occurs due to physical sputtering of silicon atoms by the impinging Ga ions. Apparently swelling dominates physical sputtering for the investigated dose range (see figure 3 apart for the very highest dose). Studying the change in morphology of the silicon crystal due to ion irradiation, the implanted Ga ions have to be taken into account where the Ga concentration might even exceed the solubility limit for higher doses (about $10^{17}$ cm$^{-2}$ [4]) leading to Ga precipitations. For such high doses an equilibrium between all mentioned effects will be reached. With even higher doses, the amorphised and Ga-rich regions will be sputtered at the sample surface but simultaneously be pushed ahead into the silicon bulk due to the additional Ga ions.

The resistance increase for the first SSRM regime at very low ion doses is feasible because the carrier mobility strongly decreases with increasing ion dose due to strongly enhanced carrier scattering at displaced silicon atoms. The interpretation of the SSRM signal in regimes II and III, on the other hand, is not that simple since there is not a common understanding of the influence of ion implantation on the electrical characteristics without a subsequent temperature anneal of the sample. Therefore only a qualitative and to a certain extent speculative analysis of the measurement results is suggested for
these regimes. The decrease of the spreading resistance in the second regime is probably correlated with the final stage of the change from crystalline to amorphous silicon. The conduction mechanism and thus the resistivity of non-hydrogenated a-Si depends on various parameters [7] and it seems to be smaller than that of strongly disordered crystalline silicon. It is also known that the resistivity of non-hydrogenated a-Si decreases with increasing implanted dose [8] and pressure [9] which will also result in a decrease of SSRM signal. The increase in spreading resistance in the SSRM regime III can be explained by an increase in the overall resistance due to the increase in incorporated gallium concentration and related distortion in the silicon crystal below the a-Si layer. The estimated saturation of the SSRM signal for the highest doses might be correlated with the equilibrium between sputtering, amorphisation and Ga incorporation as mentioned above and thus constant conditions for the current paths.

Additional effects can occur during ion bombardment where some of them have not been discussed or considered within this work like self-annealing due to the introduced energy during implantation, influence of ion beam current, substrate doping level or temperature. These effects will be investigated in future studies which will also include high dose effects and the detailed characterisation of damage outside of the purposely irradiated regions. Here, high resolution transmission electron microscopy (HRTEM) will be necessary to fully understand and verify the SSRM measurements.

Considering the SSRM results it is suggested that FIB processing of highly integrated micro and nano systems should be performed at the final stage of processing as devices are usually passivated and thus protected from the unavoidable ion irradiation during FIB inspection, alignment and processing itself.

4. Conclusion
A systematic study of the dose dependence of electrical damage in silicon after Ga ion bombardment at low doses has been performed. The presented results clearly demonstrate that SSRM is a highly suitable technique to detect and quantitatively characterise damage electrically after the irradiation with energetic particles at high lateral resolution. Due to the additional high-resolution detection of changes in topography the technique can give a rather complete picture of the sample after irradiation. Although vertical information is not directly provided, a major advantage of the technique is its simplicity compared to other techniques like HRTEM allowing for savings in preparation times and costs.

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