Complementary Metrology – A Prerequisite for Reliable and Traceable Characterization of Surfaces and Nanolayers

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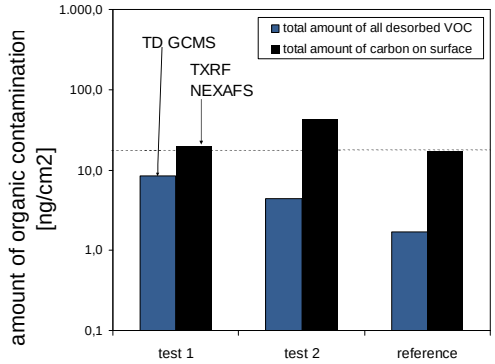
PTB X-ray and IR Spectrometry

Concepts of Complementary Metrology

Recently, complementary metrology was studied in a distributed joint laboratory for characterization of samples from nano electronics. In a first step the analytical tasks or samples were defined with respect to possible measurands. Secondly, the analytical capabilities were determined. Based on these two criteria a matrix was set up which assigns each measurand analytical techniques. The complementarity of the metrology could be achieved by considering a measurand and its associated analytical techniques. The methods benefit from each other by supporting the information on e.g. thicknesses or density that cannot be obtained but are required by other methods.

Assessment of Carbon on Surfaces

Reliable quantification of surface carbon contamination was achieved using complementary analysis of samples from wafer manufacturing using TD GCMS for the detection of volatile organic compounds and TXRF-NEXAFS for the.



Benchmarking of ToF-SIMS, TD-GCMS, and TXRF-NEXAFS for carbon analysis

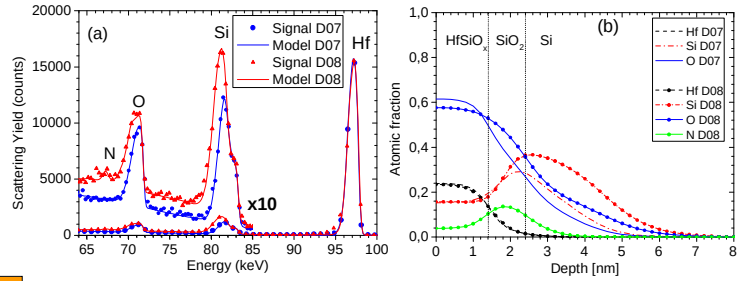
Comparison of analytical techniques TOF-SIMS, TDGCMS and TXRF-NEXAFS for characterization of organic contamination on semiconductor surfaces.

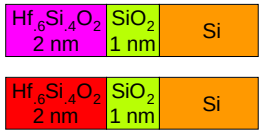
Method	TOF-SIMS	TD-GCMS	TXRF-NEXAFS
probing conditions	vacuum; beam raster of 200 x 200 µm²	atmosphere whole wafer	Vacuum spot of 70x140 µm² projected on the wafer
detection of volatile organics	limited	strength: detection of VOC	limited
detection of non-volatile Compounds	strength: detection of non-volatile organics	not possible	strength: determination of total carbon amount on surfaces
identification of organic compounds	limited due to method and database	strength: identification using a database	limited to specific lines e.g. Br etc.
quantification	difficult	semi quantitative to n-hexadecane	reference-free

Characterization of Nano Layers

Accurate structural and compositional characterization of high-k hafnium silicate (HfSiOx) layers enables an understanding of their properties and the ability to control these. Those high-k layers were used to compare Medium Energy Ion Scattering (MEIS) with Spectroscopic Ellipsometry (SE), X-ray Photoelectron Spectroscopy (XPS), and high resolution Transmission Electron Microscopy (XTEM) and reference-free synchrotron radiation based X-Ray Fluorescence (XRF) analysis

Medium Energy Ion Scattering





Sample D 07

Sample D 08

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wafer prepared by IMEC in Leuven (Belgium)

Comparison of the layer thickness measurements for the samples D07 and D08 using different analytical techniques. Results are given for two different densities of the silicate layers.

Sample	Layer / density [g cm^{-3}]		$d_{nominal}$ [nm]	MEIS d [nm]	SE d [nm]	XPS d [nm]	XRF d [nm]	XTEM d [nm]
D07	SiO_2	2.2	1	~0.8 ± 0.2	(.5) + 0.73	1.0 ±0.2		1.6 ±0.4
	$HfSiO_x$	6.1	2	1.5 ±0.1	2.1-2.5	1.9 ±0.1	1.5±0.2	1.3 ±0.3
	$HfSiO_x$	6.7		1.4 ±0.1		1.8 ±0.1	1.4±0.2	
D08	SiO_2	2.2	1	~1.3 ±0.2	(.5) + 0.94	1.2 ±0.1		2.7 ±0.3
	$HfSiO_x$	6.1	2	1.75 ±0.1	2.2-2.3	1.9 ±0.1	1.6±0.2	1.4 ±0.4
	$HfSiO_x$	6.7		1.6 ±0.1		1.8 ±0.1	1.5±0.2	

Conclusion

The use of complementary metrology as well as of reference samples is crucial for the advancement of analytical methodologies.
The combination of TD-GCMS, TOF-SIMS, and TXRF-NEXAFS allows for the characterization of surfaces with respect to all organic compounds. The combination of methods compensates for the limitations due to measurement conditions, e.g. localized measurement or vapor pressure and boiling point of organic compounds.
The use of various analytical techniques for the analysis of nano layers was found to substantially improve the reliability and validation of the achieved results. This was demonstrated using approx. 2 nm thick high-k layers. Such a well-characterized sample is ideal as a reference sample for various purposes.

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