

ANALYTICAL SERVICES

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Fraunhofer IPMS-CNT has established a versatile analytical facility. Our expertise ranges from wafer-level characterization with inline X-ray scattering methods and three-dimensional Atomic Force Microscopy to two- and three-dimensional device characterization with analytical transmission electron microscopy and atom probe tomography.

Our in-line metrology enables us to determine physical and chemical properties of structures on 300 mm wafers with x-ray diffraction, angle-resolved x-ray photoelectron spectroscopy, three-dimensional atomic force microscopy, spectral ellipsometry and energy dispersive x-ray spectroscopy. Furthermore, we have facilities that allow for automatic particle inspection, classification and characterization on wafer level. All our tools for wafer level analysis are stationed in a class 1000 cleanroom environment that meets industrial standards.

X-RAY
SCATTERING

RAMAN
SPECTROSCOPY

ATOM PROBE
TOMOGRAPHY

FOCUSED
ION
BEAM (FIB)

ELECTRON
MICROSCOPY

TIME-OF-
FLIGHT
SECONDARY
ION MASS
SPECTROMETRY

INLINE
METROLOGY

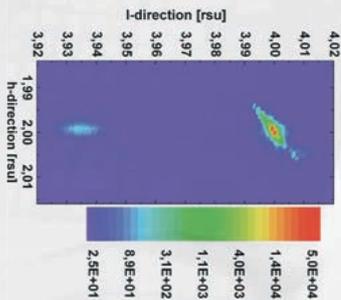
X-ray scattering (X-ray diffraction & X-ray reflectometry)

Bruker D8 Discover

X-ray scattering probes the arrangement of atoms in a sample by utilizing the interference of X-rays scattered at lattice planes or interfaces. It provides information about structural properties (e.g. crystallographic phases, lattice constants, degree of crystallization) and microstructural properties (e.g. grain size, preferred orientation, stress, film thickness, roughness, density). The penetration depth can be varied between a few nm and several μm . The sensitivity is about 1% phase content. The method allows for non-ambient measurements.

Epi layer characterization

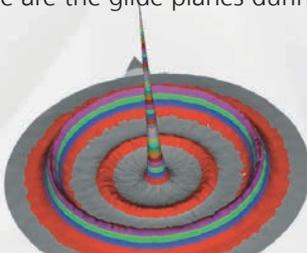
The thickness, composition and degree of relaxation of epitaxially grown SiGe layer were determined by high resolution X-ray diffraction. This kind of analysis can also be carried out inline on full 200mm and 300mm wafers.



Reciprocal space map of a SiGe (224) reflection showing that the layer is still pseudomorphically strained

Texture analysis of tungsten layers showing different CMP behavior

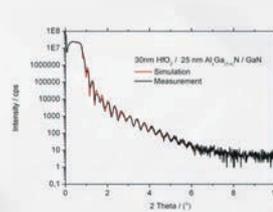
The texture of a tungsten layer was analyzed by recording its orientation distribution function (ODF). Differences in the recorded ODF allowed us to determine the reason for the Chemical Mechanical Polishing (CMP) behavior of these layers. We could show that the endpoint detection during CMP is inaccurate if the (110) planes parallel to the surface as these are the glide planes during polishing.



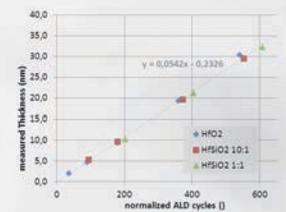
(110) Pole figure of a tungsten layer showing a pronounced (110) fiber texture leading to poor endpoint detection during CMP

Growth kinetics of Hf(Si)O₂ films

The thickness, density and roughness of ALD deposited HfO₂ films were determined by means of X-ray reflectometry (XRR) measurements to evaluate the growth kinetics and film quality.



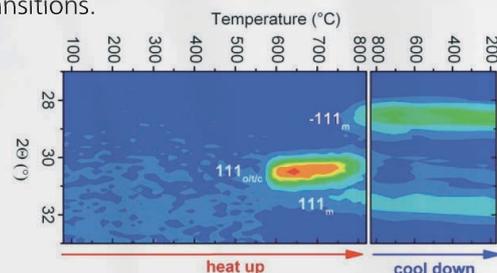
XRR analysis of Hf(Si)O₂



Growth kinetics of the Hf(Si)O₂ determined from several XRR analyses

Crystallization of thin ferroelectric films

The crystallization properties of a ALD grown ferroelectric HfSiO_x film on Si for FeRAM applications was investigated. These properties depend on a number of parameters, such as film thickness, substrate material or doping. X-ray analyses allow us to determine the temperature dependent crystal structures and the exact temperature of the phase transitions.



Temperature dependent crystal structure of a 10nm HfSiO_x film: The amorphous material crystallizes at ~550°C and forms a cubic phase. At ~800°C the phase changes into a monoclinic structure.

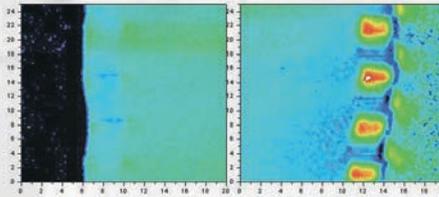
Raman Spectroscopy

Renishaw InVia Reflex

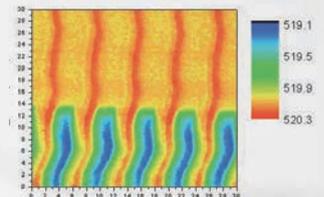
Raman Spectroscopy utilizes inelastic scattering of laser light to locally excite and image characteristic vibrational modes in a material. This scattering process involves the excitation or decay of characteristic vibrations of chemical bonds. As a result Raman spectroscopy can be used for the analysis of the orientation, phase and composition of a material as well as or lateral resolved stress and temperature mappings. Our tools allow for a lateral resolution down to 300nm. Different lasers allow us to vary the surface sensitivity, varying the integration depth from only a few nm to a few micrometer.

Stress maps of trenches (top down & cross section)

The stress distribution in different poly-Si filled trench structures has been studied using Raman spectroscopy in top down and cross section geometry.



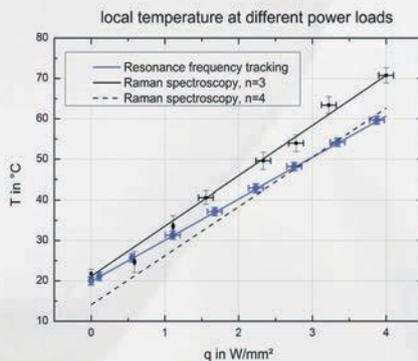
Raman maps of the cross sections of wide (left) and dense (right) trenches, showing areas of tensile and compressive stress.



Raman map of the structure from the top: showing the stress distribution parallel to the surface.

Laterally resolved temperature measurements

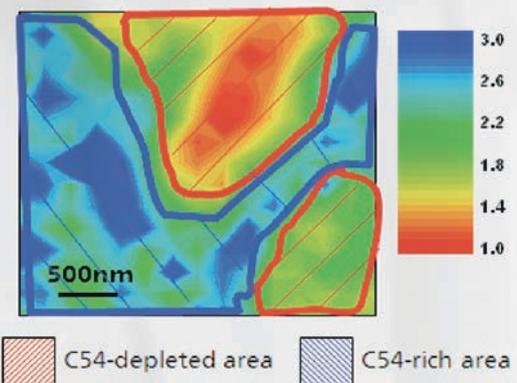
Raman spectroscopy was used to determine the absolute temperature in powered Surface Acoustic Wave devices with high lateral resolution.



Local temperature in a SAW device depending on the applied power

Lateral resolved phase analysis

Raman spectroscopy was used to record phase maps of TiSi_2 -based transistor contacts. TiSi_2 crystallizes in different structures (C54 and C49). Using Raman spectroscopy, the phase distribution can be mapped on a μm scale



Raman mapping of different TiSi_2 phases showing C54 depleted and enriched areas in the sample.

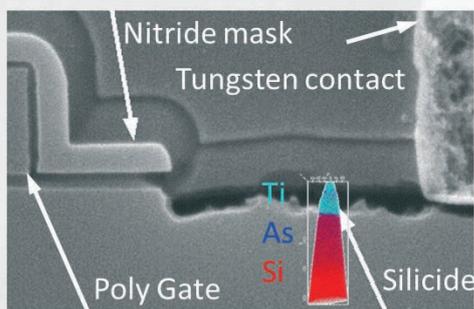
Atom Probe Tomography

LEAP 3000X Si

Atom probe tomography is based on the field evaporation of atoms from a very sharp nanometer-sized tip containing the region of interest of the sample. The method is used to generate 3D atomic maps that show the distribution of atoms in the sample including their elemental nature. This allows for the standard-less quantification of any sub-micrometer-sized volume within a sample. The spatial resolution is less than 1nm and the sensitivity reaches down to 1×10^{18} at/cm³ at best. Typical applications are studies of diffusion processes, the characterization of interfaces and the analysis of small precipitates or defects. It can also be used to analyze single devices such as transistors or capacitors.

Transistor construction analysis

APT was used to analyze the atomic make-up of a silicided contact in a DMOS-transistor. The method allows for quantification of all constituents in the region of the transistor.



APT analysis in the contact region of a DMOS-transistor showing the main constituents in the analyzed region.

Study of diffusion of Al in Cu

APT was utilized to study the diffusion of Al from a PVD Al-doped Cu layer into an ECP Cu layer after different annealing treatments. It can be shown, that Al atoms preferentially migrate along grain boundaries into the copper.

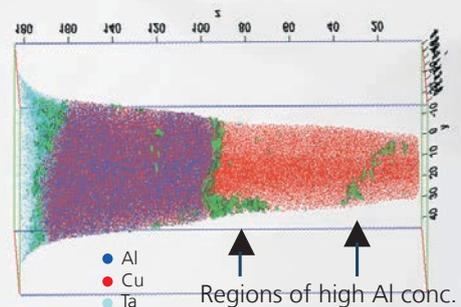
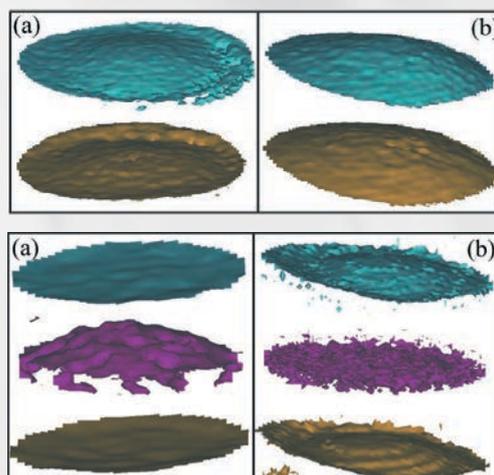


Image of a 3D atomic map showing diffusion of Al from Al-doped Cu on the bottom into the electroplated Cu on the top along the grain boundaries of the polycrystalline Cu layer.

Influence of radiation on AlInN/GaN HEMT structures*

APT was used to study AlInN/GaN and AlGaIn/GaN high electron mobility transistor structures before and after exposure to ⁶⁰Co irradiation. The analyses revealed that the interfaces in the AlInN/GaN structure are roughened by the radiation while the AlGaIn/GaN are not. Electrical measurements confirm that the AlGaIn/GaN structure is more radiation tolerant.



Interface roughness of the Al (blue) and Ga (yellow) interfaces in an AlGaIn/GaN HEMT structure before (left) and after (right) ⁶⁰Co irradiation exposure.

Interface roughness of the Al (blue), In (purple) and Ga (yellow) interfaces in an AlInN/GaN HEMT structure before (left) and after (right) ⁶⁰Co irradiation exposure.

*Dawahre et al., Journal of Vacuum Science & Technology B 31, 041802 (2013); doi: 10.1116/1.4807321

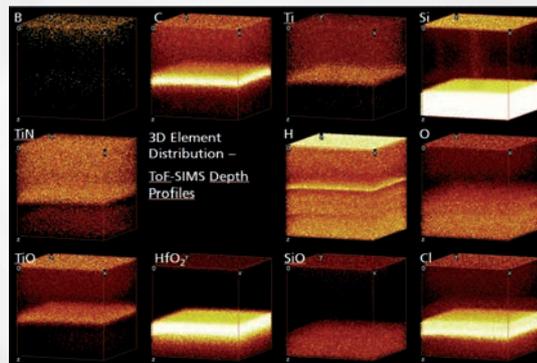
Time-of-flight Secondary Ion Mass Spectrometry

ION-TOF ToF-SIMS 300R

In Time-of-flight Secondary Ion Mass Spectrometry (ToF-SIMS) an ion beam is used to facilitate the emission of charged (secondary) ions from the sample surface. The mass of the emitted ions is analyzed using Time-of-flight mass spectrometry. As the ion beam creates a crater in the sample, the distribution of different species within the sample volume can be recorded. We can achieve a lateral resolution of a few hundred nanometers and a depth resolution of a few nanometers. In order to quantify the absolute concentration of the elements in the sample it is necessary to compare the analyses results to standards.

Analysis of a RRAM stack

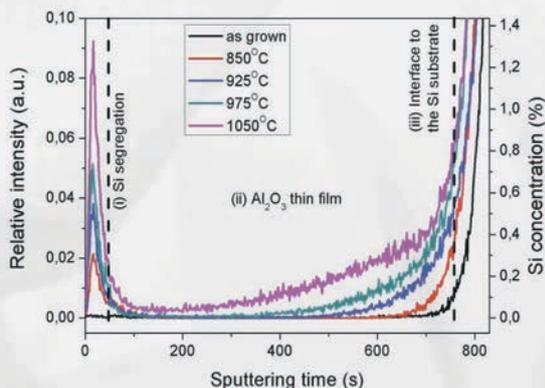
ToF-SIMS was utilized to study the 3D elemental distribution of a Ti/TiN/HfO₂ stack on a silicon substrate. The analysis revealed the formation of a TiO₂ layer on top of the HfO₂.



3D-distribution of different ions in a RRAM layer stack recorded by ToF-SIMS

Diffusion of Si in Al₂O₃

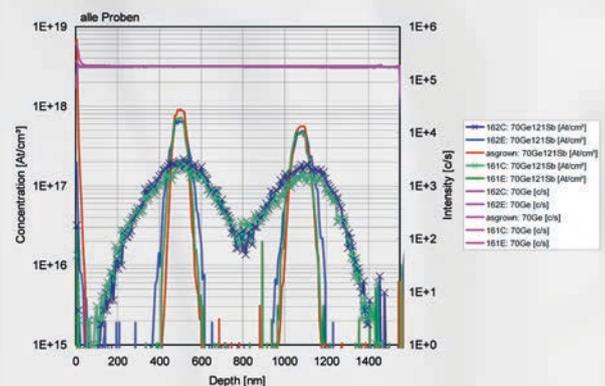
ToF-SIMS was used to study the diffusion of Si into ALD deposited Al₂O₃ in dependence of the temperature of the post-deposition anneal (PDA). The measurements make it possible to determine the diffusion constants and activation energies for silicon diffusion in crystalline and amorphous Al₂O₃.



SIMS profile shows, that Si content in Al₂O₃ increases with PDA temperature

Impact of proton irradiation on the Sb distribution in a Ge / Ge+Sb stack

ToF-SIMS was utilized to study the effect of irradiation on a layer stack consisting of an alternating sequence of 3 Ge layers and two Ge-Sb layers on a silicon substrate. The sample was irradiated with protons through a dot mask, so only the center was affected. The SIMS profiles show that Sb is diffusing into the neighboring Ge layer under the influence of proton irradiation and thermal activation.



SIMS depth profile of Ge/Ge+Sb layer stack, Sb profile of irradiation affected areas are marked with crosses

Electron Microscopy

FEI Tecnai F20/XT & Hitachi S5000

Electron microscopy uses an electron beam to illuminate a specimen and create a magnified image. Two different types of electron microscopes are available: scanning electron microscopes (SEM; resolution down to ~1nm) and transmission electron microscopes (TEM, resolution down to 0.1 nm). In SEM the electron beam is scanned over the sample and either the emitted secondary electrons or the back scattered electron are used for imaging the sample surface.

In TEM the electron beam is passed through a thin lamella containing the region of interest. The emerging beam carries information about the structure of the sample that can be evaluated in different ways. There are six different ways we can utilize the information created by the transmission of the electron beam: bright field imaging, dark field imaging, high angle annular dark-field scanning TEM (HAADF-STEM), Energy-dispersive X-ray spectroscopy (EDX), Electron energy loss spectroscopy (EELS) and Energy Filtered TEM (EFTEM).

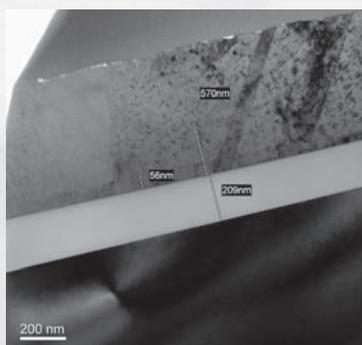
In bright field imaging the contrast is mainly formed by occlusion and absorption of electrons in the sample. Thicker regions of the sample, or regions with a higher atomic number will appear dark, whilst regions with no sample in the beam path will appear bright. In dark field imaging the unscattered beam is masked out and the image is formed by selected electrons that underwent (Bragg) scattering with the (crystalline) sample. This allows for imaging information related to the crystal, like orientation of crystals in polycrystalline materials or crystal defects.

In the HAADF-STEM mode the electron beam is focused onto a narrow spot and scanned over the sample. A high angle detector is used to form an image by the electrons that underwent incoherent scattering creating a contrast that is directly related to the atomic number of the imaged atoms.

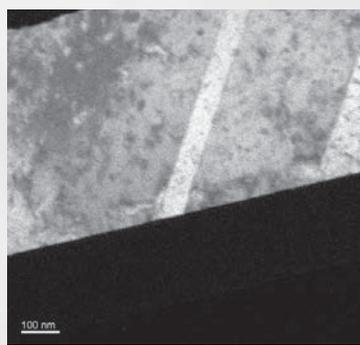
Finally, the (focused) electron beam used in (S)TEM can also be used for material analysis. The interaction of the electron beam with the material excites characteristic X-ray radiation that can be collected and used to characterize the material composition of the sample in EDX. Furthermore, some of the electrons will be inelastically scattered by the atoms in the material. The energy loss of the electrons is again characteristic for the material and hence can be used for material analysis in EELS or EFTEM.

EFTEM

The distribution of metal dopants in a layer stack of 50nm Mn doped Cu seed layer followed by 500nm electroplating of Cu was studied by means of EFTEM. It shows that Mn is enriched in a very thin slice.



bright field image



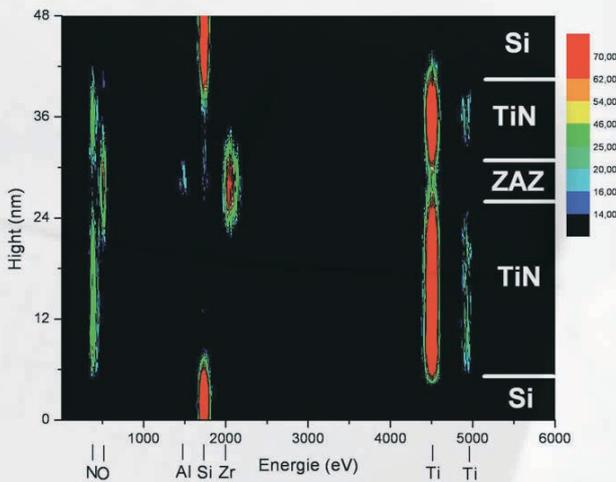
Cu mapping



Mn mapping

EDX profiling of thin film stacks

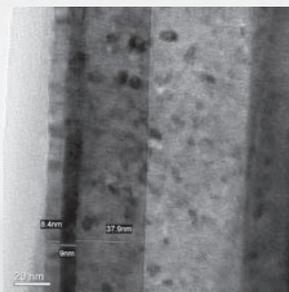
EDX was utilized to characterize the elemental distribution in a TiN/Al-doped ZrO₂/TiN MIM stack for DRAM capacitor applications.



EDX line scan of an MIM stack, the intensity of the element specific X-ray energy vs. depth is displayed.

HAADF-STEM

HAADF-STEM and bright field imaging was used to evaluate the step coverage of a HfO₂ layer within a MIM stack deposited in an array of staggered deep trenches. The HfO₂ appears dark in the BF image and light in HAADF-STEM image.



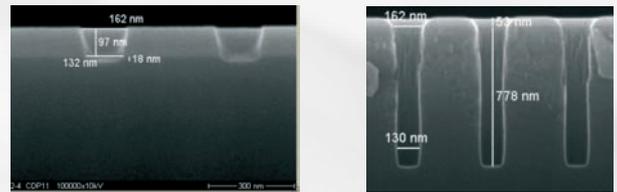
Bright field image of the MIM stack on the side wall of the trench: the HfO₂ layer is imaged as a dark layer.



Bright field image of the MIM stack on the side wall of the trench: the HfO₂ layer is imaged as a bright layer as it contains the heaviest atoms.

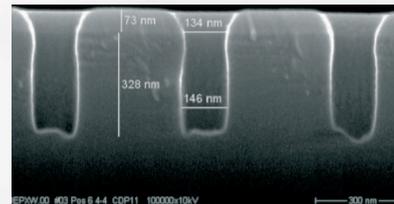
Evaluation of an etching process

SEM was used to determine the etch rates and etch profiles of a trench etching process.



pre etch

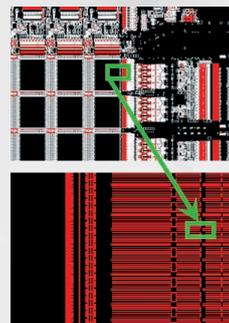
After 30s of etching



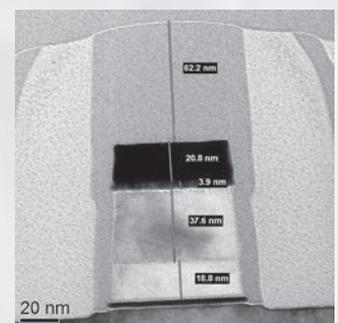
After 45s of etching

Physical failure analysis

TEM lamellas can be prepared site-specific to contain specific devices given a floor plan. A subsequent TEM analysis can identify the cause for a failure of the specified device.



Floor plan for device identification



TEM BF image of the gate stack of the specified NFET device

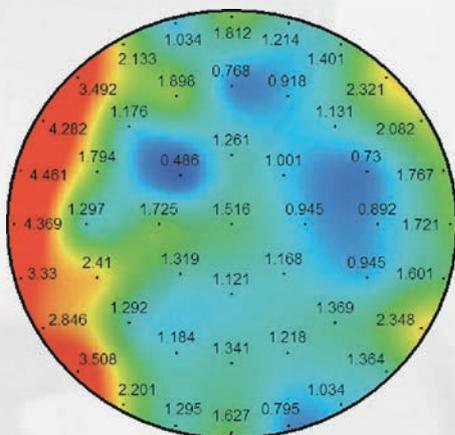
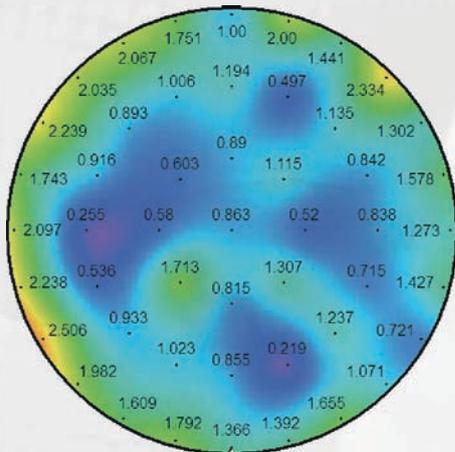
Inline metrology

Physical and chemical characterization of full wafers with high throughput without affecting the functionality of the wafer dies is a key to monitor the production of semiconductor devices. At Fraunhofer IPMS-CNT we have a number of different in-line metrology tools for the measurement of film thicknesses, sheet resistance, surface composition, chemical binding states, surface- and sidewall topographies and for defect inspection on 200 and 300mm wafers.

Surface composition wafer-mapping

ThermoFischer Theta300i ARXPS

The surface contamination of a 300mm wafer after different processing steps was quantified from X-ray photoelectron spectroscopy measurements.

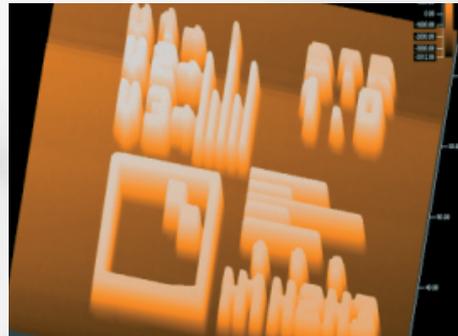


C-distribution over a full 300mm wafer after different process steps

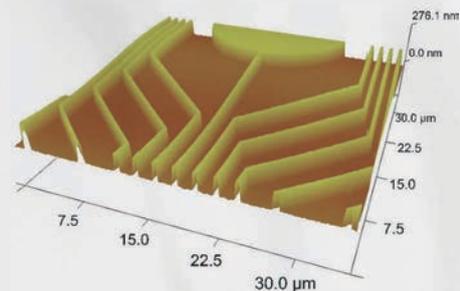
Profilometry and 3D-AFM

KLA Tencor HRP340 & Bruker Nano X3D

The surface profile of a horizontally and vertically aligned test structures can be determined with high resolution using a profiler or an 3D-Atomic Force Microscope.



Surface profile of a test pattern recorded with a high resolution profiler

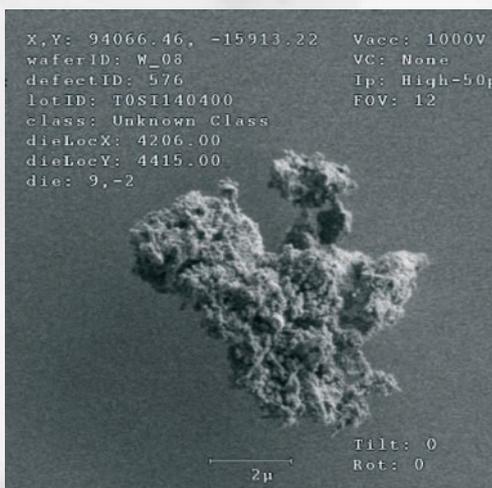
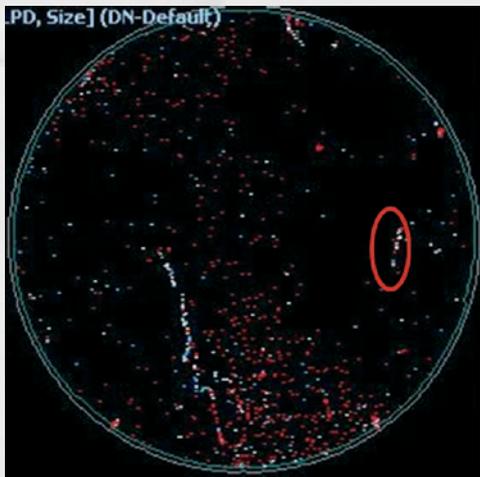


Surface profile of a test pattern recorded with 3D Atomic Force Microscopy

Defect inspection

KLA Tencor SP2 SurfScan &
Applied Materials G3 SEMVision

The automatic mapping and classification of the distribution of defects such as particles can be carried out in our SP2 tool. The tool provides a data format that can be read by a review SEM for closer defect inspection including EDX material analysis.

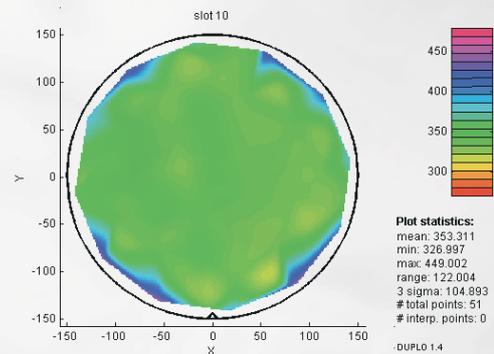


SP2 map of a blanket wafer after handling test (top) and image of a particle from the red circled region of the wafer (bottom)

Film thickness wafer-mapping

KLA Tencor Spectra FX100

The homogeneity of the layer thickness of a spin coated 350nm thick ZrO_2 layer over a 300mm wafer was checked by spectral ellipsometry. The mapping revealed that the layer thickness increases at the edge of the wafer.

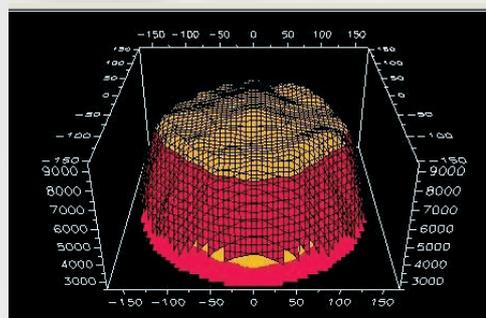


Thickness map of a spin coated thick ZrO_2 film

Sheet resistivity

EURIS WS3000 & KLA Tencor RS100

The sheet resistivity of a metal layer deposited on a 300mm wafer was mapped over by four-point electrical probing in order to probe the homogeneity of the deposited metal layer.



Sheet resistance of a blanket metal layer on a 300mm wafer

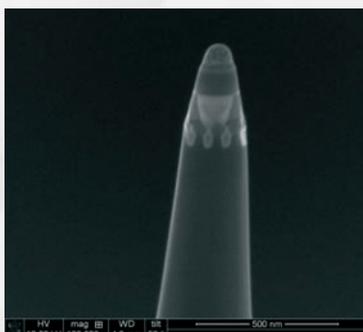
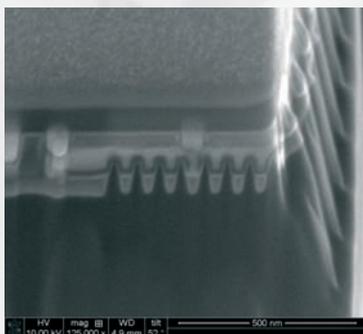
Focused Ion Beam (FIB)

FEI Strata 400

Focused Ion Beam is an essential tool in modern physical failure analysis. A finely focused ion beam allows for precise cutting into a sample. This tool is indispensable for the site-specific preparation of TEM lamellae and APT tips. FIB tools nowadays are usually dual-beam machines equipped with both an ion beam column and an electron beam column; hence images with electrons and ions can be taken in parallel. In addition our tool is equipped with a micromanipulator and a Platinum - as well as a Carbon Gas Injection System allowing for local deposition of Platinum and Carbon respectively. As a result the apparatus can furthermore be utilized for nanolithography and circuit modification.

Preparation of tip-shaped samples for atom probe tomography

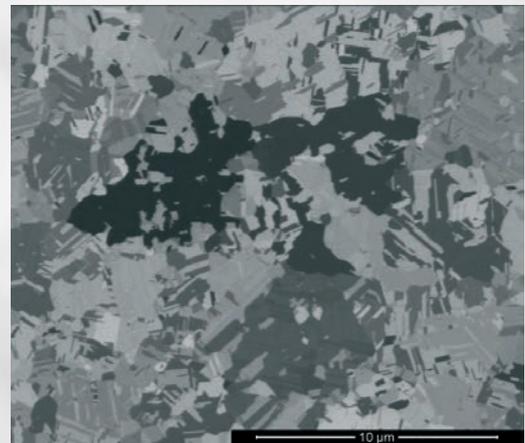
The preparation of site-specific samples for atom probe tomography analysis of nano-devices is not possible without focused ion beam technique. Different steps are necessary to prepare a nanometer sized tip comprising e.g. a transistor device. Initially the region of interest is cut- and lifted out from the wafer, then placed on top of a suitable post and finally milled into a tip. The entire process can be carried out placing the final tip with sub-10nm precision.



Original sample and final ready-to-run APT tip comprising a transistor device

Ion Beam Imaging

The ion beam imaging mode that the FIB offers allows for highlighting certain material properties. In particular it is frequently used for imaging the grain structure of Cu metallization layers.



Cu grain structure of electroplated Cu on a silicon wafer