



# Analytical services

---

Our in-line metrology enables us to determine physical and chemical properties of structures on 300 mm wafer with X-ray diffraction, angle-resolved X-ray photoelectron spectroscopy, spectral ellipsometry and energy dispersive X-ray spectroscopy. All our tools for wafer level analysis are stationed in a class 1000 (class 6 ISO 14644-1) cleanroom environment that meets industrial standards. A huge number of analysis is additionally available in the physical failure analysis labs. Our well established staff offers wafer analysis with X-ray (XPS, XRD/XRR, TXRF), electron microscopy (SEM, FIB, EBSD/TKD, TEM, EFTEM) and many more methods (AFM/PFM, FTIR, Raman, chemical). Electrical characterization complements the portfolio of products and services.

X-RAY  
SCATTERING

X-RAY  
PHOTOELECTRON  
SPECTROSCOPY

TIME-OF-  
FLIGHT  
SECONDARY  
ION MASS  
SPECTROMETRY

ELECTRON  
MICROSCOPY

FOCUSED  
ION  
BEAM

INLINE  
METROLOGY

RAMAN  
SPECTROSCOPY

## Contact

---

Dr. Nora Haufe  
+49 351 2607 3020  
[screening@ipms.fraunhofer.de](mailto:screening@ipms.fraunhofer.de)

Fraunhofer Institute for  
Photonic Microsystems IPMS

Center Nanoelectronic Technologies  
An der Bartlake 5  
01109 Dresden  
Germany

[www.ipms.fraunhofer.de](http://www.ipms.fraunhofer.de)

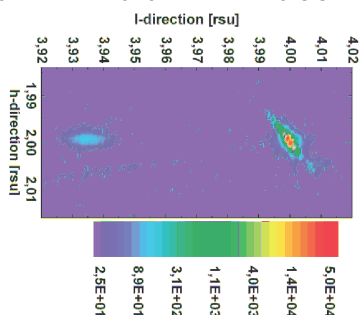
# X-RAY SCATTERING

(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  $\mu\text{m}$ . The sensitivity is about 1% phase content. The method allows for non-ambient measurements.

## ■ EPI LAYER CHARACTERIZATION

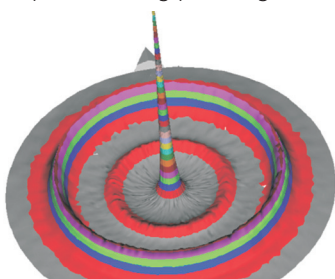
The thickness, composition and degree of relaxation of epitaxially grown SiGe layer were determined by high resolution X-ray diffraction. This kind of analysis can also be carried out inline on full 200 mm and 300 mm wafers.



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

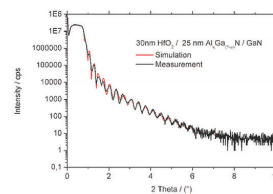
The texture of a tungsten layer was analyzed by recording its orientation distribution function (ODF). Differences in the recorded ODF allowed us to determine the reason for the Chemical Mechanical Polishing (CMP) behavior of these layers. We could show that the endpoint detection during CMP is inaccurate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



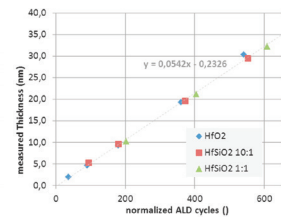
Pole figure of a tungsten layer showing a pronounced (110) fiber texture leading to poor endpoint detection during CMP

## ■ GROWTH KINETICS OF HF(SI)O<sub>2</sub> FILMS

The thickness, density and roughness of ALD deposited HfO<sub>2</sub> films were determined by means of X-ray reflectometry (XRR) measurements to evaluate the growth kinetics and film quality.



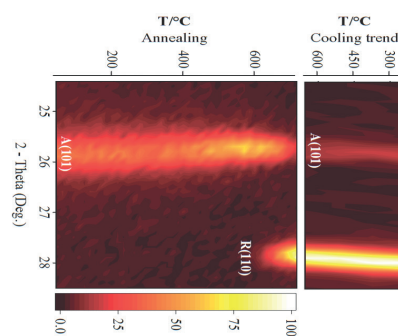
XRR analysis of Hf(Si)O<sub>2</sub>



Growth kinetics of the Hf(Si)O<sub>2</sub> determined from several XRR analyses

## ■ CRYSTALLIZATION OF TiO<sub>2</sub> THIN FILM

The crystal transformation of an ALD grown TiO<sub>2</sub> film on the 35 nm TiN was investigated. The crystal structure of the sample is monitored during annealing with HT-XRD. The as-deposited anatase phase (101) changes from 600 °C; the rutile phase (110) starts to form, and the intensity of the anatase phase decreases. Using TiN as substrate affects the nucleation process by changing the chemical characteristics of interfaces.



Phase change during annealing

# X-RAY PHOTOELECTRON SPECTROSCOPY

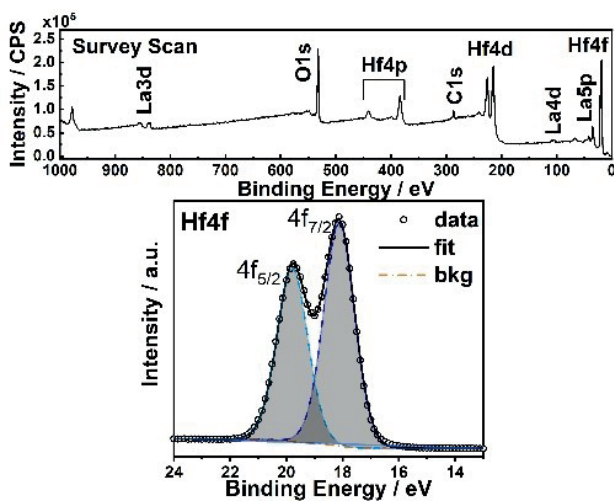
PHI QUANTES SCANNING XPS/HAXPES MICROPROBE

X-ray photoelectron spectroscopy is a quantitative technique that probes the chemistry of a material. When the X-ray source impinges a sample, electrons are excited by the photoelectric effect. The energies of the photoelectrons ejected are analyzed to obtain information on the chemical state and elemental composition of a sample. We offer a unique lab-based combination of monochromatic X-ray sources: a soft X-ray source (Aluminium  $K\alpha$ ) and a high energy X-ray source (HAXPES using Cr  $K\alpha$ ) for a wider range of analysis needs. The Cr  $K\alpha$  source offers a wider measurement range and a deeper analysis depth of about 3 times larger than with the Al  $K\alpha$  source.

The X-ray excitation sources' beam sizes can be focused between 7 and 200  $\mu\text{m}$  in diameter, giving way to microprobe analysis where points, lines, and mapping areas can be defined. Angle and sputter profiling depth analysis determines material composition across layer stacks or bulk material. Sample imaging using the X-ray sources is possible to create SEM-like images for the analysis of structured and inhomogeneous surfaces. In addition, in-situ XPS temperature dependent measurements can be performed in the range:  $-120\text{ }^\circ\text{C}$  to  $+300\text{ }^\circ\text{C}$ .

## ■ DETERMINATION OF COMPOSITION

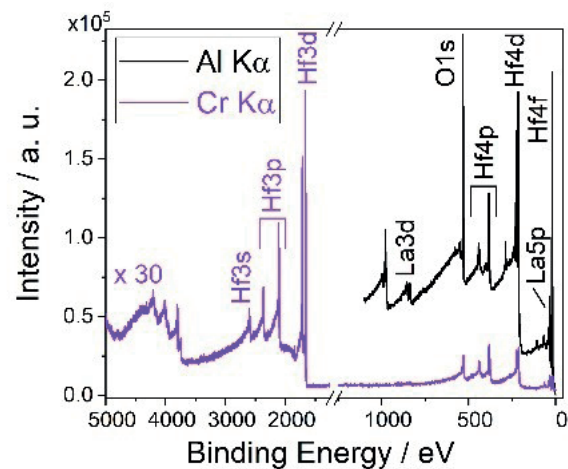
Peak identification and fitting gives information about what elements are present, the oxidation state, chemical environment and concentration of the elements. This is used to conclude material composition.



XPS survey spectrum (top) of La containing  $\text{HfO}_x$  thin film showing elemental peaks and corresponding Hf4f region showing peak fit (bottom)

## ■ DUAL MONOCHROMATIC EXCITATION: WIDER SPECTRAL RANGE

The presence of both the soft and high energy X-ray sources is complementary. With the HAXPES feature, a wider spectral range to higher binding energies can be measured. The high energy Cr  $K\alpha$  excitation source probes core electronic levels that are not possible to irradiate and measure using the conventional Al  $K\alpha$  source. In addition, the two X-ray sources are used to resolve peak overlap.



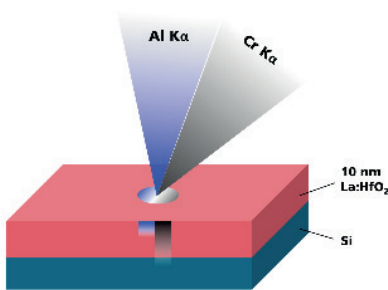
XPS survey spectra of La containing  $\text{HfO}_x$  thin film using Al  $K\alpha$  and Cr  $K\alpha$ , additional photoelectron lines at higher binding energies by HAXPES feature

# X-RAY PHOTOELECTRON SPECTROSCOPY

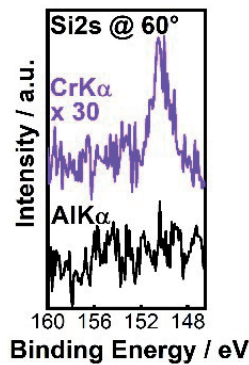
PHI QUANTES SCANNING XPS/HAXPES MICROPROBE

## ■ DUAL MONOCHROMATIC EXCITATION: ANALYSIS DEPTH

The Cr K $\alpha$  X-ray source probes 3 times deeper through a material than the Al K $\alpha$  source. Both sources allow for the measurement of single and multilayer stacks of different thicknesses, as well as buried interfaces. Using Cr K $\alpha$ , depth profiling can be done without the need to use ion beam sputtering, which helps avoid ion-based material degradation.



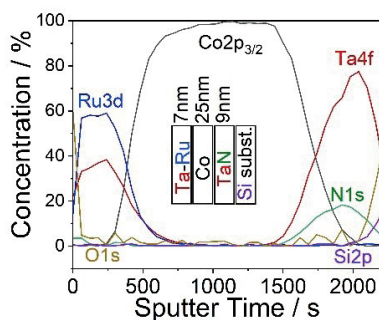
Differential analysis depth with Al K $\alpha$  and Cr K $\alpha$  (HAXPES)



High resolution scans of the Si<sub>2s</sub> region for a La containing HfO<sub>x</sub> material at a take-off angle of 60°, substrate detection exclusively with Cr K $\alpha$

## ■ DEPTH PROFILE ANALYSIS: SINGLE AND MULTILAYER STACKS

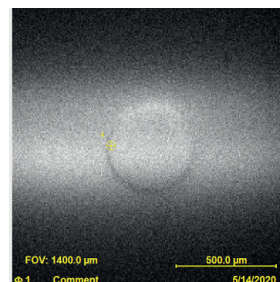
Both angle dependent and argon ion sputter depth profiling can be performed with Al K $\alpha$  and Cr K $\alpha$ . A range of take-off angles are available and ion gun voltages can be tuned. Depth profiling provides information about elemental diffusion and composition throughout a bulk material or multilayer stacks.



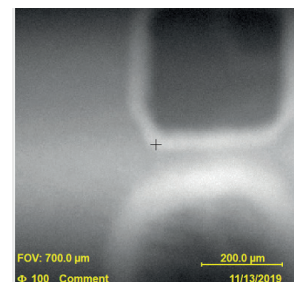
Argon sputter depth profiling of a multilayer stack (diagram in inset) using Al K $\alpha$  at a 45° take-off angle

## ■ X-RAY INDUCED SECONDARY ELECTRON IMAGING (SXI) FOR THE ANALYSIS OF STRUCTURED SAMPLES

Imaging of structured and inhomogeneous samples can be done using the X-ray sources. Fast real-time imaging produces SEM-like images for point, area, and line analyses. The same optics are used for the collection of the SXI and XPS analysis, which means that data is collected from the chosen point / area of interest.



Capacitor structure with a defined measurement point (yellow cursor)



Sputter analysis area from a TOF-SIMS measurement (top) and contamination area (bottom)

# TIME-OF-FLIGHT SECONDARY ION MASS SPECTROMETRY

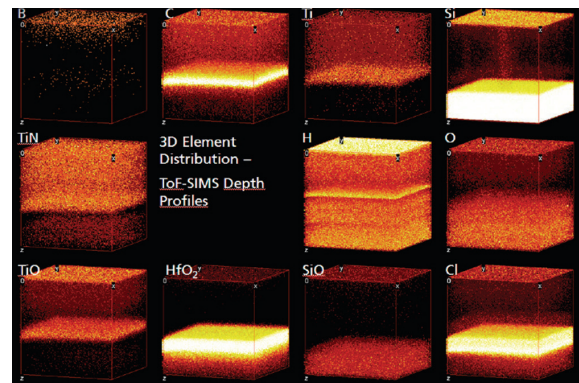
ION TOF TOF.SIMS 300R

In time-of-flight secondary ion mass spectrometry (ToF-SIMS), a primary ion beam is used to produce monatomic and polyatomic particles (secondary ions) from the sample surface. The technique used to characterize the surface and sub-surface region of materials based on  $m/z$  ratio measurement of ejected particles under ion bombardment. The mass of the emitted ions is analyzed using a mass spectrometer. 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 monolayers. In order to quantify the absolute concentration of the elements in the sample, it is necessary to compare the analysis results to standards.

## ANALYSIS OF A RRAM STACK

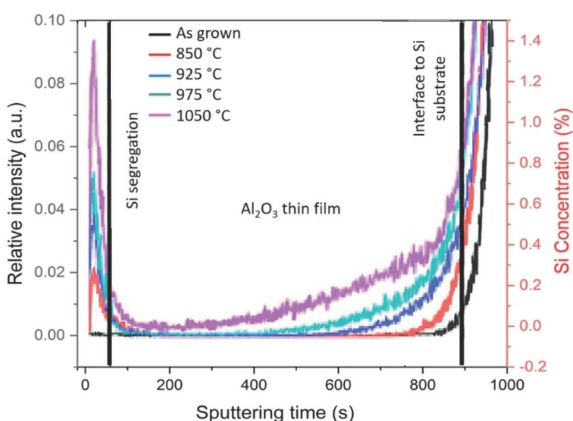
ToF-SIMS was utilized to study the 3D elemental distribution of a Ti/TiN/HfO<sub>2</sub> stack on a silicon substrate. The analysis revealed the formation of a TiO<sub>2</sub> layer on top of the HfO<sub>2</sub>.

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



## DIFFUSION OF SI IN AL<sub>2</sub>O<sub>3</sub>

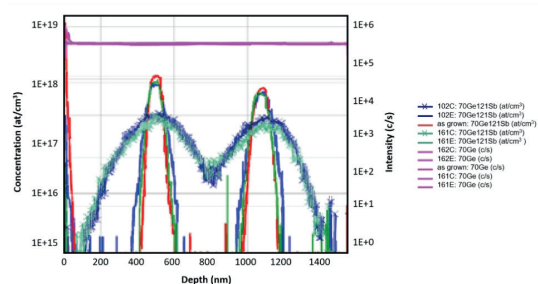
ToF-SIMS was used to study the diffusion of Si into ALD deposited Al<sub>2</sub>O<sub>3</sub> in dependence of the temperature of the post-deposition anneal (PDA). The measurements make it possible to determine the diffusion constants and activation energies for silicon diffusion in crystalline and amorphous Al<sub>2</sub>O<sub>3</sub>.



SIMS profile shows, that Si content in Al<sub>2</sub>O<sub>3</sub> 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

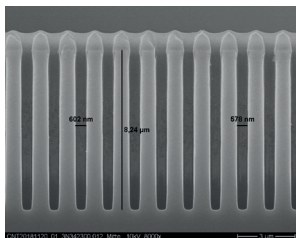
# ELECTRON MICROSCOPY

THERMO FISHER APREO S, HITACHI S5000, FEI TECNAI F20

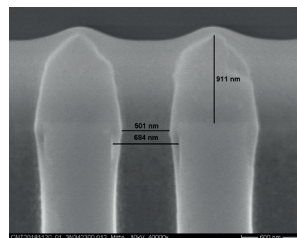
Electron microscopy uses an electron beam to illuminate a specimen and create a magnified image. Two different types of electron microscopes are available: scanning electron microscopes (SEM, resolution down to ~1 nm) and transmission electron microscopes (TEM, resolution down to 0.1 nm). In SEM the 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).

## ■ EVALUATION OF AN ETCHING PROCESS

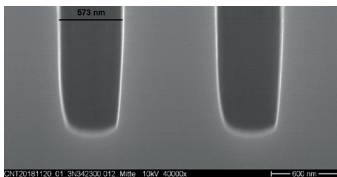
SEM was used to determine the etch rates and etch profiles of a trench etching process.



Overview with trench depth



Detailed top



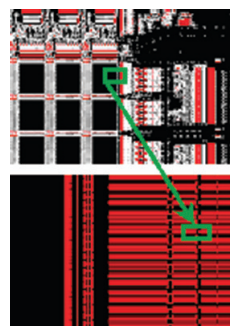
Detailed bottom

## ■ HAADF-STEM

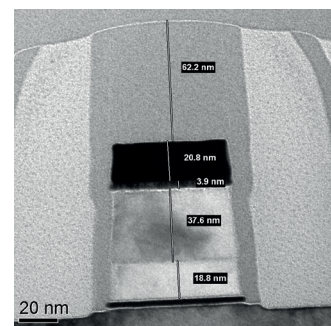
HAADF-STEM and bright field imaging was used to evaluate the step coverage of a  $\text{HfO}_2$  layer within a MIM stack deposited in an array of staggered deep trenches. The  $\text{HfO}_2$  appears dark in the BF image and light in HAADF-STEM image.

## ■ PHYSICAL FAILURE ANALYSIS

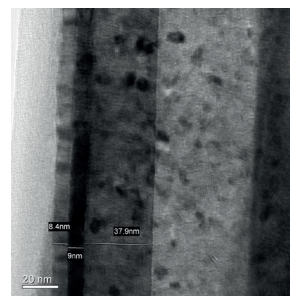
Site-specific TEM lamellas can be prepared by means of FIB based on detailed floor plans of devices. Subsequent TEM analyses are used to identify failure causes.



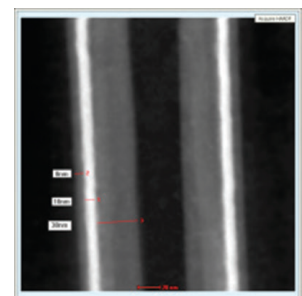
Floor plan for device identification



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



Bright field image of the MIM stack on the side wall of the trench: the  $\text{HfO}_2$  layer is imaged as a dark layer



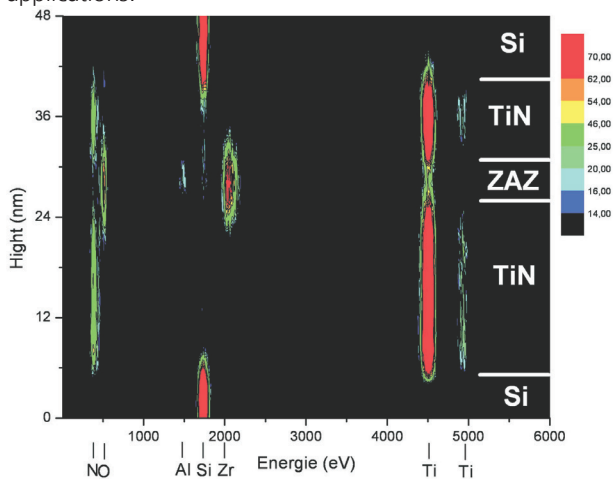
Dark field image of the MIM stack on the side wall of the trench: the  $\text{HfO}_2$  layer is imaged as a bright layer as it contains the heaviest atoms

# ELECTRON MICROSCOPY

THERMO FISHER APREO S, HITACHI S5000, FEI TECNAI F20

## EDX PROFILING OF THIN FILM STACKS

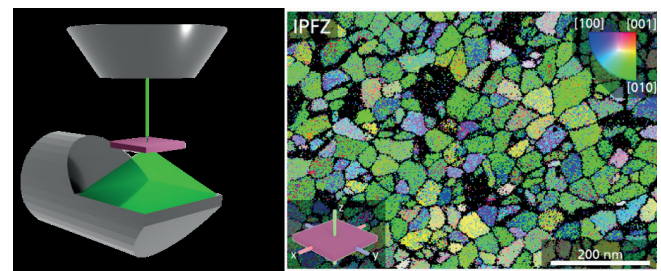
EDX was utilized to characterize the elemental distribution in a TiN/Al-doped ZrO<sub>2</sub>/TiN MIM stack for DRAM capacitor applications.



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

## EBSD/TKD

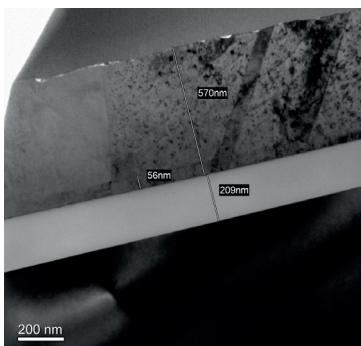
Electron backscatter diffraction methods for bulk (EBSD) and transmission Kikuchi diffraction (TKD) samples are used to identify present phases based on crystallography. Furthermore detailed grain and texture analysis up to a spatial resolution of 10 nm are carried out to investigate and improve semiconductor thin films.



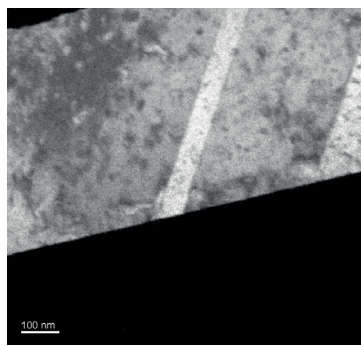
TKD principle in the SEM with new flat detector head geometry (Bruker) and analyzed grains of HfO<sub>2</sub> thin films on Si wafer substrate

## EFTEM

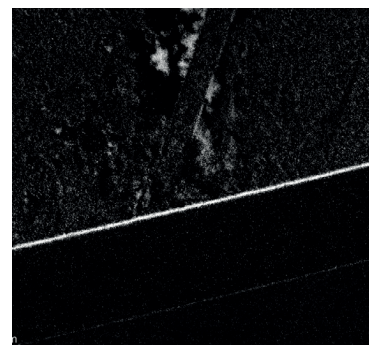
The distribution of metal dopants in a layer stack of 50 nm Mn doped Cu seed layer followed by 500 nm electroplating of Cu was studied by means of EFTEM. It shows that Mn is enriched in a very thin slice.



Bright field



Cu mapping



Mn mapping

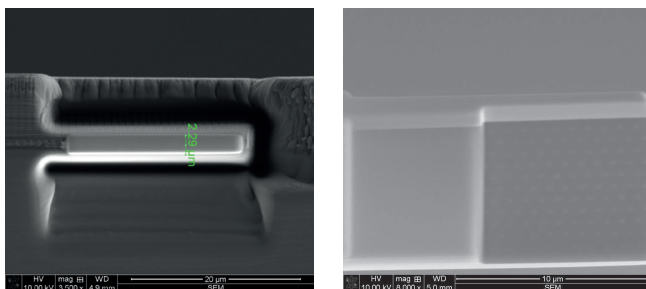
# FOCUSED ION BEAM

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 EBSD/TKD samples. 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 ELECTRON TRANSPARENT LAMELLAE FOR TEM OR EBSD

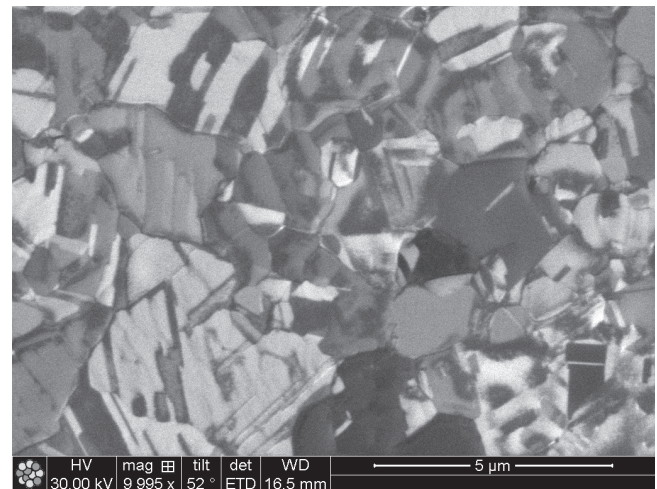
The preparation of site-specific samples for transmission electron microscopic analysis of nano-devices is not possible without focused ion beam technique. Different steps are necessary to prepare a 20–100 nm thin sample comprising e.g. a transistor device. Initially the region of interest is cut and lifted out from the wafer, then placed on a suitable grid and finally milled into a lamellae. The entire process can be carried out placing the final tip with sub-10 nm precision.



Formation of a lamellae. The ROI is covered by protective layers of C and Pt, digged out with the beam, placed on a grid, thinned until electron transparent

## ■ 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



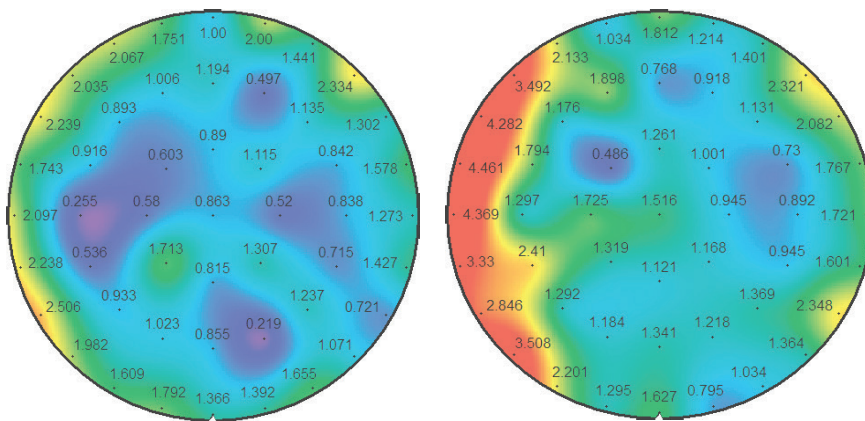
# INLINE METROLOGY

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 300 mm wafers.

## ■ SURFACE COMPOSITION WAFER-MAPPING

ThermoFischer Theta300i ARXPS

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

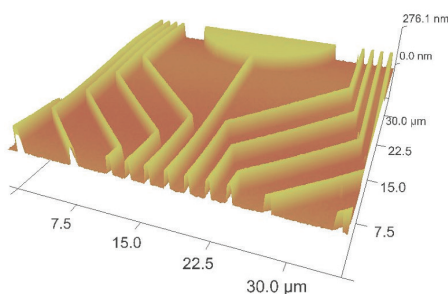


*C-distribution over a full 300 mm wafer after different process steps*

## ■ 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 3D/atomic force microscope.*

## ■ XRD

Bede HR, video system, micro focus X-ray tube

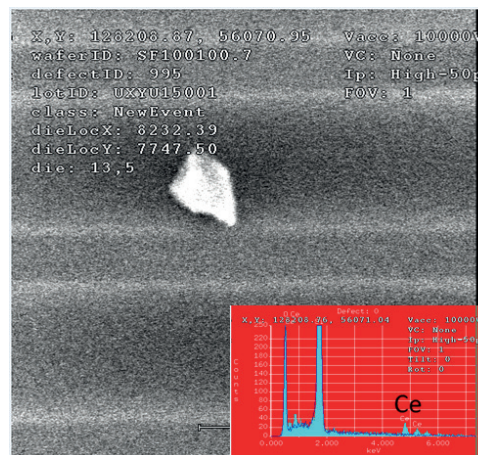
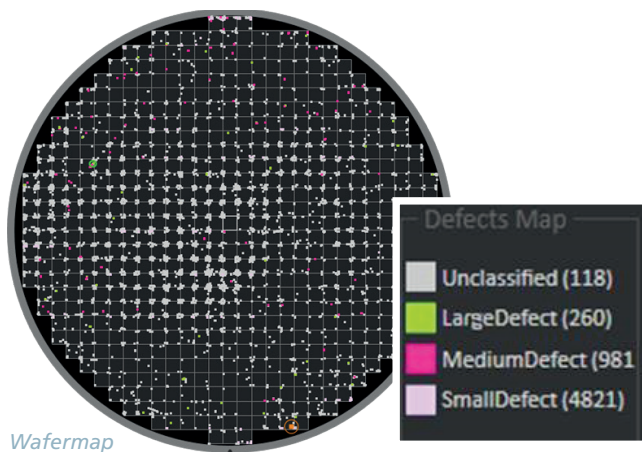
X-ray analysis - diffraction and reflectivity enables us to look at crystallographic structures. XRD gives information on structures, phases, texture and average grain size, crystallinity, strain or crystal defects.

# INLINE METROLOGY

## DEFECT INSPECTION

KLA Tencor SP3 SurfScan & Applied Materials G3E FIB, AMAT Verity CD SEM, NextIn Solutions AEGIS Wafer Inspection System

The automatic mapping and classification of the distribution of defects such as particles, structural defects, scratches can be carried out for unstructured wafer in our SP3 tool and for structured wafer in our Aegis tool. The tools provide a data format that can be read by a review SEM for closer defect inspection including EDX material analysis and FIB root cause investigations.

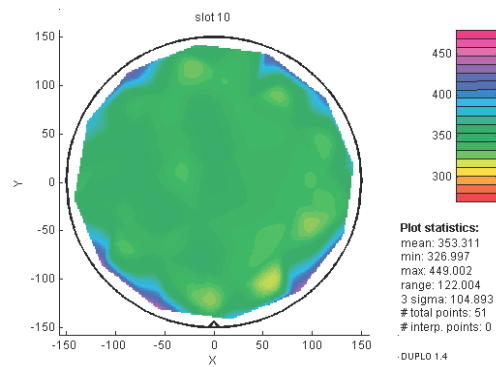


RevSEM image showing wafer pattern and CeO<sub>2</sub> polishing particle

## FILM THICKNESS WAFER-MAPPING

KLA Tencor Spectra FX100

The homogeneity of the layer thickness of a spin coated 350 nm thick ZrO<sub>2</sub> layer over a 300 mm wafer was checked by spectral ellipsometry. The mapping revealed that the layer thickness increases at the edge of the wafer.

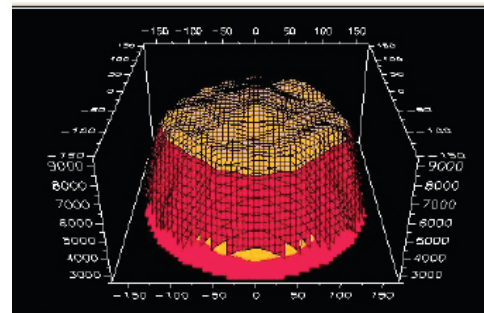


Thickness map of a spin coated thick ZrO<sub>2</sub> film

## SHEET RESISTIVITY

EURIS WS3000 & KLA Tencor RS100

The sheet resistivity of a metal layer deposited on a 300 mm 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 300 mm wafer

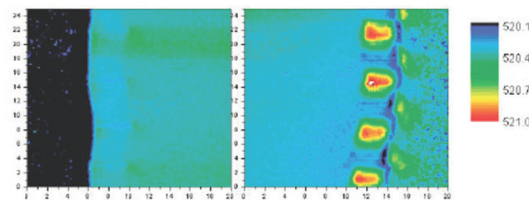
# RAMAN SPECTROSCOPY

RENISHAW INVIA REFLEX

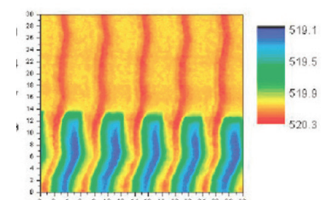
Raman spectroscopy utilizes inelastic scattering of laser light to locally excite and image characteristic vibrational modes in a material. This scattering process involves the excitation or decay of characteristic vibrations of chemical bonds. As a result Raman spectroscopy can be used for the analysis 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 300 nm. Different lasers allow us to vary the surface sensitivity, varying the integration depth from only a few nm to a few micrometers.

## ■ 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.



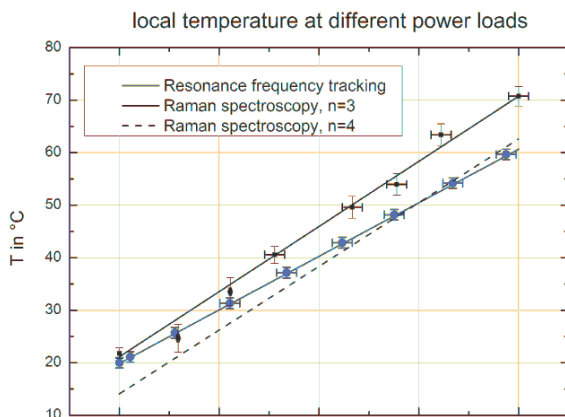
Raman maps of the cross sections of wide (left) and dense (right) trenches, showing areas of tensile and compressive stress



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

## ■ LATERALLY RESOLVED TEMPERATURE MEASUREMENTS

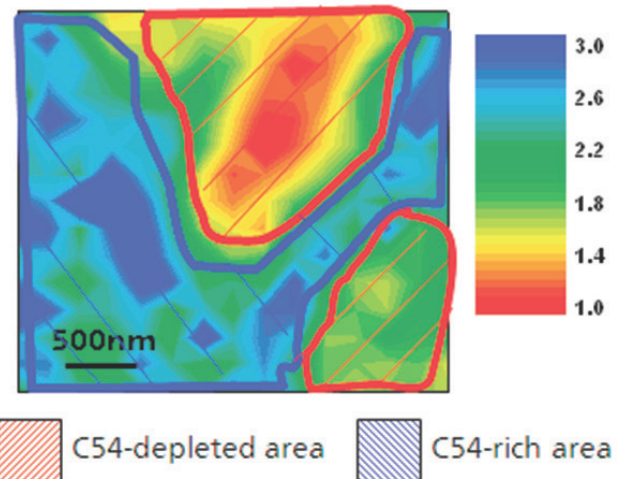
Raman spectroscopy was used to determine the absolute temperature in powered Surface Acoustic Wave devices with high lateral resolution.



Local temperature in a SAW device depending on the applied power

## ■ LATERAL RESOLVED PHASE ANALYSIS

Raman spectroscopy was used to record phase maps of  $\text{TiSi}_2$ -based transistor contacts.  $\text{TiSi}_2$  crystallizes in different structures (C54 and C49). Using Raman spectroscopy, the phase distribution can be mapped on a  $\mu\text{m}$  scale.



Raman mapping of different  $\text{TiSi}_2$  phases showing C54 depleted and enriched areas in the sample