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#### Full length article

## Investigation of tip-depletion-induced fail in scanning capacitance microscopy for the determination of carrier type



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#### ABSTRACT

Scanning capacitance microscopy (SCM) was performed on an n-type Si multilayer structure doped by phosphorus whose concentration ranges from  $2\times10^{17}$  to  $2\times10^{19}$  cm<sup>-3</sup>. Three types of tips were used, i.e. fresh Pt/Ir coated tip, worn Pt/Ir coated tip and non-coated commercial Si tip. The use of fresh Pt/Ir coated tips produces SCM result in good agreement with the doping profile including the correct identification of the carrier type. In contrast, a worn Pt/Ir coated tip which has lost its metal coating and a non-coated tip will fail to recognize successfully the carrier type for phosphorus dopant concentration above  $8\times10^{18}$  cm<sup>-3</sup> (identifying as p instead of n) due to the tip depletion effect. These results alert us to carefully interpret the SCM results, especially in the case for identification of carrier type inside the sample of interest which is unknown.

#### 1. Introduction

Scanning capacitance microscopy (SCM) has emerged in the last two decades as a significant technique for determining two-dimensional (2D) dopant/carrier profile to meet the challenge of characterization on continuously decreasing scale of semiconductor devices [1,2]. In SCM, an alternating voltage (Vac) is applied along with a direct voltage ( $V_{\rm dc}$ ) on the tip-sample configuration. While  $V_{\rm dc}$  determines the measurement operation point, Vac periodically accumulates and repels local carriers inside the sample, making changes of the tipsample capacitance (dC/dV) which is detected by an ultra-highfrequency (~1 GHz) sensor, yielding SCM signal. The magnitude of SCM signal depends on the carrier concentration (a higher concentration normally corresponds to a smaller SCM amplitude) whereas its polarity (dC/dV phase) is related to the carrier type since electrons and holes respond to the variation of V<sub>ac</sub> in an opposite way. In an ideal case, SCM signal on p-type and n-type material should have a phase difference of  $\pi$ .

Since its development, SCM has been widely studied on various semiconductor materials [3–10] for dopant profiling and/or p-n junction delineation [11,12]. Also, it has been applied as a tool for analysis on materials in the absence of information, for example, for the identification of carrier type [13,14]. Factors influencing the capacitance-voltage characteristics of tip-sample system in SCM measurement have been extensively investigated such as stray laser light [15], choice of  $V_{\rm dc}$ [16], modulation voltage  $V_{\rm ac}$ [17], probe geometry [18] and the sample preparation process [19–21]. One special concern in SCM is the tip depletion, which means the possible participation of

In this work, we performed SCM measurement using three types of tips (fresh Pt/Ir coated tip, worn Pt/Ir coated tip and non-coated Si tip) on a phosphorus doped multilayer Si structure with different doping levels to investigate the influence of the tip depletion on SCM measurements in real cases. It is shown that although SCM can help recognizing local carrier type, loss of tip metal coating can produce results misleading for the interpretation of carrier type in heavily n-type doped material.

the tip itself in the SCM signal in consequence of its response to V<sub>ac</sub>[1]. After use of etched tungsten wire as probe tip, metal coated (for instance Pt/Ir and Co/Cr) Si cantilever/tip combinations are in regular use for SCM. This kind of tip does not deplete under voltage bias during SCM measurements. Nevertheless, it has to confront serious tip wear problems concerning the thin metallic coating layer, whose lost can cause the exposure of the Si underneath. V. V. Zavyalov [19] compared the quality of heavily doped Si tips with Co/Cr coated tips and investigated related tip depletion phenomenon, pointing out that this phenomenon arising for Si tips can distort SCM data and thus impact unfavorably quantitative dopant profiling. M. L. O'Malley [22] simulated SCM imaging of Si p-n junction using semiconductor probe tips, revealing the interaction between the tip and the studied sample, in which the contribution of the tip dC/dV to the overall dC/dV is most significant when the tip concentration is on the order of or less than that of the sample. Also, simulations results showed a tip of same conduction type as the sample may produce SCM signal of opposite sign for a certain high sample carrier concentration [22].

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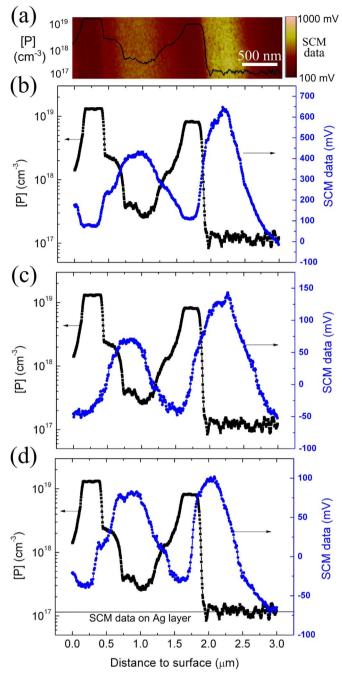


Fig. 1. (a) Original SCM image using a fresh Pt/Ir coated tip. (b) Averaged SCM profile of (a) plotted with SIMS result. (c) SIMS and SCM profile using a worn SCM-PIC tip. (d) SIMS and SCM profile for an ESP tip. The line at SCM data=–70 mV indicates SCM data with measurement on Ag metal layer which can serve for zero calibration of the SCM signal. Measurement conditions:  $V_{\rm dc}$ =–1 V,  $V_{\rm ac}$ =1000 mV for the fresh Pt/Ir coated tip and the ESP tip.  $V_{\rm dc}$ =–1 V,  $V_{\rm ac}$ =2000 mV for the worn Pt/Ir coated tip. Surface is to the left (same for Fig. 2).

#### 2. Experimental

The sample under study is phosphorus doped n-type Si multilayer structure. It consists of 5 regions with varying doping concentrations between  $2\times10^{17}$  and  $2\times10^{19}$  cm<sup>-3</sup> on a p-type substrate with a hole density of  $\sim10^{15}$  cm<sup>-3</sup> (see SIMS profile in Fig. 1(b)). Simple cleavage was conducted to obtain its cross section and native oxide layer was used as insulating layer in SCM measurements. It is known that the oxide layer plays an important role in the SCM characterization performance. Typically, the quality of the surface oxide in terms of

trapped charges and interface states can cause a shift in the flatband voltage. Consequently, contrast reversal may occur which makes it necessary to choose a proper  $V_{\rm dc}$  to avoid such phenomenon [16,23]. During the development of SCM, different types of surface oxidation layer including native oxide, low temperature thermal oxide, wetchemical oxide and ultraviolet (UV) ozone oxide, have been compared, and Si surface treated by UV ozone is believed to have a better quality thus is mostly used in SCM study for improved imaging [19,20]. However, in our case, we have chosen to use the native oxide for all experiments to make sure that the free parameter remains the quality of the tip coating. Furthermore, as will be shown, we have successfully obtained a monotonic behaviour of the SCM signal with the doping level, which is of essential importance. In this sense, we believe that the use of native oxide layer will not affect the reliability of this work.

The tips used are fresh Pt/Ir coated Si tips (SCM-PIC type from Bruker) used for the first time, worn tips where Pt/Ir coating at tip apex is lost after some previous measurements, exposing the Si underneath, and tips without metal coating on front side but just etched Si (ESP type from Bruker). It must be pointed out that the etched Si in all tips here is antimony (n-type dopant) doped and shares a resistivity of  $0.01-0.025~\Omega$  cm corresponding to a dopant concentration of  $\sim 5\times 10^{18}~\rm cm^{-3}$  close to the doping range of our analyzed sample.

Secondary ion mass spectroscopy (SIMS) was done separately to obtain the profile of dopant concentration for doped layers. SCM measurements were carried out with a  $V_{\rm ac}$  of 50 kHz using a Veeco Dimension 3100 atomic force microscope equipped with an SCM module in ambient atmosphere. For all results presented here,  $V_{\rm dc}$  was applied on the sample rather than the tip. Since laser-induced photoelectronic generation may distort the Si sample response in SCM [15,24], this laser effect has been avoided by displacing the laser spot relatively far from the tip end.

For clarity, in this work, lock-in phase was set in such a way that positive SCM data (positive dC/dV phase) would correspond to n-type and negative SCM data (negative dC/dV phase) corresponds to p-type.

#### 3. Results and discussion

The SCM results with the use of the three types of tips are presented in Fig. 1. Fig. 1(a) shows an original SCM image of the sample using a fresh Pt/Ir coated tip. The contrast between layers with various doping concentrations is clearly visible. Fig. 1(b) presents a comparison of the averaged SCM data profile (in Fig. 1(a)) with phosphorus density profile from SIMS. It is seen that results from the two techniques agree well with each other in a way that an increase in doping concentration produces a reduction in SCM data amplitude, as expected. Here, a  $V_{\rm dc}$  of  $\sim$ -1 V has been selected for SCM in order to obtain a monotonic SCM behaviour with doping concentration [16]. Along with the agreement between SCM and SIMS, all the SCM data for the doped layers are positive and have a positive and stable phase, which indicates a correct recognition of SCM signal for all the layers of n-type.

The SCM profile obtained with the worn Pt/Ir coated tip and its comparison with SIMS are shown in Fig. 1(c). The opposite sign of SCM data can be noticed for the two most heavily doped layers, i.e. the  $8\times10^{18}$  and  $2\times10^{19}$  cm<sup>-3</sup> layers. It indicates that using this tip, the two most heavily doped layers have both negative SCM data values instead of positive ones, which, according to the way we have set lock-in phase, should correspond to p-type carriers. This means SCM in this case fails to successfully recognize the carrier type for the two regions. This error of SCM data polarity is related to the tip depletion originated from the wear of the tip: In the tip wear process, the metal coating acting as the gate is gradually removed and the Si inside is exposed. Thus depletion of the tip in response to Vac occurs. Since the material of Si tip has a carrier density around 5×10<sup>18</sup> cm<sup>-3</sup>, for carrier concentration lower than this value, the worn tip could still perform well as a gate and does not cause a reversal of SCM phase, but for carrier density higher than this value, the tip depletion effect might not be neglectable or even can

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