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Interpretation of scanning capacitance microscopy for thin oxides characterization

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ABSTRACT

In this paper, the reliability of the characterization of thin oxides at the nanoscale using Scanning Capacitance Spectroscopy (SCS) is addressed. 5 nm thick thermal silicon dioxide layers are probed by SCS and compared to capacitance versus voltage (*C*-*V*)curves obtained with a classical ("macroscopic") bench using large (e.g. 100 μ m × 100 μ m) electrodes. The detrimental influence of the laser used for the measurement of the deflection of the AFM cantilever is pointed out. It is shown that SCS is not able to provide the correct shape of the d*C*/d*V* versus voltage curve, compared to macroscopic *C*-*V* curves. In particular, a hysteretic behaviour and an increase of the signal in the inversion region are measured by SCS whereas they are not present in macroscopic *C*-*V* curves. We show that this can be explained by a bad electrical contact (presence of contaminants and/or charges) between the Atomic Force Microscopie's tip and the oxide. Macroscopic *C*-*V* curves leads to the correct shape. This shows that SCS won't be fully comparable to macroscopic *C*-*V* curves leads to the correct shape. This shows that SCS won't be fully comparable to macroscopic *C*-*V* curves leads to the correct shape. This shows that SCS won't be fully comparable to macroscopic *C*-*V* curves leads to the correct shape. This shows that SCS won't be fully comparable to macroscopic *C*-*V* curves leads to the correct shape. This shows that SCS won't be fully comparable to macroscopic measurements unless the influence of the contact between the AFM tip and the sample is taken into account.

1. Introduction

The decrease of the size of Metal-Oxide-Semiconductor (MOS) structures and the reduction of the voltages applied on devices come with a reduction of the thickness of the oxides of the MOS capacitors, leading to a growing need for characterization techniques able to provide information at the nanometer scale on the thin oxides electrical properties.

Techniques derived from the Atomic Force Microscope (AFM) are good candidates for the nanoscale probing of the properties of oxides, since the (conductive) AFM tip can be used as a nanometric sensor to perform electrical measurements, taking advantage of the nanometric precision of the positioning of the AFM tip thanks to the piezoelectric ceramics controlling its motion.

Scanning Capacitance Microscopy (SCM, technique derived from the AFM) has been widely used for more than fifteen years for dopant mapping in semiconductor structures (a large literature exists which describes the performances and weaknesses of this technique. A general review can be found in reference [1]). In SCM mode, the conductive tip plays the role of the metal electrode in a nanometric Metal-Oxide-Semiconductor (MOS) structure, where either the underlying oxide or semiconductor is the element to be studied. In the former case, a semiconductor substrate with a constant doping level is chosen for the measurement. SCM measures the change of capacitance $\Delta C (\simeq 10^{-18} \text{ F})$ corresponding to an alternating voltage $V_0(t) = V_{AC} cos(\omega t)$ applied on the MOS structure.

SCM can also be used in a spectroscopic mode, called Scanning Capacitance Spectroscopy [2]. In this case, the tip is positioned on a fixed location on the surface while a voltage (superimposed on the alternating $V_0(t)$ voltage) is ramped and the ΔC signal is measured, leading to a ΔC -V curve which can be compared to the derivative of the classical Capacitance versus Voltage (C-V) curves obtained on macroscopic electrodes (usually, macroscopic measurements are made on square or circular electrodes several hundreds of microns large). SCS has already been used for the evaluation of the properties of the silicon dioxide used for SCM operation ([3,4]), to study charge trapping and trap creation in SiO₂ [5], or for the local characterization of the properties of very thin ZrO₂ films [6]. Theoretical studies have even been conducted in order to measure interface traps densities using the width of the SCS curves [7].

However, the SCS curves obtained this way are still qualitative and many experimental problems have to be solved before SCS can be considered as a metrological tool, because many experimental aspects play a major role which prevents SCS from being directly compared with macroscopic C-V curves. In this paper, we test the reliability of the SCS for oxide characterization by comparing the C-V curves measured by a classical impedance analyzer with the SCS obtained on the same samples and same electrodes. We emphasize the role of the tip–sample contact, which has never been taken into account (whereas the procedure used to deposit electrodes on clean oxide surfaces for macroscopic C-V measurements suggest that this point

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Fig. 1. SCS on a native oxide grown on a low doped p-type substrate $(10^{15} \text{ at. cm}^{-3})$ with and without laser. The SCS without laser has been obtained by displacing the laser along the cantilever axis.

should not be negligible at all), and appears to be one of the main limiting factors that prevents SCS to be entirely comparable to macroscopic C-V measurements. This study aims at being a step towards a better control of the nanometric measurements of the electrical properties of very thin dielectric oxides.

2. Experimental details: control of the signal to noise ratio of SCM

All measurements have been made with a Veeco Dimension 3100 apparatus equipped with a Nanoscope V controller. Using the AFM, the voltages are applied on the sample's back electrode while the tip is grounded, contrary to macroscopic *C–V* curves where the voltages are applied on the tip while the sample's back electrode is grounded.

Before comparing SCS with macroscopic *C–V* curves, it is important to clarify the origin of the SCS signal. As mentioned before, Scanning Capacitance Microscopy (SCM) does not measure the capacitance of a MOS structure directly but only the *variation* of the capacitance ΔC with respect to the applied voltage. Following Brezna et al. [8], it must be pointed out that the voltages applied in SCS are still far from the conditions of small signal required in the macroscopic setups (typically $V_{AC} = 20-50$ mV), since typical voltages used in SCS are in the 300 mV range in this study. If macroscopic *C–V* curves (and their derivative $\frac{dc}{dV} - V$) are to be compared with SCS, it is important that the applied voltages are as low as possible to avoid any artificial averaging effects masking important features of the curves. That's why a systematic optimisation of the signal to noise ratio of SCS must be performed.

Doped diamond-coated tips (stiffness in the 1-10 N/m range) have been used in this work: frequently encountered for dopant mapping, they allow to work with low applied alternating voltages thanks to their large radius, and provide a superior resistance to wear, which is an important property for the reproducibility of the measurements [9]. The use of *PtIr*₅-coated tips, as often encountered in the literature [5,10–12], leads to the application of far higher voltages for the contrast to be acceptable (up to 2 V [13]), which has to be avoided in the light of the discussion of the previous paragraph, especially when the spatial resolution is not the most important parameter of the measurement.

In order to maximize the SCS signal, and considering the fact that it is inversely proportional to the doping level of the semiconductor, all measurements in this paper have been conducted on low doped $(10^{15} \text{ at. cm}^{-3})$ silicon p-type substrates covered by a thin SiO₂ oxide. The low doping level of the substrate implies that the influence of the laser light (needed for the measurement of the deflection of the AFM cantilever, and thus for the topographic mode of the AFM) is strictly

controlled because of the generation of electron-hole pairs which strongly influences the SCS signal [14,15]. Fig. 1 illustrates this point: SCS is operated on a native oxide with and without the presence of the AFM laser light. The difference between the resulting SCS measured in both conditions emphasizes the decrease of the signal to noise ratio due to the electron-hole pairs generation originating from the impact of the laser light on the surface. It must also be pointed out that the generation of electron/holes pairs by the laser light has also created an additional peak due to minority carriers in the negative voltage region corresponding to the inversion zone.

One solution to minimize this influence is to use long cantilevers and displace the laser along the axis of the cantilever so that the laser is located far (several tens of microns) from the point where the tip touches the surface (for 10^{15} at. cm⁻³, the minority carrier diffusion length in silicon is in the 5 µm range [16]: the pairs generated by the laser should recombine before reaching the point where the SCS is performed). For the final positioning of the laser, a compromise must be found between the topography (laser must be reasonably at the end of the cantilever) and the SCS measurements (the laser must be moved away from the tip area). Moving the laser away from the cantilever end decrease the sensitivity of the measurement of the deflection, which may lead to higher forces applied to the sample surface. Combined with the relatively high stiffness of the cantilevers used for SCM measurements, engraving of the oxide becomes a more probable risk.

The use of diamond tips and the control of the influence of the laser allow to use typical V_{AC} as low as 300 mV for SCS measurements, which is still not as low as the voltages used for macroscopic C-V curves.

3. Study of the tip-sample contact

When macroscopic C-V curves are used to test the quality of a thin oxide layer, a metal electrode is deposited (e.g. Nickel–Gold or Aluminium electrodes) so that the interface between the electrode and the oxide layer is perfectly clean. When it is not the case, abnormal C-V curves can be recorded, showing e.g. an increase of the capacitance signal in inversion, in a region where no minority carriers should be detected when a alternating voltage of sufficiently high frequency is applied. Fig. 2 shows an example of C-V curve obtained on a 5 nm thick thermal oxide (which will be used throughout the rest



Fig. 2. *C*–*V* characteristic obtained on 100 μ m × 100 μ m Ni–Au electrodes on a thermal 5 nm thick SiO₂ oxide (applied alternating voltage: 100 kHz, *V*_{AC}=50 mV). Insert: Example of electrodes used for macroscopic *C*–*V* curves, grown on a 5 nm thick thermal oxide. The smallest electrode size is 50 μ m × 50 μ m.

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