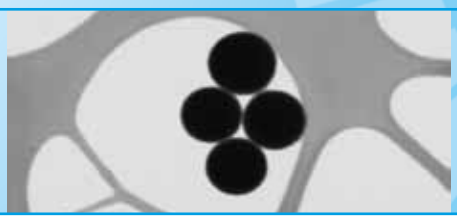
PTB news

3/2010

The Science Newsletter of the Physikalisch-Technische Bundesanstalt

A well-rounded sphere, *Page 2*



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The "new" kilogram is approaching

Avogadro constant determined with enriched silicon-28

A milestone in the international Avogadro project has been reached: With the aid of a single crystal of highly enriched ^{28}Si, the Avogadro constant has now been measured as exactly as never before with a relative overall uncertainty of $3 \cdot 10^{-8}$. Within the scope of the redefinition of the kilogram, the value $N_A = 6.02214078(18) \cdot 10^{23}$ mol^{-1} permits the currently most exact realization of this unit.

Especially interesting for
- *The fundamentals of metrology*
- *All mass determinations*

The crucial phase of the long-term Avogadro project – which is coordinated by PTB – started in 2003: In that year, several national metrology institutes launched – together with the Bureau International des Poids et Mesures (BIPM) and in cooperation with Russian research institutes – the ambitious project of having approximately 5 kg of highly enriched ^{28}Si (99.99 %) be manufactured as a single crystal, of measuring the Avogadro constant with it and of achieving – by the year 2010 – a measurement uncertainty of approx. $2 \cdot 10^{-8}$. Meanwhile, the first measurements have been completed on the two 1 kg spheres of ^{28}Si – which had been

The high-purity silicon sphere of the Avogadro experiment reflects a copy of the international kilogram prototype – the last embodiment of a unit via a physical body. The sphere, in contrast, stands for the definition on the basis of atomic properties or fundamental constants.

polished in Australia – and their density, lattice parameter and surface quality have been determined.

The single steps: After an extensive check of the crystal perfection, the influence of the crystal lattice defects was assessed. Then, the lattice parameter was determined at the Italian metrology institute (INRIM) by means of an X-ray interferometer, and confirmed by comparison measurements with a natural Si crystal at the American NIST. At BIPM, NMIJ (Japan) and PTB, the masses of the two silicon spheres were linked up in vacuum to the international mass standards. In the respective Working Groups of NMIJ, NMI-A (Australia) and PTB, the sphere volume was measured optically – with excellent agreement – by means of interferometers with different beam geometries. The surface layer (basically composed of silicon dioxide) was spectroscopied with electron radiation, X-ray radiation and synchrotron radiation in accordance with different procedures, analyzed and taken into account for the determination of the silicon density. The unexpectedly high metallic contamination of the sphere surfaces with copper and nickel silicides which occurred during the polishing process was measured, and its influence on the results of the sphere volume and of the sphere mass was assessed. This resulted in a higher measurement uncertainty.

What was decisive for the success achieved – i.e. a relative overall measurement uncertainty of $3 \cdot 10^{-8}$ – was the development of a new mass-spectrometric method for the determination of the molar mass at PTB.

The result is a milestone on the way towards a successful realization of the new kilogram definition on the basis of fundamental constants whose values have been fixed. At present, the agreement of this value with other realizations of the kilogram is not good enough to change the existing definition of the mass unit. The present state of the Avogadro project is, however, so promising that – on the basis of new measurements with improved sphere interferometers – the measurement uncertainty of $2 \cdot 10^{-8}$ demanded by the Consultative Committee for the Mass (CCM) will in the near future be achieved on contamination-free spheres and will probably even be undercut.

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Scientific publication

Andreas, B.; Azuma, Y.; Bartl, G.; Becker, P.; Bettin, B.; Borys, M.; Busch, I. M.; Gray, M.; Fuchs, P.; Fujii, K.; Fujimoto, H.; Kessler, E.; Krumrey, M.; Kuetgens, U.; Kuramoto, N.; Mana, G.; Manson, P.; Massa, E.; Mizushima,S.; Nicolaus, A.;. Picard, A.; Pramann, A.; Rienitz, O.; Schiel, D.; Valkiers, S.; Waseda, A.: An accurate determination of the Avogadro constant with ^{28}Si single crystals. Phys. Rev. Lett. 106 (2011), 030804 (4p.)

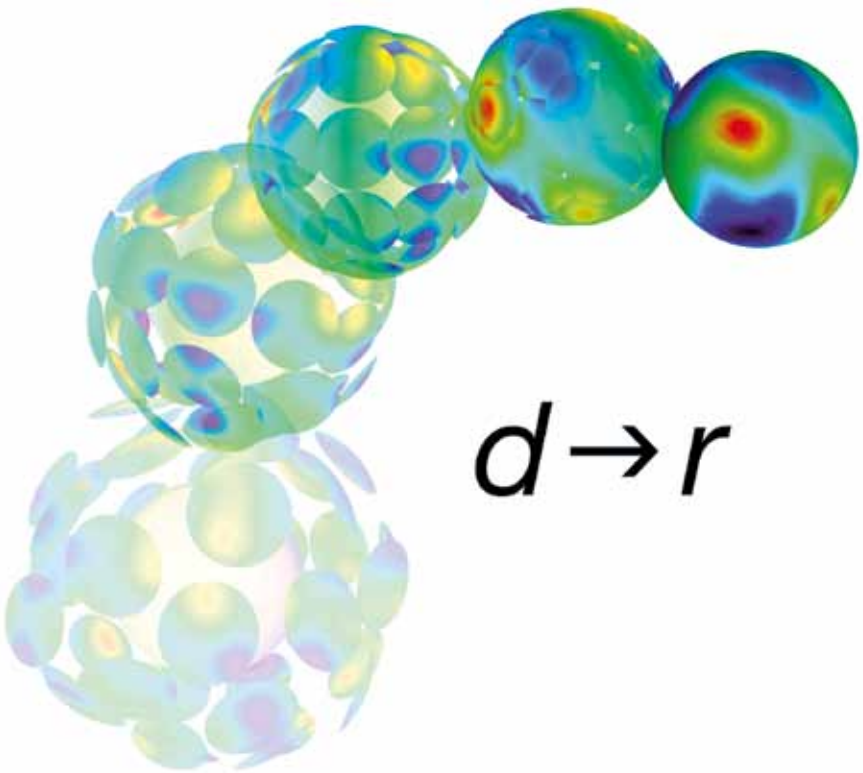
A well-rounded sphere

Measuring the form of spherical standards absolutely – and exact to a few nanometers

Thanks to a new evaluation method, it is now possible to reconstruct the absolute form of spherical measurement objects from the measurement data of PTB's sphere interferometer with an uncertainty of just a few nanometers. This opens up new application possibilities in production measurement technology.

Especially interesting for
- *production measurement technology*

Originally, PTB's sphere interferometer had been designed for measuring the volume of spherical standards – especially that of the silicon spheres of the Avogadro project – via precise diameter determinations with an uncertainty of one nanometer or less. The new method now also allows an absolute determination of the radius topography and expands the application possibilities of the sphere interferometer. In addition, the method applied so far for the volume determination of the Avogad-



For the determination of the diameter topography, the individually measured surface segments are distributed around the sphere and partly overlap. From these overlaps, the topography of absolute sphere radii can be reconstructed with the aid of an optimization calculation.

ro spheres via the determined diameters was validated. Using the radii instead of the diameters becomes necessary for the volume determination only in the case of spheres with clearly larger roundness deviations.

So-called stitching procedures, where individually measured segments of an (optical) surface are combined to form a total topography, are already known in the optical industry. For complete sphere topographies, the measurement uncertainty has so far, however, still been larger than the form deviation of the spherical test pieces to be measured. The new evaluation method yields – for the first time – the absolute form of a sphere, exact to a few nanometers, and not the diameter topography (as has been common practice so far) which is point-symmetric to the sphere centre and, thus, does not allow any clear side assignment of the topographic characteristics to the sphere surface.

The associated algorithm was first checked on the basis of simulated data sets. The comparison with the results of real measurements and with independent results from roundness measurements shows very good agreement – and this with a mean deviation of less than 5 nm.

The new evaluation method opens up the possibility of using the sphere interferometer also for the precision form characterization of spherical measurement objects used in production measurement technology. In addition to the established tactile roundness measurements, the new method is characterized by contact-free scanning of the surfaces at a high lateral resolution and achievable measurement uncertainties of just a few nanometers.

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Scientific publication
Bartl, G.; Krystek, M.; Nicolaus, A.; Giardini, W.: Interferometric determination of topographies of absolute sphere radii using the sphere interferometer of PTB. Meas. Sci. Technol. 21 (2010), 115101

Measuring resistances faster and more precisely

Improved cryogenic current comparator system for the calibration of resistances

It is almost a natural law: when attempting to improve something, the effort to do this increases disproportionately to the benefit. This also applies when it comes to reducing measurement uncertainties: to become twice as precise, usually a four times longer measurement time is required. With the newly developed system for resistance measurements, however, a four times smaller uncertainty is achieved in half the measurement time.

Especially interesting for
- *metrology institutes*
- *calibration laboratories*
- *manufacturers of electric precision metrology*

In connection with the superconducting quantum interferometer in one of the helium cans, the new system does not only measure with larger velocity and higher precision than the old one, but it is also more compact and easier to handle. This is illustrated by the photomontage: the functions of all the electronic components accommodated in the rack on the left are now bundled in one compact housing.

Electric resistance is one of the most important measurands, as many physical quantities are determined via a resistance measurement. The extreme precision with which resistances are calibrated is achieved by traceability to fundamental constants via the quantum Hall effect in combination with a complex metrological set-up employing so-called cryogenic current comparators which use superconducting effects.

Thereby, measurement uncertainties of less than 10^{-9} are achieved – but at the same time, measurement times of almost one hour have to be tolerated. In the case of the cryogenic current comparator system developed at PTB, there is no such disadvantage.

This is, basically, achieved by two innovations: firstly, the frequent change of the direction of the measurement current – which is indispensable for the measurement procedure used – can be perfor-

med with considerably increased velocity. This was achieved by the development of a fast current source which can be triggered digitally, and by the use of the most recent magnetic field sensors which have been developed at PTB and are based on super-conducting quantum interferometers. Secondly, a new detector with a higher bandwidth has been developed for the measurement of the bridge voltage, which amounts to a few nanovolts only. In addition, its disturbing backaction on the interferometer has been considerably reduced.

The improvements result in a four times lower uncertainty, which is reached in half the measurement time. At present, the system is combined with a new low-temperature magnet for the quantum Hall effect so that – after more than twenty years of successfully using the previous quantum resistance standard – PTB now has the latest standard of this type worldwide at its disposal.

Due to the cooperation with PTB, Magnicon GmbH in Hamburg – which developed and manufactured the required electronic components on behalf of PTB – is now able to offer both the highly sensitive fast voltage detector as an independent measuring instrument and the complete cryogenic current comparator systems.

The efficiency of the new system, which was demonstrated by PTB at an international technical conference, has already resulted in the first orders for the medium-sized enterprise.

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Scientific publications

Götz, M.; Drung, D.; Pesel, E.; Ahlers, F.-J.: Settling Behavior of the Bridge Voltage in Resistance Ratio Measurements with Cryogenic Current Comparators. IEEE Trans. Instrum. Meas. In print.

Drung, D.; Storm, J.-H.: Ultra-low Noise Chopper Amplifier with Low Input Charge Injection. IEEE Trans. Instrum. Meas. In print

Molar mass of silicon is determined

The novel method for determining extremely variable isotopic abundances of silicon is also suitable for the analysis of other elements

Within the scope of the Avogadro Project, PTB has measured – in a novel way – the molar mass of silicon with a relative measurement uncertainty of $1 \cdot 10^{-8}$. The mass-spectrometric method developed for this purpose is also interesting for the determination of isotopic distributions of other elements in analytical chemistry and in semiconductor technology.

Especially interesting for
- *analytical and physical chemistry*
- *semiconductor (particularly silicon) technology*

In order to determine the exact isotopic composition and – thus – the molar mass of the highly enriched ^{28}Si crystal, a novel measuring method was developed on a theoretical and experimental basis. Thereby, samples of the ^{28}Si material are wet-chemically converted quantitatively in a single step in alkaline silicate solutions. Then, the ratio of the isotopes ^{30}Si and ^{29}Si is determined, which are present in extremely small amounts in the "Avogadro crystal". In this way it is possible to avoid the very difficult direct determination of the ^{28}Si fraction.

The method is based on modified isotope dilution mass spectrometry (IDMS). The silicon sample of the "Avogadro crystal" was mixed with a second silicon sample – highly enriched in ^{30}Si – in such a way that the isotopes ^{30}Si and ^{29}Si were present in a ratio of about 1:1.

The resulting relative measurement uncertainty of less than $1 \cdot 10^{-8}$ could only be achieved through the use of a Multicollector Inductively Coupled Plasma Mass Spectrometer (MC-ICP-MS) with simultaneous ion current determination.

Both the ICP-MS and also the modified IDMS have for the first time been used for the determination of a molar mass. The method is generally suitable for the determination of the molar mass of elements with several isotopes which have one isotope with a heavy surplus or deficiency – as is, for example, also the case with calcium or strontium.

The novel exact correction factor determination for the ICP-MS is interesting for analytical chemistry. Possible industrial applications are, for example, purity determinations for semiconductor (particular silicon) technology.

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Scientific publications

Rienitz, O.; Pramann, A.; Schiel, D.: Novel concept for the mass spectrometric determination of absolute isotopic abundances with improved measurement uncertainty: Part 1 – theoretical derivation and feasibility study. Int. J. Mass Spectrom. 289 (2010) 47-53

Mana, G.; Rienitz, O.; Pramann, A.: Measurement equations for the determination of the Si molar mass by isotope dilution mass spectrometry. Metrologia 47 (2010) 460-463

Pramann, A.; Rienitz, O.; Schiel, D.; Güttler, B.: Novel concept for the mass spectrometric determination of absolute isotopic abundances with improved measurement uncertainty: Part 2 – development of an experimental procedure for the determination of the molar mass of silicon using MC-ICP-MS. Int. J. Mass Spectrom. 299 (2011), 78–86

Detecting breast cancer more reliably

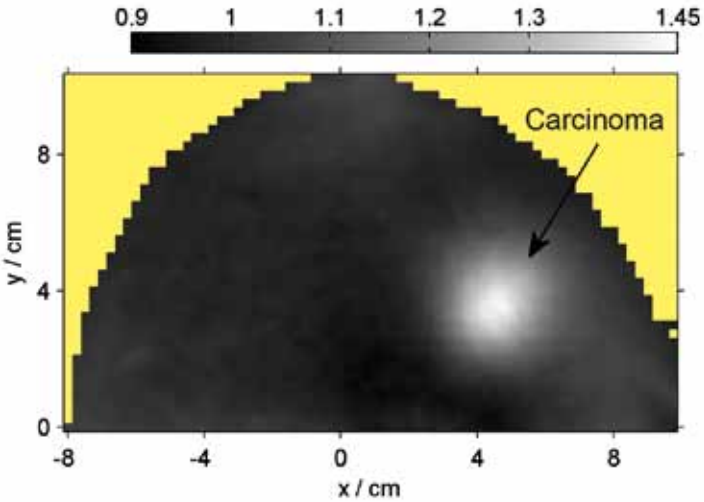
Optical measurement aids in differentiating between cancer and benign changes in breast tissue

PTB has developed a non-invasive measuring method with which small amounts of a fluorescent contrast agent can be detected in the breast. Initial patient trials have shown that malignant and benign changes in the breast tissue can be differentiated on the basis of vascular permeability.

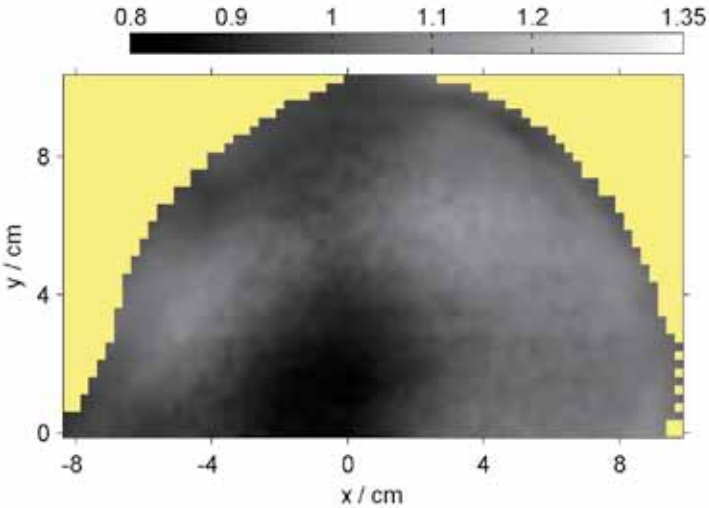
Especially interesting for
- *physicians*
- *patients*
- *manufacturers of medical devices*

Breast cancer. One in ten women is confronted with this diagnosis at a certain point in her life. The chances of being healed are good if the cancer is detected at an early stage. Therefore, preventive screenings are so important. On the other hand, benign changes shown on the X-ray mammogram are often falsely rated as “highly suspicious”. Then the patients must live with the stressful suspicion of having breast cancer, until a biopsy shows that it was a false alarm. Therefore, PTB, together with physicians of the Charité Berlin, have developed a measuring method that offers more certainty in differentiating between benign and malignant tumors. Fluorescence mammography using the contrast agent indocyanine green (ICG) could be used as a supplement to the standard methods and contribute to a reduction in the number of biopsies.

After being administered intravenously, ICG binds quickly and completely to plasma proteins in blood. The resulting macromolecules are too large for the small openings in the vessel walls of benign tumors. In malignant tumors, on the other hand, the blood vessel walls are much more permeable, so that the dye-labeled proteins can pass through the capillary walls and accumulate in the extracellular space. They remain there for a longer period, because decomposition in the tumor tissue is disrupted. If the time of measurement is chosen such that the ICG circulating in the blood vessels has already been washed out by the liver, then the portion of the contrast medium extravaseated into the carcinoma can be imaged.



Fluorescence mammogram of a patient with a carcinoma. It appears as a bright spot due to the locally increased fluorescence of the contrast agent.



Fluorescence mammogram of a patient with a benign lesion (fibroadenoma) in the left area of the image. This lesion does not exhibit any noticeable fluorescence, since there is no uptake of the contrast agent.

For detection, near-infrared laser radiation is used which causes the contrast agent to become fluorescent. This fluorescent light is measured with high sensitivity. Initial patient trials confirmed that an optical mammogram produced in this way does indeed only show an increased fluorescence contrast – i.e. an increased amount of contrast agent in tumorous tissue – if the tumor is malignant.

The prospects of this method must be extensively examined in further clinical studies. Thereby, it is particularly a matter of whether the vascular permeability can be quantitatively determined as a physiological parameter from the measured fluorescence contrast and thus be used for an improved diagnosis of breast cancer.

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Scientific publication
Hagen, A.; Grosenick, D.; Macdonald, R.; Rinneberg, H.; Burock, S.; Warnick, P.; Poellinger, A.; Schlag, P. M.: Late-fluorescence mammography assesses tumor capillary permeability and differentiates malignant from benign lesions. Opt. Expr. 17 (2009), 17016-17033

How large is “small”?

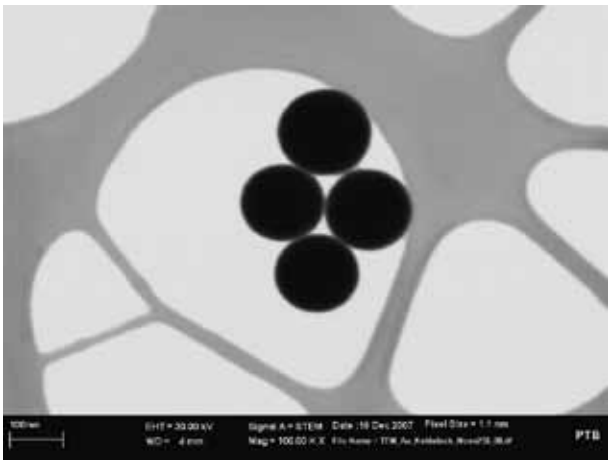
Traceable and accurate: electron-microscopic measurement procedure for nanoparticles

A scanning electron microscope with a transmission detector, simulation calculations for the signal contrast, and automated image analysis: by combining these three components, the size distribution of nanoparticles can be determined fast and accurately.

Especially interesting for
- *nanotechnology*
- *electron microscopy*
- *toxicology*

Nanoparticles often exhibit other properties than “large” particles made of the same material. This exactly is what makes them so interesting for various applications – but it also bears risks for man and the environment. To estimate the risks, and to advance the technical development, procedures to determine the exact size of these particles are important. PTB has therefore developed a new measurement procedure for nanoparticles which is based on a scanning electron microscope (SEM) equipped with an additional transmission detector. Such equipment brings about a solution to a general problem which occurs when spherical nanoparticles are measured with high precision: the precise determination of the particle boundary, which – even in high-resolution electron microscopic images – is somewhat “blurred”.

The question is: At which grey scale value does the particle begin, and which image pixel still belongs to the background? To determine this signal threshold level, a deeper understanding of how the image is created inside the electron microscope is necessary. The detector signal can be calculated by means of a Monte Carlo simulation which was developed at PTB and which takes into account the interactions of the electrons with the spherical particle and the properties of the detector. This has shown that the signal threshold level at the edge of the particle depends on the properties (material and size) of the particle.



Silica particles with a nominal diameter of 160 nm on a holey carbon film. The image was recorded with a scanning electron microscope equipped with a transmission detector. It measures the electrons which traverse the sample. The nanoparticles appear black because hardly any electrons traverse them; the background appears light-coloured.

To be able to take into account these two properties, an automatic image evaluation algorithm has been developed. It computes iteratively for each single particle an individual signal threshold level for the particle's edge based on the simulation results. This enables an accurate determination of the size, adapted to the corresponding particle. Despite the complexity of the procedure, hundreds of images can be analysed in less than an hour. In addition, the total time needed to fully characterise a nanoparticle sample is considerably reduced by the use of a newly developed routine which makes it possible to capture numerous images of nanoparticles.

Thanks to this new procedure, medium-sized particles of down to approx. 7 nm can be determined with low measurement uncertainties from 1 nm to 2 nm. The work was carried out within the framework of the European Metrology Research Project “Traceable characterization of nanoparticles” – in which also an extension of the procedure to non-spherical particles is aspired to. Within the scope of this project, also integral measurement procedures are developed by PTB in which light- and X-ray scattering at particle ensembles in an aqueous environment is measured in order to determine the size distribution of nanoparticles.

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Scientific publication
Buhr, E.; Senftleben, N.; Klein, T.; Bergmann, D.; Gnieser, D.; Frase, C. G.; Bosse, H.: Characterization of nanoparticles by scanning electron microscopy in transmission mode. Measurement Science and Technology, 20 (2009), 084025 (9p).

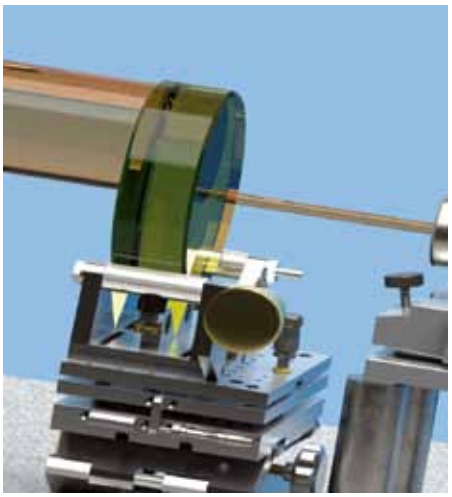
DKD Technical Committees go back to their roots

The future of the DKD Technical Committees is secured: PTB will take on the responsibility for this proven instrument of technical coordination between the calibration laboratories and PTB.

Due to the new regulation of the accreditation system in the EU Member States, as a result of which the accreditation body of the Deutscher Kalibrierdienst (DKD) was integrated into the Deutsche Akkreditierungsstelle (DAkkS) (German Accreditation Body), the role of and responsibility for the DKD Technical Committees were at first unclear. PTB therefore agreed to take on the patronage of the DKD Technical Committees, to supervise them technically and on an organisational basis, and – through its membership of EURAMET e.V. – to act as a link between the national and the European levels, e.g. in issuing calibration directives. As the DKD and also the DKD Technical Committees originally developed from PTB, the Technical Committees are thus going back to their roots.

Measuring the flatness of large surfaces

Licence partner wanted



Especially interesting for
- *the optical industry*
- *semiconductor technology*

Science and industry's need for measuring the flatness of increasingly larger optical surfaces is constantly growing. Test pieces with a diameter of 600 mm are no longer rare. Such large surfaces can now be measured with interferometers which are smaller than the surface itself. With the new rotation stitching, as many partial topographies as desired are taken, while the test piece to be measured is rotated. Through the special arrangement of the test piece, gravity's disturbing influences can moreover be eliminated, as measuring near the points of support can be avoided. The procedure is, thus, not only considerably cheaper than previous measuring methods, but also more accurate in such an application. Measurement uncertainties in the nanometer range are achievable.

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Thermal conductivity – with pinpoint accuracy

Licence partner wanted

Especially interesting for
- *the development of plastics*
- *the development of building products*

Determining the thermal diffusivity and thermal conductivity over an area of 10 mm^2 could be very attractive for various applications in material analysis, such as in the development of new materials or in building physics. So far it has been impossible to reliably and precisely determine these thermal transport properties on such small areas. The "Hotpoint-Sensor" developed at PTB can measure thermal conductivity with pinpoint accuracy as it can be flexibly adapted to each measurement requirement. A measurement takes only a few minutes and – with the newly developed software – can be precisely and quickly evaluated. Thanks to their simple design, “Hotpoint-Sensors” can be combined, so that the process can be extended to a complete measurement matrix of several measuring sensors.

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Mobile CCM for ultra-large components

Licence partner wanted

Especially interesting for
- *manufacturers of wind energy converters*
- *shipping and aviation*

Due to the desire for increasing capacities and long lifetimes, the requirements on the accuracy of components are growing for wind turbines and other ultra-large components in shipping and aviation.
With the mobile coordinate measuring machine (CMM) developed at PTB jointly with the University of Bremen, the manufacturing precision can be tested economically and with high precision in the machine itself. The measuring device itself has only small dimensions and consists of just a few high-precision components. This brings about a noticeable cost reduction, for instance, in the production of gears and other large-sized components.

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Awards

Ernst O. Göbel

The President of PTB and of the Committee of the International Metre Convention (Comité International des Poids et Mesures, CIPM) received the Grand Cross of Merit of the Federal Republic of Germany for his nationally and internationally recognized work, his outstanding degree of commitment as well as his mediatory contributions to the interface between science, the economy and politics. Göbel has, according to his laudation, played a personal part in PTB being a world-wide leading centre of competence of metrology.

Udo Gerlach, Ulrich Johannsmeyer and Thomas Uehlken

These three staff members of PTB's Department 3.6 Intrinsic Safety and Safety of Systems were awarded the Innovation Transfer Prize 2010 of Braunschweig's Chamber of Industry and Commerce for developing the new intrinsic safety concept "Power-i"/ DART and its successful technology transfer to industrial explosion protection.

Roman Schwartz

The Head of PTB Division 1 Mechanics and Acoustics was elected as the new Vice-President of the International Organization of Legal Metrology (OIML) at last year's meeting of the International Committee of Legal Metrology (CIML).

Stefanie Dencks

This staff member of PTB Department 1.6 Sound received a DEGUM Lecturing Prize from the Gesellschaft für Ultraschall in der Medizin e.V. (German Society for Ultrasound in Medicine) for her work "Monitoring of the formation of lesions in HIFU treatments on the basis of radiated shear waves".

Dates

5.–6.4.2011 Plenary Meeting CEN/TC 92r

Location: PTB Braunschweig, Sicherheitstechnik. Contact: Gudrun Wendt. Phone: +49 (0) 531 592-1500 Organizer: PTB Department 1.5

1.–6.5.2011 5th CCM International Conference on Pressure and Vacuum Metrology and 4th International Conference IMEKO TC 16

Location: Hotel Stuttgarter Hof, Berlin. Contacts: Karl Jousten, Margit Kleinsorge. Phone: +49 (0) 30 3481-7262, +49 (0) 30 3481-7276. Organizer: PTB Berlin

3.–6.5.2011 25th Control

International Trade Fair for Quality Assurance. Location: Messe Stuttgart (Stuttgart Exhibition Centre). PTB Stand: Hall 1, Stand No. 1313. Contact: Christine Haubold. Phone: +49 (0) 531 592-3007

18.–20.5.2011 Joint Dissemination and EURAMET DC-QM Meeting

Location: PTB Berlin; Helmholtz-Building. Contact: Katrin Volkmer. Phone: +49 (0) 531 592-2023. Organizer: Euramet

22.–26.5.2011 SPIE Konferenz Optical Metrology

20th International Congress on Photonics in Europe. Location: International Congress Centre, Munich. Contact: Bernd Bodermann. Phone: +49 (0) 531 592-4222. Organizer: SPIE Europe

23.–26.5.2011 Laser World of Photonics

Location: Neue Messe München (New Munich Trade Fair Centre). Contact: Heiko Klawitter (CC UPOB). Phone: +49 (0) 531 592-5131

28.5.2011 Long Night of Sciences

in Berlin and Potsdam with the presentation of the rebuilt Observatory in the Berlin Institute of PTB. Location: PTB Berlin. Contact: Frank Melchert. Phone: +49 (0) 30 3481-7446. Organizer: PTB

For further information: www.ptb.de > English Version > What's new > Calendar of events

Publications

PTB-Mitteilungen Issue 3/2010

Focus: Terahertz-Metrologie

PTB-Mitteilungen Issue 4/2010

Focus: Technology Offered for the Economy

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Imprint

PTBnews 3/2010, English edition, March 2011, ISSN 1611-163X
The PTBnews are the science newsletter of the Physikalisch-Technische Bundesanstalt (PTB). They are aimed at PTB's cooperation partners in the economy and science as well as anyone else who is interested. The PTBnews are published three times each year in a German as well as in an English edition and can be subscribed to free of charge – either in the printed version or as a pdf file or both.
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html and pdf versions: www.ptb.de > English Version > Publications > PTB-news
Publisher: Physikalisch-Technische Bundesanstalt (PTB), Braunschweig und Berlin
Editors: Franz Josef Ahlers, Peter Becker, Harald Bosse, Mathias Richter, Erika Schow, Jens Simon (chief editor), Florian Schubert, Peter Ulbig
Layout: Volker Großmann, Alberto Parra del Riego (concept)
Editorial Office: Press and Information Office, PTB, Bundesallee 100, D-38116 Braunschweig, phone +49 (0)531 592-3005, fax +49 (0)531 592-3008,
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