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with Local Electrode Atom Probe**

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# Abstract

Hafnium-based high-k dielectrics are currently replacing the SiON gate in CMOS transistors due to their high dielectric constant (high-k), large bandgap and high thermodynamic stability with Si. The basic capability of atom probe tomography (APT) to analyze this type of material with good spatial resolution has been presented in the last few years. However, little has been said about the reproducibility and the measurement yield. Major questions like reproducibility and reliability of the measurements and possible measurement artifacts have hardly been discussed when this work was started. The main focus of the work presented here is to analyze high-k and metal gate materials and to describe the procedures necessary to obtain reliable and reproducible results with high spatial resolution and high measurement yield. Using these procedures, APT has a high potential of being widely used in the semiconductor industry on a daily basis.

The developed procedures in the following areas are presented: specimen preparation, cap layer engineering, measurement parameters and data analysis.

Specimen preparation by focused ion beam (FIB) for APT analysis is an important step to increase the reproducibility of APT measurements and the measurement yield. Procedures to obtain operator-independent tips are described in detail.

In order to protect the region of interest during FIB specimen preparation, cap layers are deposited via electron beam deposition. Different material properties like adhesion, number of isotopes, evaporation field and the sputtering rate in the FIB are investigated and their influence on the results is presented. Due to the introduction of the cap layer stack Cr / Si / Ni, high measurement yield can be achieved.

To obtain meaningful results, measurement parameters in the APT like the laser energy or the specimen temperature have to be chosen carefully. The influence of the specimen temperature is very small, whereas the high laser energy causes diffusion of the analyzed material. Using low laser energies leads to reproducible results.

Data treatment is the final important step to obtain correct results. Background subtraction of the random noise is described and the influence on the chemical composition and the depth scale is discussed. By applying the background subtraction and depth scale correction, reproducible results are obtained.

The reconstruction procedure in APT and its limitations are summarized and in measurements of boron delta layers deviations from the currently used reconstruction model are observed and discussed.

After the investigation of these procedures, their application to a number of different high-k and high-k / metal gate samples is presented. The comparison of APT results with different analysis techniques proves the high reproducibility of APT. For the analysis of 2 nm thick HfO<sub>2</sub> / ZrO<sub>2</sub> layers sub-nm depth resolution, correct chemical composition and high measurement yield are achieved. Different high-k samples, deposited with Metal Organic Chemical Vapor Deposition (MOCVD) and Atomic Layer Deposition (ALD), are analyzed and good reproducibility with high measurement yield is obtained. More complex material stacks consisting of HfO<sub>2</sub> and metal gate (TiN), deposited on silicon or silicon / germanium are analyzed and sub-nm depth resolution with correct chemical composition is achieved.