Ultra-trace analysis of light elements and speciation of minute organic contaminants on silicon wafer surfaces by means of TXRF in combination with NEXAFS

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Total reflection X-ray fluorescence spectroscopy (TXRF)[1-3] using high flux synchrotron radiation (SR) for the specimen excitation is a powerful technique for the non-destructive analysis of very small amounts of both metal and low Z elements on silicon wafer surfaces. Several different TXRF experiments were conducted at the plane grating monochromator (PGM) beamline[4] for undulator radiation within the radiometry laboratory[5] of the Physikalisch-Technische Bundesanstalt (PTB) at the electron storage ring BESSY II, which provides photon energies between 0.1 keV and 1.9 keV for the specimen excitation. The PGM beamline is also very suitable for the standard XRF analysis of thin multi-elemental, multi-layered structures deposited on silicon surfaces.

The lower limits of detection (LOD) of TXRF analysis were investigated for some low Z elements[6,7] such as C, N, Na, Mg and Al in two different detection geometries for various excitation modes. The absolute LOD values for these light elements ranged between 0.3 pg and 1.3 pg. The calculated vapor phase decomposition (VPD) detection limits, derived from the assumption that the sample droplets would have been collected from a 200 mm wafer, range from 2 10^7 atoms / cm^2 to about 10^8 atoms / cm^2 for Na, Mg and Al, thus fulfilling current analytical requirements of the semiconductor industry. For the sake of an application-oriented TXRF approach, droplet samples on silicon wafer surfaces were prepared by Wacker Siltronic and investigated in the TXRF irradiation chamber of the Atominstitut (ATI) and the ultra-high vacuum TXRF irradiation chamber of the PTB.

Employing a new deconvolution technique for TXRF spectra using experimental detector response functions instead of conventional peak fitting functions, it could even be shown that, in the very soft x-ray range, no relevant continuous scattering or Bremsstrahlung background exists when exciting the wafer samples with radiation of high spectral purity.

Using a thin window 30 mm² Si(Li) detector of the Technical University Berlin, the LOD value of B and the resonant Raman scattering effect were studied in the ATI chamber. A LOD value of 7 ng could be stated for B. Varying the angle of incidence, TXRF investigations of a 5 nm C layer and of a 1.6 nm C – 2.1 nm Ni – 1.6 nm C multilayered structure were performed in the soft X-ray range allowing the respective layers to be characterized.

Organic contaminants are starting to play an important role in the production and quality control of Si wafers. For the traceability of the source of contamination, information on the chemical surroundings is very valuable. By tuning the photon energy provided by the plane grating monochromator around the K-shell absorption edges of C, N and O, minute amounts of low Z and organic contaminants deposited on silicon wafers were investigated in the PTB chamber combining TXRF analysis with the near edge X-ray absorption fine structure (NEXAFS) method. The experimental results will be presented and the reproducibility of the measurements, as well as different approaches for the data evaluation, will be discussed.

Also to be presented is a novel instrumentation recently designed and constructed by the PTB for the explicit purpose of the semiconductor industry that fully utilizes the good TXRF and XRF excitation conditions at the PGM undulator beamline. 300 mm Si wafers, as well as 200 mm wafers, are transported directly from their respective shipping cassettes (FOUP, SMIF) via a pre-aligner into a high vacuum load-lock by an adapted commercial equipment front end module (EFEM). A vacuum robot, which is located inside the load-lock, takes the sample and places it inside the UHV analysis chamber onto an electrostatic chuck (ESC) mounted on an 8-axis-manipulator. To take advantage of the linear polarization of the synchrotron radiation, the ESC is moved into a vertical orientation. The manipulator allows the whole surface of a 200 mm or a 300 mm wafer to be scanned. To extend the capability of the system into the XRF region, the available angles of incidence are in the range from 0° to 45°. With respect to the alignment, an energy-dispersive detector, such as a Si(Li) or a superconducting tunnel junction detector, is mounted on a 3-axis stage. The load-lock is designed to reach a pressure of 10^{-7} mbar within one hour to allow transfer, whereas the base pressure of the analysis chamber is to be $5 \cdot 10^{-9}$ mbar. To speed up sample exchange, two wafers of any size can stay inside the load-lock and there is no need to interrupt the measurements during the pump down. The EFEM, including a class 1 mini-environment, is surrounded by a mobile class 100 environment to further reduce the risk of unintentional cross-contaminations of the samples.

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