PTB news
Scientific Newsletter
3
2015

Three times a year, PTB News provides topical information from the varied spectrum of activities of the Physikalisch-Technische Bundesanstalt (PTB) consisting of fundamental research, legal metrology and PTB's various activities in the service of the economy.



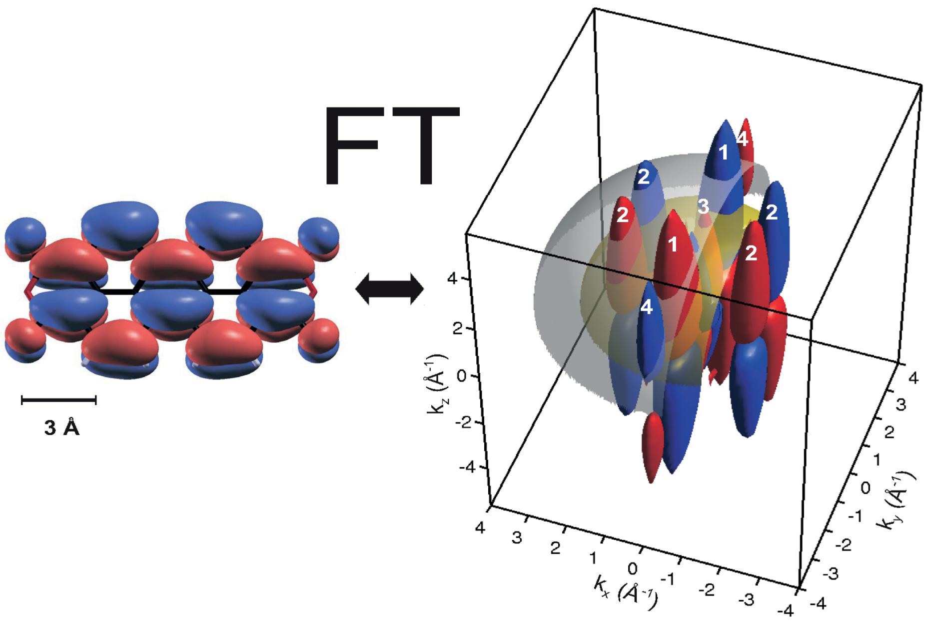
# Orbital tomography

## Angle-resolved electron spectroscopy – direct reconstruction of molecule orbitals in three dimensions

**Especially interesting for**
- fundamental research
- quantitative surface analysis

**The three-dimensional distribution of electrons inside molecules has been made visible for the first time by means of electron spectroscopy. For this purpose, molecules which were aligned on a metallic surface were irradiated with vacuum-ultraviolet radiation, and the angle and energy distribution of the electrons that were detached by the photoelectric effect was measured. This procedure, which is called orbital tomography, was developed at TU Graz (Austria) and at *Forschungszentrum Jülich* (Germany); in cooperation with PTB, it was, for the first time, successfully extended to three dimensions. These results could be achieved especially by a precise radiometric characterization of the exciting radiation and they justify to a large extent the assumption of free photo electrons for this procedure – which is controversial in the scientific community.**

The measurements were carried out at the *Metrology Light Source* (MLS) of PTB in Berlin-Adlershof with an electrostatic toroidal electron spectrometer and with monochromatized undulator radiation in the photon energy range from 14 eV to 55 eV. The electron spectrometer allows the angle- and energy-resolved measurement of the electrons detached by the photo emission process in the whole semi-infinite space above the sample. At the MLS beamline used, the relative fractions of higher spectral grating diffraction orders (i. e. photons with an energy which is different from the set energy) are reduced down to less than 1 % by filters, so that it was possible to determine the photon flux of the exciting radiation by means of calibrated semiconductor photodiodes




Electron orbital of a PTCDA molecule (left) and three-dimensional impulse distribution of the electrons after the photoemission (right). Assuming that there are free photoelectrons, the relation mainly consists in a Fourier transform (FT). The numbers 1 to 4 designate the four most important lobes of the distribution in the momentum space.

with relative uncertainties in the order of 1 %. This allowed the datasets which were measured at various photon energies and photon fluxes to be standardized for the first time. Thus, it was not only possible to measure the relative photoelectron intensities as a function of the direction of the electron impulse in two dimensions across the angular distribution, but also extended to the third dimension as a function of the impulse value by varying the photon energy – and thus the electron energy.

From the three-dimensional impulse distribution of the photoelectrons thus obtained, the three-dimensional local distribution of the electrons of the original molecule orbital could be determined numerically. In the case of this "forward pass" via a Fourier transform, it was assumed that not the quantum-mechanical final state of the photoelectrons after the emission, but solely their molecular initial state before the emission determines the impulse distribution. The results seem to confirm the validity of the assumption of free photoelectrons that are, to a large extent, not influenced by the residual molecules: especially the evolution of the integral photoelectron yield as a function of the photo energy showed only small deviations from the theoretical predictions for a free photoelectron.

The fundamental findings on the charge distribution (and thus also on the orientation) of individual molecules, which were obtained by means of metrological procedures, is highly relevant for the development of functional surfaces, e.g. of organic semiconductor materials on metallic surfaces, which open up new perspectives for photovoltaic components with increased efficiency. In turn, orbital tomography represents a very interesting metrological approach to quantitative electron spectroscopy, since this method allows reliable uncertainty budgets. With the established indirect methods, this is possible to a limited extent only, since the theoretical multi-particle models which are required for this purpose are very difficult to validate.

It is planned to continue this cooperation in the future to further develop orbital tomography for the quantitative characterization of electronic properties of organic semiconductors and of solar cells. ▪

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**Scientific publication**
*S. Weiß, D. Lüftner, T. Ules, E. M. Reinisch, H. Kaser, A. Gottwald, M. Richter, S. Soubatch, G. Koller, M. G. Ramsey, F. S. Tautz, P. Puschnig: Exploring three-dimensional orbital imaging with energy dependent photoemission tomography. Nature Communications 6, 8287 (2015)*
*DOI 10.1038/NCOMMS9287*

# Ultra-precise joints under the microscope

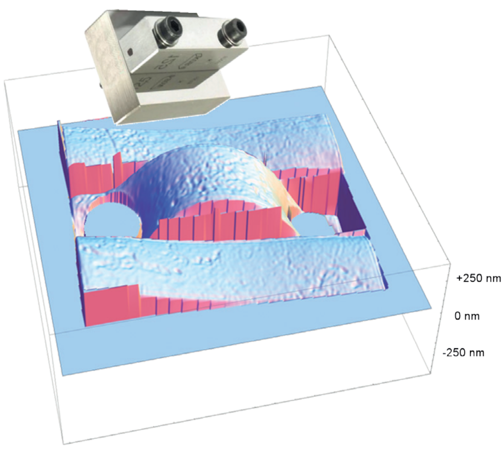
## Groundbreaking investigations on the dimensional and thermal stability of different joint technologies

**Especially interesting for**
- mechanical engineering
- precision engineering

**Within the scope of the European research project "Thermal design and dimensional drift" (T3D), various joining technologies have been investigated at PTB for the first time with regard to their long-term behavior and to their reaction to thermal influences with a precision of approx. 1 nm.**

"Conventional" technologies (such as screwings or soldering) are frequently used even for the assembly of ultra-precise instruments. In assemblies in machine tools, in optical instruments, in scanning force microscopes or in semiconductor production facilities, this may lead to changes in length that are so small that they cannot be detected by means of conventional measuring devices. These changes often occur due to structural changes inside the construction materials, but are also due to external influences such as thermal or mechanical stress.

PTB and the Fraunhofer Institute for Applied Optics and Precision Engineering (IOF – Jena, Germany) have manufactured diverse joint variants (screwed, glued and soldered connections as well as silicate bonding), especially for the T3D project. Due to the expected changes in length in the nanometer range, gauge blocks were used as base elements. As a reference, the traditionally used wringing technique without additional connection material was chosen. To measure the dimensional stability both vertically




A gauge block screwed onto an end plate (top photo in the figure). Phase topography allows very accurate measurement of topographic changes within one wavelength. In this representation, the phase shifts occurring around integral multiples of the wavelength at the transitions between the base plate and the gauge block or in the vicinity of the screws have not been corrected so as to make the actual deflection visible. It can be clearly seen that both the gauge block surface (central area) and the base plate (area at the top and at the bottom) exhibit deformations that are due to the forces applied when tightening the joint.

and laterally to the connected surfaces, the gauge blocks were connected with each other either by their end face or by their side face. In addition, also the orientation of the surfaces was determined by analyzing the interference phase topography. The measurements were carried out using PTB's high-precision interferometers. The temporal stability of the connections was checked over a period of one year. The thermal behavior from 10 °C to 30 °C was investigated within the same time interval.

With regard to length and orientation, screw joints did not exhibit any detectable change. Glued joints, in contrast, may behave very differently, depending on the hardening and on the moisture absorption of the glue or on the parallelism of the adhesive joint. In the case of soldered and bonded connections, the behavior mainly depends on the thickness of the joint layer: the thinner the layer, the more constant the length and orientation.

From these findings, a "Good Practice Guide" has been elaborated for the users of joint techniques in ultra-precise mechanical engineering which also contains guidance from EURAMET Technical Committee L's experts group. This document as well as an online tutorial on the interferometric measurement of the stability of joints are available on the T3D website (http://projects.npl.co.uk/T3D/publications.html). ■

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**Scientific publication**
*H. Lorenz, E. Beckert, R. Schödel: Phase topography-based characterization of thermal effects on materials and joining techniques. Applied Optics 54, 2046–2056 (2015); DOI 10.1038/NCOMMS9287*

# Secret partner exchange made visible

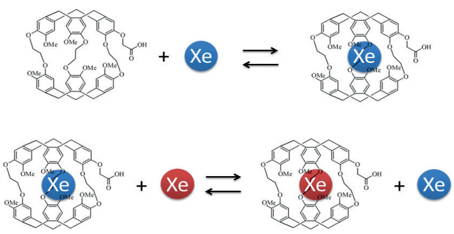
## Dynamics in a molecular host/guest relationship

**Especially interesting for**
- bioanalytics
- medical imaging
- contrast agents

**Atomic xenon can be temporarily encapsulated in molecular cages. Scientists from PTB have discovered that the direct exchange of xenon guest atoms – without the usual transition via a temporarily emptied host molecule – plays an important role here. This process could be used in new biomedical analysis and imaging procedures.**

The cage molecule cryptophane A (CrA) has been known for some time for its ability to bind individual xenon molecules reversibly. The inclusion compound has been used in recent developments in bioanalytics and in molecular imaging to make biomarkers or tissue (e.g. tumors) visible. The required specificity is attained by functionalizing the CrA, for example by linking it with a specific antibody or ligand. This CrA/antibody (or ligand) conjugate represents a biosensor whose location, quantity and state can be determined by applying nuclear magnetic resonance spectroscopy and imaging to the bound xenon molecules. To obtain high sensitivity, it is hereby extremely important to generate excess (by several orders of magnitude) magnetization for the $^{129}$Xe isotope and to accumulate, over a longer period of time, signals that are attributed to the biosensor. All this leads to very low limits of detection in the range of picomoles – which is an important precondition to be able to investigate biomolecular processes in native systems in situ.

The basic interaction between xenon and CrA resides in the occupying or leaving of an – otherwise – empty cage molecule. Furthermore, even in an already occupied cage molecule, the xenon molecule could still be supplanted by another, formerly free one – i.e. a direct change in occupancy could take place. This exchange process would be degenerated, since the initial state and the final state could not be differentiated, so that the change would remain undetected – contrary to the basic interaction. To make degenerate exchange processes visible, scientists from PTB have used a trick: by varying the xenon concentration, they could detect a modulation of the frequency of xenon replacements in CrA which is solely characteristic of degenerate exchange. In an aqueous environment – such as that prevailing in living organisms – degenerate exchange can even become the dominant mode in the host/guest interaction. The findings are of great importance for the measurement sensitivity and for quantitative analyses in the new approaches using the CrA/xenon host/guest system and are currently being investigated as to their potential for biomedical applications. ■



Basic and degenerated exchange process

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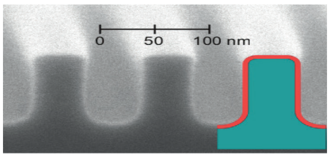
**Scientific publication**
*S. Korchak, W. Kilian, L. Mitschang: Degeneracy in cryptophane-xenon complex formation in aqueous solution. Chem. Comm. 51, 1721 (2015)*

# Optical characterization of sub-wavelength structures

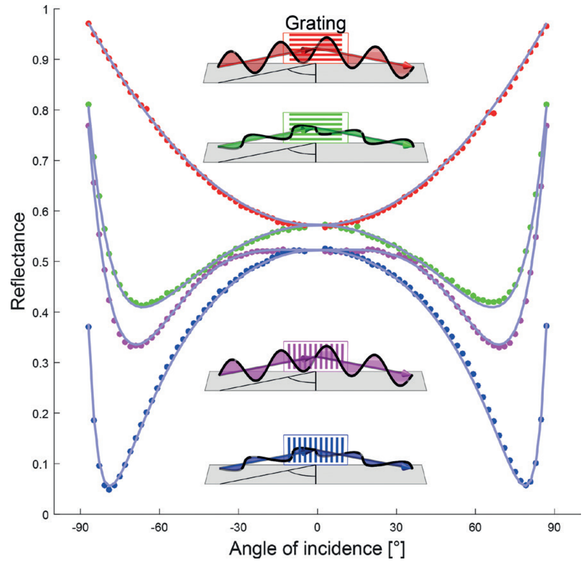
## Non-imaging procedure allows the size of grating structures to be measured far below the optical resolution limit

**Especially interesting for**
- the semiconductor industry
- the optical industry
- fundamental research

**A measurement method developed at PTB offers the possibility of measuring the size of grating structures that are clearly smaller than the optical wavelength used. Such structures play an important role, for example, in developing novel, more efficient lens systems and in manufacturing modern nanoelectronic components lithographically; however, they cannot be measured by means of conventional optical procedures. A fast procedure to measure the dimensions of sub-wavelength structures is now available.**




Top: Cross section profile (red curve) of a silicon grating structure, determined optically. For comparison, in the background: cross section profile of an identically manufactured specimen, measured by means of electron microscopy; the grating had to be cut perpendicular to the grating lines to take this picture.




Right: Comparison of the measured reflection curves (dots) with the best fit model computation (lines) at varying angle of incidence. The graphics illustrate the measurement configurations used (plane of incidence relative to the grating orientation and to the polarization of the light).

In numerous fields of natural sciences and technology, ever smaller structures are being used whose dimensions are clearly below 100 nm. Hereby, it is important, for example, for the operability of a nanoelectronic component to be able to measure the geometric dimensions of these structures very accurately and efficiently, without influencing, contaminating – or even destroying – these. Microscopy and classical scatterometry have, to date, been used successfully for this purpose.

In the case of scatterometry, the grating geometries are reconstructed based on the measurement of the intensity distribution of the different diffraction orders. In the case of dimensions far below the optical (Abbe's) resolution limit, these methods can, however, no longer be used, since in the case of grating structures with too short a periodicity, no higher diffraction orders occur. This is the reason why alternative optical measurement procedures are urgently needed – especially in the semiconductor industry – to develop and manufacture improved electronic components.

PTB has shown that even grating-like structures with periods down to a few 10 nm can be reliably characterized by means of modified optical scatterometry procedures at an illumination wavelength of 266 nm. Hereby, the remaining "diffraction order" – the specular reflection – is measured in various configurations as a function of the angle of incidence. From the measured data, it is possible to reconstruct the structure geometries very exactly. One can even detect small details of the edge profiles (such as rounded edges) which are only a few nanometers in size.

Thus, a highly efficient optical procedure is now available to measure and calibrate the dimensions of sub-wavelength structures as well as linewidth standards. This will be used, for example, in the semiconductor industry in the field of wafer metrology where it may be applied, especially for process control during manufacturing. ■

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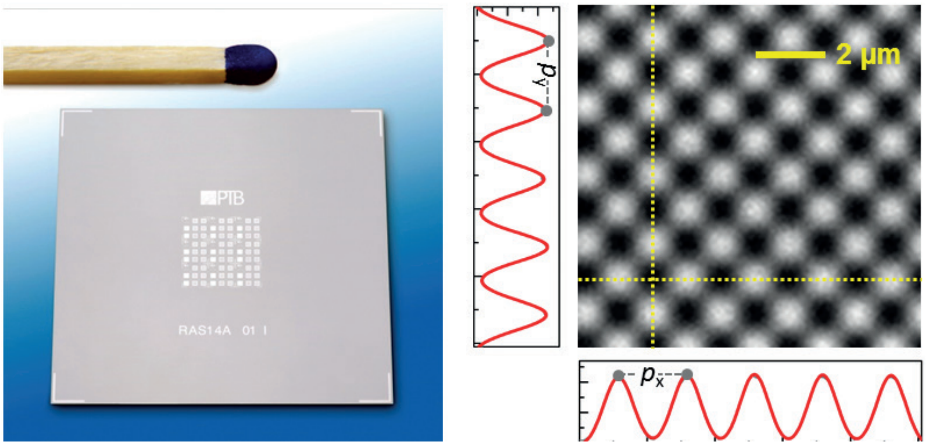
**Scientific publication**
*J. Endres, M. Wurm, J. Probst, M. Schoengen, A. Diener, B. Bodermann: Determination of sub-wavelength grating structure geometry by optical scatterometry (submitted to Optics Letters)*

# Standard for Raman microscopy

## Imaging spectrometry combined with dimensional metrology

**Especially interesting for**
- manufacturers and users of Raman microscopes
- metrology institutes
- quality assurance and process monitoring (pharmacology, semiconductor industry)

**A standard for the dimensional calibration of Raman microscopes, which was developed at PTB, allows both lateral calibration of the scanning facility and determination of the optical resolution. The quantitative analysis of Raman mappings thus becomes traceable chemical surface analysis.**




Silicon chip with arrays of different calibration structures (left); Raman mapping of a checkerboard pattern with a nominal periodicity of 2 µm (right). Horizontal and vertical profiles of the Raman signal intensity of silicon (dashed lines) are used to determine calibration factors for the (x,y) positioning unit of the Raman microscope.

In Raman spectroscopy, characteristic vibrations in molecules are excited by means of laser irradiation. The stray light contains redshifted wavelengths (Stokes lines) which are measured by means of a spectrometer and are used to identify the molecules. Confocal Raman spectrometry, as a spatially resolved surface analysis method, has meanwhile become widely used in chemical analysis as well as in quality and process checks. It is a non-invasive imaging procedure in which the sample surface is scanned point by point with a spatial resolution better than 1 µm. Hereby, each measurement point is attributed a full Raman spectrum, which results in a 2D representation of the surface composition ("Raman mapping") when individual, substance-specific bands are analyzed. If the increment and the number of measurement points enter into the analysis, then parameters such as the domain size or the degree of coverage of the detected components can be determined quantitatively.

A fundamental aspect of spatially resolved Raman microscopy is the combination of chemical and dimensional metrology. To ensure the traceability of the measurement results to the International System of Units (SI), both a chemical standard and a dimensional standard are therefore required. Whereas the reference spectra of numerous substances can either be retrieved from databases or can be generated by means of reference materials, there had, to date, been no dimensional standard adapted to the requirements of Raman microscopy.

Such a standard has now been developed at PTB for which a patent application has been filed. The standard consists of a silicon chip (a material with high Raman activity) on whose surface individual areas have been covered with a thin Au/Pd layer, so that the areas generating an intensive Raman signal are clearly delimited from areas with attenuated Raman intensity. Thus, one-dimensional and two-dimensional structures (lattices, checkerboard pattern) of different periodicity (from 4 µm to 0.8 µm) as well as point scattering centers (circles) of different sizes (from 10 µm to 0.1 µm), which are arranged separately and in pairs, have thus been generated in separate areas. This allows both the two lateral main axes (x,y) of the positioning unit to be calibrated – simultaneously and under the same conditions as those prevailing during the subsequent sample measurement – and the optical resolution of the Raman microscope to be determined for diverse configurations of the device. The dimensional quantities were traced to the SI unit "meter" by means of a calibrated scanning force microscope.

We are currently looking for a license partner for the new standard. ■

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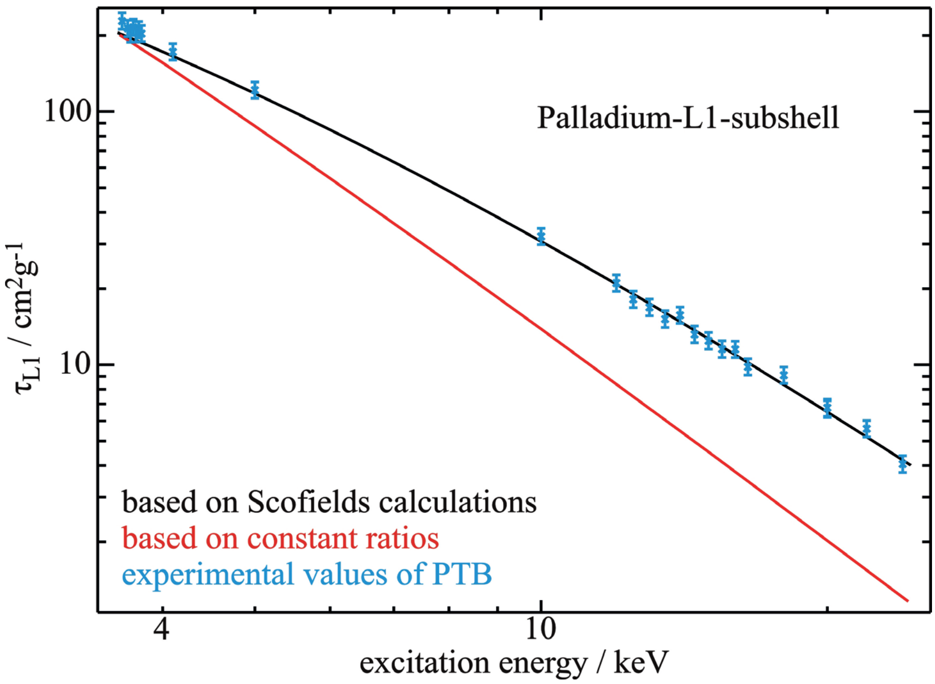
**Scientific publication**
*S. Zakel, B. Pollakowski, C. Streeck, S. Wundrack, A. Weber, S. Brunken, R. Mainz, B. Beckhoff, R. Stosch: Traceable quantitative raman microscopy and X-ray fluorescence analysis as non-destructive methods for the characterization of Cu(In,Ga)$Se_2$ absorber films. Applied Spectroscopy 70 (2016)*

# Scofield's theory confirmed

## Measuring partial photoionization cross sections makes X-ray-analytical laboratory investigations more reliable

**Especially interesting for**
- non-destructive analysis of materials
- process control
- manufacturers of X-ray-analytical laboratory devices
- validating models in nuclear physics

**By means of improved measurement procedures, PTB employees from the field of spectrometry with synchrotron radiation have succeeded – in cooperation with partners from industry – in experimentally confirming the different energy dependences of partial photoionization cross sections which could so far be predicted only by theoretic calculations. Due to this, the quantification approach of an energy-independent ratio of the partial cross sections, which is often used in particular in X-ray fluorescence analysis, has been refuted.**




Comparison of different data for the partial photoionization cross sections of the palladium L1 subshell. At higher excitation energies, the approach of an energy-independent ratio (red) shows deviations of up to a factor of 4.

X-ray fluorescence (XRF) analysis is frequently used to analyze materials in the lab in order to measure the total content of certain elements or the mass per area. Hereby, the content of certain elements is deduced from the intensity of element-specific X-ray fluorescence curves. The quantitative indication of this content is done using Sherman's fundamental parameter approach which was drawn up in 1955. Here, atomic fundamental parameters – such as photoionization cross sections and fluorescence yields – describe the probabilities of X-ray excitation and of X-ray fluorescence decay in the interaction between X-radiation and matter.

The partial photoionization cross section indicates the probability of the excitation of an electron from a specific subshell to be excited. There are two different approaches to describe the way the cross sections depend on the energy of the exciting X-ray photons for different subshells. For simplification purposes, the approach stating that the ratio of the cross sections is assumed to be constant for different subshells is often selected. Model computations in nuclear physics realized by Scofield had, however, predicted as early as in the 1970s that this does not apply to subshells with orbitals of different symmetry and that the relation with the exciting photon energy changes in this case.

In PTB's laboratory at the electron storage ring BESSY II, this predicted energy dependence has now been successfully demonstrated experimentally for the first time by using radiometrically calibrated XRF instrumentation. The results clearly show that the different energy dependences must be taken into account for a reliable elemental analysis with small uncertainties. ■

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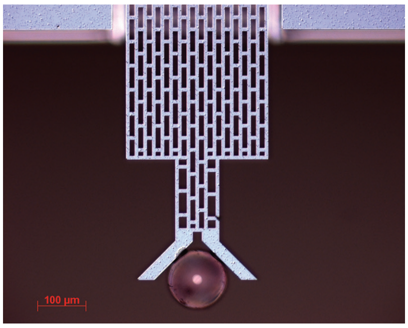
**Scientific publication**
*P. Hönicke, M. Kolbe, M. Müller, M. Mantler, M. Krämer, B. Beckhoff: Experimental verification of the individual energy dependencies of the partial L-shell photoionization cross sections of Pd and Mo. Physical Review Letters 113, 163001 (2014)*

# Hardness of microscopically thin layers

**Especially interesting for**
- optics
- microelectronics, microsystems technology
- biology, medicine

An MEMS-based nanoindentor, which was developed at PTB, allows simultaneous measurement of the indentation depth and of the indentation force. Tactile extraction of the indentation imprint is no longer necessary, which saves time. Due to the compact design and the possibility of precise mass production, it is possible to use a large number of sensors arranged in parallel. Since the MEMS can measure small forces, it is particularly well-suited to determine the elastic/plastic properties of ultra-thin layers having a thickness between 10 nm and 1 µm, which play a significant role in optics, in microelectronics or in the production of microsystems parts. It is also possible to carry out measurements on cell membranes. (Technology Offer 0135) ■



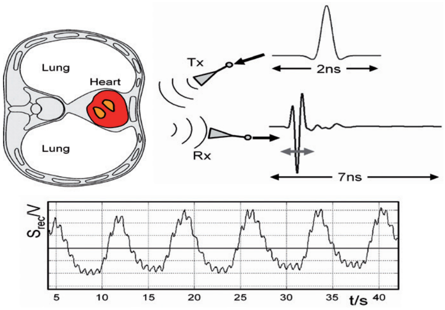

MEMS nanoindentor system with a ruby sphere as an indentor

**Advantages**
- can be used down to nanolayer thicknesses
- on-site applications possible
- small dimensions
- high precision thanks to MEMS structure

# Sharp images of organs in motion

**Especially interesting for**
- medical diagnostics

In magnetic resonance imaging (MRI), the motions of the heart, respiration or other movements of the patient may affect the imaging quality. This problem can be solved by simultaneous irradiation with ultra-wideband (UWB) radar signals. Together with the TU Ilmenau, PTB has developed a system of antennae which allows the UWB technology to be used in innovative ultra-high-field MRI. Thus, the advantages of the UWB technology (avoiding motion artifacts) are combined with those of ultra-high-field MRI (shorter measuring times and enhanced spatial resolution). (Technology Offers 0219 and 0187) ■




Correction for motion in an MRI system by means of UWB radar

**Advanatges**
- motion artifacts suppressed
- MRI spatial resolution enhanced
- dynamic MRI becomes possible
- suitable for UHF MRI

# Vector network analyzers

**Especially interesting for**
- high-frequency technology

Vector network analyzers (VNA) which, in high-frequency technology, calibrate the transmission characteristics of active and passive components in the frequency range of micro- and millimeter waves, can now be calibrated faster thanks to a new method developed by PTB. Instead of working with calibrators of the identical connector system, as has been the case to date, different – but compatible – connectors are now used. Discontinuity effects which occur here are compensated for during the modeling. (Technology Offer 0335) ■

Various coaxial calibrators

**Advantages**
- only one calibration kit required for mechanically compatible connector systems

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## Awards

**Martin Kahmann**
The Head of Department 2.3 "Electrical Energy Measuring Techniques" was awarded the DKE badge of honor for the numerous years he spent acting as a dedicated convener, spokesman, president and director of various subgroups of DKE and CEN as well as for his activities in favor of standardization at the national and the international levels.

**Best Paper Award received by Thomas Kleine-Ostmann's group**

Thomas Kleine-Ostmann from Department 2.2 and co-authors Christian Jastrow, Kai Baaske, Bernd Heinen, Michael Schwerdtfeger, Uwe Kärst, Henning Hintzsche, Helga Stopper, Martin Koch and Thorsten Schrader were honored with the 2015 THz Science and Technology *Best Paper* Award of the IEEE *Microwave Theory and Techniques Society* (MTT-S) for the publication entitled "Field Exposure and Dosimetry in the THz Frequency Range".

## Projects and initiatives

**Scientific Alliance Braunschweig/ Hannover**
Within the scope of a scientific alliance, the Technische Universität Braunschweig and the Leibniz Universität Hannover have agreed upon cooperating on scientific research in the fields of life sciences, mobility and metrology. To make the most of the total potential of the scientific region Braunschweig-Hannover, the associated master plan will explicitly also include close cooperation with other universities such as the Medizinische Hochschule Hannover (MHH) as well as non-university institutes such as the Helmholtz Centre for Infection Research (HZI) in Hannover and PTB in Braunschweig.

## PTB at trade fairs

30 November–2 December
**bonding Aachen**
Career networking trade fair.
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## EMRP and EMPIR dates

18 January 2016
**Opening of EMPIR Call 2016**
Focusing on the following main topics: energy, the environment as well as pre- and co-normative research activities. This call is concerned with the submission of draft versions of potential research topics (PRTs). Deadline for submitting PRTs: 29 February 2016. For further information, please visit: http://msu.euramet.org/calls.html.

24–25 February 2016
**Metrology for Ammonia in Ambient Air (MetNH3)**
Stakeholder workshop. PTB Braunschweig.
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30 March–1 April 2016
**Metrology for long distance surveying (SIB60)**
Workshop for the members of the SIB60 Consortium during the 3rd Joint International Symposium on Deformation Monitoring (JISDM) (http://www.jisdm2016.org/). Vienna.
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## International Conference

9.–11.3.2016
**NanoScale 2016**
11th Seminar on Quantitative Microscopy (QM) and 7th Seminar on Nanoscale Calibration Standards and Methods – Dimensional and related measurements in the micro- and nanometre range. Wrocław, Poland. The seminar will be organized together from Wrocław University of Technology, Central Office of Measures/Glówny Urzad Miar (GUM), Warszawa, Poland, and PTB.
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For further information,
please visit: http://www.nanoscale.ptb.de

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