PTB news
Scientific Newsletter
1
2017

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.

SCIENTIFIC NEWS

TECHNOLOGY TRANSFER

MISCELLANEOUS

At the surface of the new kilogram

New instrumentation to analyze the surface of silicon spheres

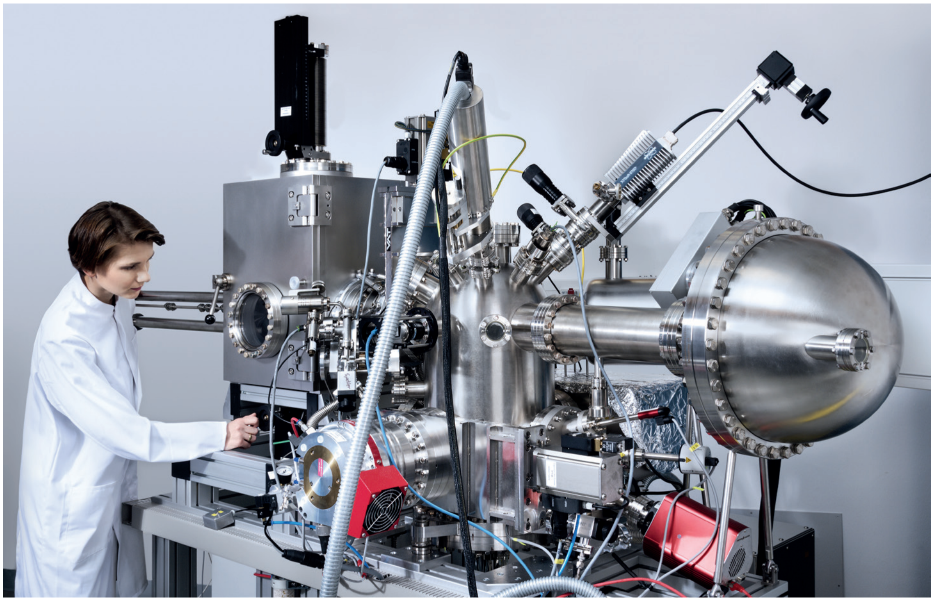
Especially interesting for
- fundamental research in physics
- materials research
- non-destructive analyzing

In order to become independent of the international prototype of the kilogram kept in Paris, the definition of the SI unit of mass, the kilogram, has to be revised. For this purpose, the number of silicon atoms in a monocrystalline silicon sphere has been determined at PTB within the scope of the Avogadro project. It is therefore necessary to measure the surface properties with the highest possible metrological accuracy. To this end, PTB has set up ultra-high vacuum (UHV) instrumentation with combined X-ray fluorescence spectroscopy and photoelectron spectroscopy.

Surface effects, such as the forming of oxide and contamination layers, have a notable influence on the mass and the volume of the silicon spheres used (the surface layers altogether have a mass of approx. 100 µg).

By combining X-ray fluorescence and photoelectron spectroscopy, it is possible to determine both the chemical composition and the mass deposition at the surface of the sphere(s) by means of a single apparatus. In this way, PTB can characterize the surface with the highest accuracy.

The core piece of this apparatus is a sample manipulator which only has minimum contact (three-point contact) with the sphere and allows measurement at any point on the surface. The instrumentation is equipped with a monochromatic Al X-ray source. By means of silicon oxide reference samples, it is possible to determine the oxygen mass deposition and thus the silicon oxide layer thickness over the total surface of the sphere with extremely small uncertainties. In addi-



UHV instrumentation for combined X-ray fluorescence and photoelectron spectroscopy on surfaces of silicon spheres tion, it becomes possible to determine contaminations which may occur during the production process or when handling the spheres. ■

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Scientific publication
R. Fliegauf, B. Beckhoff, E. Beyer, E. Darlatt, I. Holfelder, P. Hönicke, G. Ulm, M. Kolbe: Surface characterization of silicon spheres by combined XRF and XPS analysis for determination of the avogadro constant. Proc. of 2016 Conference on Precision Electromagnetic Measurements (CPEM 2016), DOI: 10.1109/CPEM.2016.7540797

DIN standard for the measurement of the thickness of polymer layers

Correction of the systematic errors in tactile and optical measurement procedures

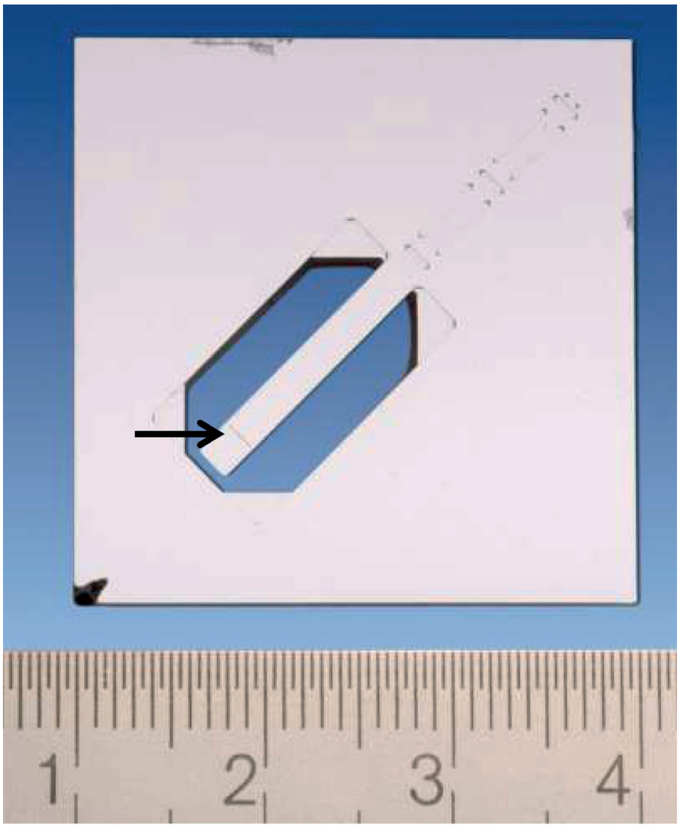
Especially interesting for
- production technology
- materials research
- microsystem technology

In microsystems, metallic components are increasingly being replaced by those from low-cost polymers. For the thickness measurement of polymers, a DIN standard, DIN 32567, is now available which was developed, among others, by PTB experts. The standard describes both optical and tactile surface measuring methods for the precise measurement of the thickness of polymer layers. The tactile methods include the determination of both the contact force and the tip radius of stylus instruments – a basic precondition for non-destructive precision tactile profile measurements.

From motion sensors to smartphones – many everyday products increasingly contain parts which are made of polymer materials. Their dimensions can be determined optically or tactically – however, with systematic deviations which are observed when measuring the thickness of transparent materials by means of optical measuring methods, and also in the stylus measurement of coatings on hard substrates. In tactile methods, the main influencing factors are the probing force and the tip radius. With viscous materials whose mechanical properties are time-dependent, also different scanning speeds affect the level of the systematic deviations.

PTB has therefore, in cooperation with other European national metrology institutes, developed a method for correcting these systematic errors, which has been standardized in DIN 32567. In this standard, the main influencing factors are shown for tactile and optical measurements, and methods for the estimation, correction and reduction of systematic errors are described. In optical measuring instruments, the main influencing factors are the effective numerical aperture of the optical system used, the position of the reflection plane in volume-scattering materials, and the phase shift at boundary surfaces upon reflection. The standard describes, for white light microscopes and focusing instruments, methods for determining these values and the correction of systematic errors on the basis of new calibration standards.

The research took place within the international MeProVisc project (Dynamic mechanical properties and long-term deformation behaviour of viscous materials) within the European Metrology Research



Reference cantilever made of silicon for determining the probing force of contact stylus instruments. The contact force is determined from the bending of the beam at the end mark (see arrow) under the influence of the contact force.

Programme EMRP. Result of the project is a comparison of creep measurements with the dynamic instrumented indentation for viscous materials. The standardization of these methods is planned in part 5 of the nanoindentation standard ISO 14577. In addition, international standardization is planned in the field relating to ISO/TC213. ■

The standard
DIN 32567: Production equipment for microsystems – Determination of the influence of materials on the optical and tactile dimensional metrology. Parts 1–5

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Scientific publication
Z. Li, U. Brand, T. Ahbe: Towards quantitative modelling of surface deformation of polymer micro-structures under tactile scanning measurement. Meas. Sci. Technol. 25, 044010 (2014)

What's inside the human brain?

Anatomically selective measurement of metabolites in vivo thanks to new technique

Especially interesting for
- medical imaging
- brain research

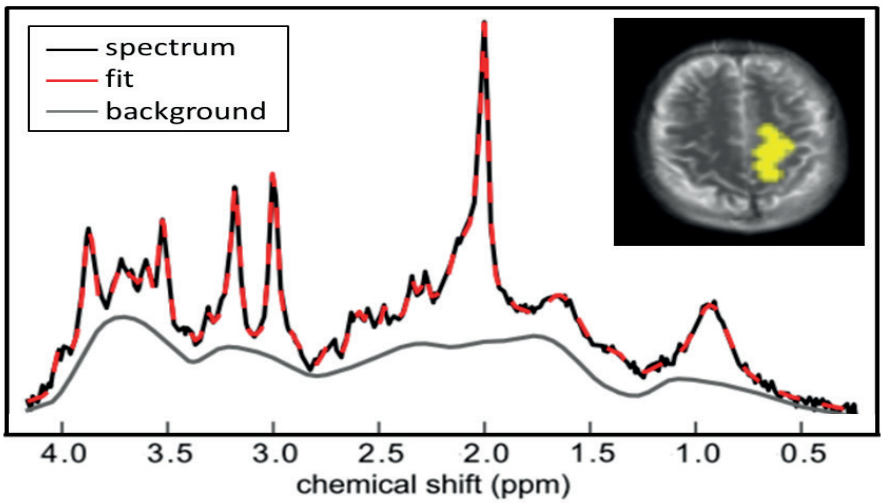
Magnetic resonance spectroscopy (MRS) allows the non-invasive determination of metabolites in the human brain in vivo. However, it is usually limited to rectangular volumes. This makes it difficult to quantify the metabolites selectively in a certain cerebral structure in order to establish a precise neurochemical profile of this structure. A new pulse sequence combined with a multi-channel transmission technique allows the excitation of the magnetic resonance signal in principle in volumes of any shape, hereby making MRS anatomically selective.

Modern magnetic resonance tomographs provide innocuous – radiation-free – imaging. In addition, they permit the concentrations of metabolites to be measured non-invasively inside the human body. Quantitative magnetic resonance spectroscopy thus contributes to finding out where, how and why neurological and psychiatric diseases occur, and it will, in the future, be very helpful in diagnosing them and controlling the therapy applied.

Research on this technique intensively deals with triggering even more precisely those anatomic target regions in which a neurochemical profile is to be measured. To date, the measuring signal in cerebral MRS could only be obtained from a rectangular target volume (voxel) in the cerebral region of interest. This limitation results from the interaction between the magnetic field's gradients which determine the voxel geometry and the radio frequency pulses which excite the spin system to be detected. Rectangles are, however, difficult to adjust to the twists of the cerebral cortex or other anatomic structures that usually exhibit irregular shapes. If the MRS voxel is selected so as to be small enough to be specific to a particular structure, the signal-to-noise ratio (SNR) is, however, lower, which affects the precision of the measurement. If the voxel is enlarged, then a large amount of cerebral matter is scanned which, in turn, results in a biased measurement.

At PTB, a new MRS sequence (SHAVE – SHAped Voxel Excitation) has therefore been developed; it allows the excitation of anatomically adjusted volumes. The core of the procedure consists in the multi-channel transmit technique for the radio frequency excitation of the volume which is realized by means of an 8-channel transmit array combined with an 8-channel radio frequency coil. Their individual channels emit radio frequency pulses whose amplitude and phase can be manipulated independently of each other. By combining such pulses, it is in principle possible to excite any kind of target volume shape. With this new measurement techniques, it was possible to obtain MR spectra from voxels without any undesirable proportion of gray matter.

Another advantage of this method was the implementation of a two-shot procedure to localize the third direction in space. In this way, the MR signal can be read out with a very short echo time. As a result, the metabolite resonance to be determined hardly has any time to decay and thus provides strong signals. It



In vivo MR spectrum with fitted result from an anatomically shaped voxel excited by means of the SHAVE method which only contains white cerebral matter (i.e. is not contaminated by gray matter). The individual peak positions in the spectrum represent metabolites, their amplitudes represent their concentrations. In the right corner of the diagram, the voxel can be seen in the brain image (yellow).

is therefore also possible to detect metabolites with a very low SNR due to their short transverse relaxation time.

To make this new method easier to apply in hospitals, the workflow will be optimized and adapted to functionally highly differentiated (e.g. cortical and subcortical cerebral) structures which are difficult to trigger.

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Scientific publication
P. Waxmann, R. Mekle, F. Schubert, R. Brühl, A. Kühne, T. Lindel, F. Seifert, O. Speck, B. Ittermann: A new sequence for shaped voxel spectroscopy in the human brain using 2D spatially selective excitation and parallel transmission. NMR Biomed. 29, 1028–1037 (2016)

Enhanced spectral color measurement procedure

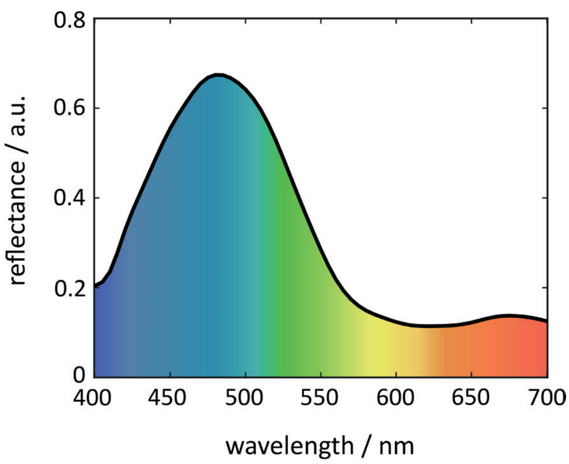
Within the scope of a cooperation project with industry, PTB has developed a novel approach to the quality control of color spectra

Especially interesting for
- spectral data analysis
- multispectral image acquisition systems
- industrial color inspection

Today, spectral color measurements are indispensable in numerous areas of science and industry. In the printing industry, for example, clear color definitions and exact color repetitions are necessary. The procedures used to date, however, only provide random and locally limited results – and some require a large amount of time and effort. Within the scope of a joint project with the Chromasens GmbH company, PTB has developed a procedure that allows color spectra to be realized with great efficiency and accuracy based on prior spectral information. A simulation study has demonstrated that this approach provides considerably better results than conventional procedures.

Each color has its individual “fingerprint”, the color spectrum which consists of different wavelengths and intensities of light. Measuring the spectrum discloses which fractions of the light are reflected and reach the observer – it is therefore called a “reflectance spectrum”. Standard procedures that are currently used for spectral color measurement determine the reflectance spectrum only punctually. Thus, large surfaces cannot be detected as a whole. Moreover, such a measurement is tedious: when checking the quality of printing, for example, a manual comparison between the target colors and the actual colors is carried out by means of special color measuring instruments on test sheets.

In the future, this step might become redundant. By means of a multispectral line scan camera and of an algorithm which was developed at PTB, it will be possible to record – fully automatically and in real time – the full reflectance spectrum of a surface to be measured. The camera used is equipped with 12 color channels which, similar to the cones in the human eye, react to light with a different sensitivity. First, the spectral sensitivity curves of the individual channels are determined. This is the basis of the subsequent reconstruction of the color spectrum from the measured data of the camera. The reconstruction procedure is based on Bayesian statistics. Hereby, the measurement results of the camera are linked up with previous knowledge on the



The multispectral line scan camera decomposes the color spectrum. (Photo: Chromasens GmbH) spectrum. This approach allows the reliable characterization of the quality of the measurement results.

A simulation study has demonstrated that the new measurement procedure yields results that are much more reliable than those obtained by means of the conventional, empirical procedure. PTB's project with the Chromasens GmbH company has been extended for another two years. The objective consists in paving the way for a wide use of this new method. Due to the acquisition of spectral properties of sample surfaces, it is thus possible to perform efficient material sorting and conformity checks in industrial production. ■

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Scientific publication
M. Dierl, T. Eckhard, B. Frei, M. Klammer, S. Eichstädt, C. Elster: Improved estimation of reflectance spectra by utilizing prior knowledge. J. Opt. Soc. Am. A. 33, 1370–1376 (2016)

Calibration of autocollimators

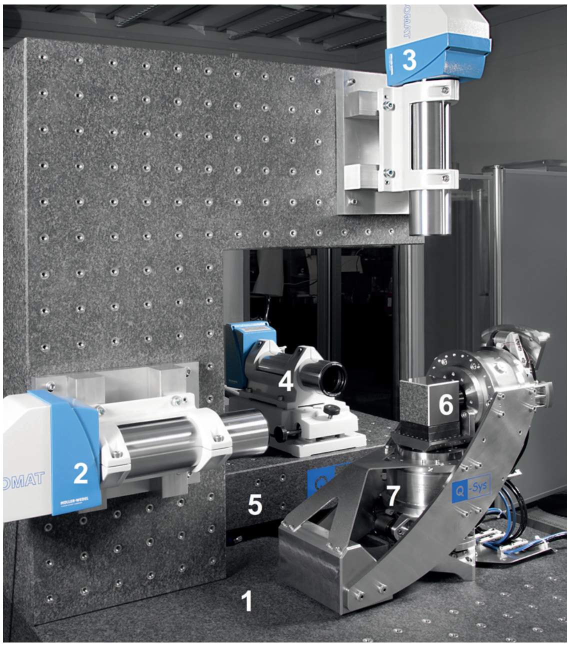
Novel system allows first-time calibration of spatial angles

Especially interesting for
- users and manufacturers of autocollimators
- metrological laboratories

At PTB, a novel calibration system for autocollimators has been set up, the Spatial Angle Autocollimator Calibrator. The calibration capabilities for optical angle measurements have thus been extended from plane to spatial angles for the first time. In addition, the system allows calibrations at distances from 250 mm to 1800 mm.

Autocollimators allow contact-free measurement of the inclination angles of reflecting surfaces via the angular deflection of a reflected measuring beam. To date, PTB has calibrated these by means of the national primary standard for the plane angle, the angle-measuring table 220 (WMT 220). These calibrations are, however, only possible along one measuring axis of the autocollimator and at a distance from 250 mm to 550 mm from the reflector. In order to extend the calibration capabilities to spatial angles and larger distances, a novel calibration system for autocollimators, the Spatial Angle Autocollimator Calibrator (SAAC), has been set up at PTB.

The SAAC is based on a Cartesian arrangement of three autocollimators which are directed towards a reflector cube. Two of them serve as reference measuring systems; the third autocollimator is the object under calibration. It is located on a linear sliding carriage with which the distance from the cube can be adjusted from 300 mm to 1800 mm. By means of a two-axis tilting system, the cube can be tilted within an angular range of (3000 × 3000) arcsec^2 around two axes that run vertically to each other. The autocollimator to be calibrated hereby records both tilting angles of the cube. Due to the Cartesian arrangement, each of the reference devices, however, only measures one of the two tilts as a practically plane angular deflection. These reference devices can be calibrated directly using the WMT 220. Measuring two plane angles thus ensures the traceability of the calibration of spatial angles to the national primary standard.



Spatial Angle Autocollimator Calibrator: granite base plate (1), reference autocollimators (2; 3), autocollimator to be calibrated (4), linear sliding carriage (5), reflector cube (6), two-axis tilting system (7)

The SAAC makes it possible to calibrate autocollimators with respect to spatial angles and to characterize distance-dependent effects. Manufacturers who would like to improve their instruments, and users who apply them to measuring situations other than plane angles and fixed spacing, both benefit similarly from the new calibration facility. An example of this is the high-precision measurement of the shape of optical surfaces that are used to shape synchrotron or free-electron laser radiation beams. ■

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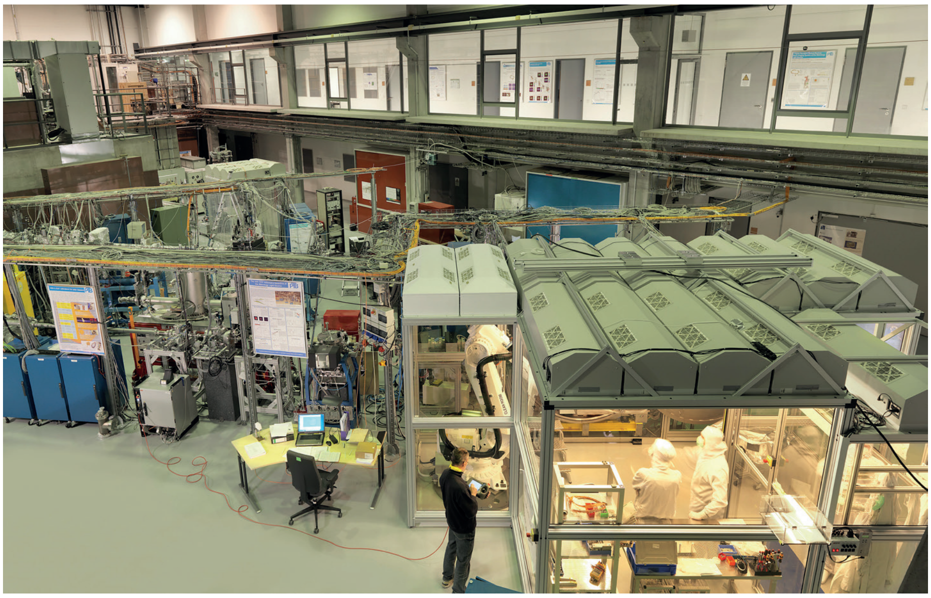
Scientific publication
O. Kranz, R. D. Geckeler, A. Just, M. Krause, W. Osten: From plane to spatial angles: PTB's Spatial Angle Autocollimator Calibrator. Adv. Opt. Technol. 4, 288–294 (2015)

Development of EUV radiometry

Cooperation project with Carl Zeiss SMT GmbH continued

Especially interesting for
- the semiconductor industry
- EUV lithography
- optical metrology



PTB's EUV beamline at the Metrology Light Source. In the foreground (right), one can see the cleanroom in which the samples are assembled. The large-scale EUV reflectometer is located at the back of it (concealed). The mirrors, some of which are very large and heavy, are mounted into the reflectometer by a robot (seen on the left in the cleanroom).

The cooperation project with Carl Zeiss SMT GmbH in the field of radiometry, which started back in 1998, has been prolonged until the end of 2020. At PTB, this cooperation project represents the starting point and the basis of a field of work which provides extensive metrological support to the German and the European semiconductor industry for the development of EUV lithography. Besides EUV reflectometry and EUV detector radiometry, the metrological developments are especially geared to EUV scatterometry in a group of cooperation partners which has considerably grown over the past few years.

EUV lithography is an optical technology at a working wavelength of 13.5 nm, by means of which it will be possible to realize high-end semiconductor components in the near future. The driving forces of this technological development are the world leader on the lithography stepper market, ASML from the Netherlands, and its German partner for precision optics, Carl Zeiss SMT GmbH. PTB has maintained cooperation agreements on EUV radiometry with both companies for several years. Meanwhile, cooperation projects are being realized with basically all renowned representatives of the German and European supply industry in order to develop EUV lithography.

Besides the number of hours now amounting to more than 6000 per year, also the qualitative requirements placed on the measurements in EUV radiometry, which is performed in several shifts with synchrotron radiation at two beamlines at the electron storage rings BESSY II and Metrology Light Source (MLS) in Berlin-Adlershof, are increasing. This is the reason why the original large-scale EUV reflectometer – which had been set up together with Carl Zeiss within the scope of a BMWi-funded project in the first years of the cooperation project – will, in the coming years, be replaced by a new system equipped with a hydrocarbon-free vacuum system and improved sample positioning. The setup of an EUV telescope system, with the aid of which the spatial resolution on the surface of the optical components shall be improved to approximately 10 µm, in contrast, is close to completion. This benefits especially the development of novel measurement procedures based on EUV reflectometry and scatterometry for the investigation of structured surfaces. Due to the short wavelength used, these procedures could in future complement or even replace optical scatterometry in structure measurements on semiconductor wafers. ■

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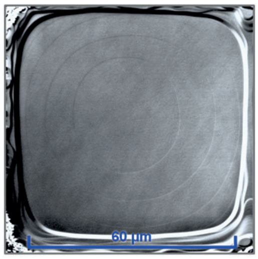
Scientific publication
V. Soltwisch, A. Haase, J. Wernecke, J. Probst, M. Schoengen, S. Burger, M. Krumrey, F. Scholze: Correlated diffuse x-ray scattering from periodically nanostructured surfaces. Phys. Rev. B 94, 035419 (2016)

Atomically smooth surfaces

Especially interesting for
- roughness measurements
- optical metrology
- manufacturers of gauge blocks

At PTB, a novel manufacturing procedure based on (111)-silicon wafers allows areas with dimensions of up to 150 µm² to be generated which exhibit both atomically smooth surfaces and surfaces with mono-atomic steps. In this procedure, a self-organizing process is triggered by using a targeted heating process under ultra-high vacuum within several lithographically structured patterns. The atomically smooth surfaces thus obtained represent huge progress in flatness measurement and, together with the mono-atomic steps, may soon be used as the basis for novel artifacts and standards in dimensional metrology. These smooth areas can also be produced on both sides of polished silicon wafers, i.e. with a discrete number of grating levels between opposite surfaces. (Technology Offer 0440) ■



Mono-atomic steps in an amphitheater structure on (111)-silicon

Advantages
- *highest-grade step-height and surface flatness standards*
- *suitable for the calibration of optical microscopes*

Tunable Helmholtz resonator

Especially interesting for
- room acoustics experts
- professional recording studios
- noise protection in industry

Whether you are a high-end music connoisseur in your living room or a recording engineer in your recording studio, you both know them well – the location-dependent low-resonance frequencies which can be extremely unpleasant when listening to music. Low-frequency noise originating from machines can be similarly amplified in industrial locations due to room resonances. With PTB's invention – a tunable Helmholtz resonator which can be finely adjusted according to the ambient environment – it is possible to optimize room acoustics in the low-frequency range. A pre-dimensioned module the size of a medium loudspeaker combined with an element which allows adjustment to the room's resonance frequencies can considerably reduce low-resonance frequencies in the range from 20 Hz to 200 Hz. (Technology Offer 0346) ■

Here, disturbing resonance frequencies in the staircase were significantly reduced thanks to the new Helmholtz resonator.

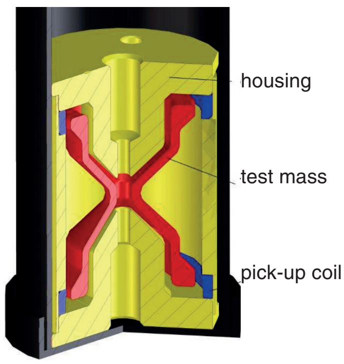
Advantages
- *reduces the disturbing effect of room resonance frequencies*
- *enhanced listening experience thanks to balanced bass reproduc tion*

Enhanced gravimeter

Especially interesting for
- gravimetry

In conventional superconducting gravimeters, a test mass is levitated by the magnetic field of a superconducting coil. An elaborate adjustment allows the gravity of the earth to be measured at geographical locations by means of this measurement principle. PTB's new invention is based on an alternative geometry of the magnetic field and thus contains a new test mass in the form of a double cone. In this arrangement, the test mass centers itself, making a complex feedback circuit obsolete. The measuring signal is generated inductively by means of pick-up coils so that it is possible to enhance the resolution by a factor of up to 100 (depending on the technology used). (Technology Offer 0429) ■

Advantages
- *increased resolution*
- *without elaborate adjustment*



Diagrammatic sketch of the test mass (red) in the measuring arrangement

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Awards

Roman Schwartz, Vice-President of PTB, was confirmed in his office as the "CIML First Vice-President" (CIML: International Committee of Legal Metrology) and re-elected for another six years at the 51st CIML session in Strasbourg.

Raya Roshana Gallas
This staff member of Department 6.2, "Dosimetry for Radiation Therapy and X-ray Diagnostic Radiology", was awarded the Otto Haxel Prize of Heidelberg University for her excellent master's paper entitled "A Novel Method for Assessment of Beam-Tissue Interactions in Helium Ion Radiotherapy".

Ludwig Büermann
This staff member of Department 6.2, "Dosimetry for Radiation Therapy and X-ray Diagnostic Radiology", received the 2016 IEC 1906 Award. The IEC award is an appreciation of his expert knowledge and of his particular commitment to international standardization.

A building for nanometrology research

LENA (the Laboratory for Emerging Nanometrology) celebrated a "topping-out" ceremony for a new building and the conversion of an old one. LENA is a research center at the Technische Universität Braunschweig founded in close collaboration with PTB and is already being operated jointly. The main floor space of 2574 m² in total provides more than 100 scientists with working space. LENA has excellent equipment in the form of large instrumentation for nano-analysis at its disposal.

Nanotechnologies Action Plan

As part of the Federal Government's "Nanotechnologies Action Plan 2020", the federal authorities responsible for human and environmental safety have extended their joint research strategy. As early as 2008, the Federal Institute for Occupational Safety and Health (BAuA), the BAM – Federal Institute for Materials Research and Testing, the Federal Institute for Risk Assessment (BfR), the Federal Environment Agency (UBA) and PTB started investigating the opportunities and risks relating to the use of nanomaterials. This has now been extended to so-called "new materials".

Exchange with Argentina

The scientific exchange between PTB and its Argentinian partner institute, Instituto Nacional de Tecnologia Industriel (INTI), is growing closer. Argentinian foreign minister Susana Malcorra and PTB's president, Joachim Ullrich, have signed a memorandum of understanding (MoU). It is already the third of its kind having been signed by the two institutions since 1967.

MetroSommer 2017

This year again, students are invited to take part in topical projects of PTB under the slogan "Dein genauester Sommer" (Your most accurate summer) from 1 August until 30 September 2017. This voluntary internship, which takes place within the scope of "MetroSommer", was already very successful in 2016. You are welcome to submit your application if you study a STEM subject at a German university between the third semester of a Bachelor's or "Diplom" study program and the beginning of your Master's or "Diplom" final paper. Participants in this voluntary internship will be remunerated € 500 per month.
You will find the application form and an overview of all projects offered within the scope of the internship at www.ptb.de/metrosommer (in German).
Application deadline: 30 April 2017
Contact: Julia Hornig (+49 (0)531 592-3511, metrosommer@ptb.de)

Summer School

This year, PTB and Braunschweig Technical University will again offer an international summer school which will be funded by the Helmholtz Fund. It addresses doctoral candidates in STEM subjects from Germany and abroad and will take place in Kloster Drübeck from 8 to 11 August 2017. The main topic will be the "Frontiers of the Measurable". Application deadline: 21 April 2017. For further information, please visit: www.ptb.de/summer-school-2017.

EMRP/EMPIR

In 2017, there will be calls for papers on the following topics for new EMPIR projects: TP Industry, TP Fundamental, TP Normative, and TP Research Potential.

- 15 June to 2 October 2017: Applications for Joint Research Projects (JRPs)
- 27–30 June 2017: Partnering Meetings, Berlin

Further locations for partnering meetings will be announced in due time.

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