PTB news
Scientific Newsletter
2
2018

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

En route to the optical nuclear clock

The coupling of nucleus and electron allows spectroscopic investigation of thorium-229

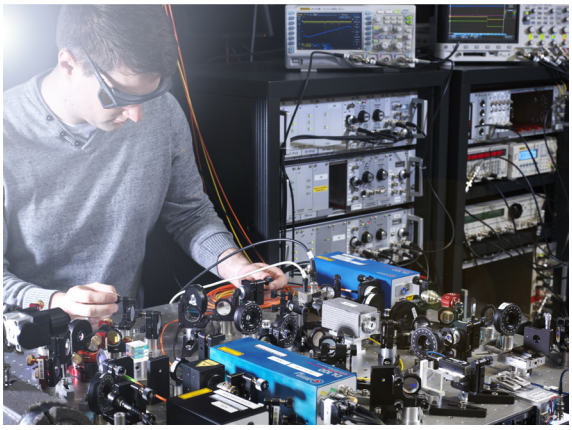
Especially interesting for
- fundamental research in physics
- developers of optical atomic clocks

The nucleus of thorium-229 possesses a property that is unique among all known nuclides: It should be possible to excite it with ultraviolet light. To date, little has been known about the low-energy state of the Th-229 nucleus that is responsible for this property. Now it has become possible for the first time to determine the charge distribution of this nuclear state by means of spectroscopic investigations of the electron shell. In this way, a laser excitation of the atomic nucleus can be monitored, thus allowing an optical nuclear clock to be realized.

As early as around 15 years ago, the concept of a new atomic clock with unique properties was being developed at PTB in Braunschweig: Instead of a transition frequency between two states in the electron shell being used as the pulse generator of their clock, as is the case in all atomic clocks in use today, they envisaged using a transition frequency in the nucleus. Because the protons and neutrons in the nucleus are packed more densely than the electrons in the shell by several orders of magnitude, they react less sensitively to outside disturbances that can change their transition frequencies – thus providing good conditions for a high-precision clock.

However, the frequencies of nuclear transitions are usually much higher (in the X-ray range); for this reason, they are unusable for atomic clocks. The sole known exception, and the foundation of PTB's proposal, is the nucleus of thorium-229, which has a transition in the frequency range of ultraviolet light. This transition is within the reach of laser technology that is similar to that used in present-day optical atomic clocks. More than ten research groups around the world are currently working on projects concerning the feasibility of a thorium-229 nuclear clock. In experimental terms, this issue has proven to be extremely difficult. For this reason, no success has been achieved thus far in observing the nuclear transition using optical methods. The resonance bandwidth is, as desired for the clock, very narrow, but its frequency is only roughly known for lack of experimental data. It therefore resembles the proverbial search for a needle in a haystack.

Within the scope of a cooperation project between PTB, Ludwig-Maximilians Universität (LMU) Munich, Johannes Gutenberg University Mainz, the Helmholtz Institute Mainz and GSI in Darmstadt, an important breakthrough has now been achieved: for the first time, it has been possible for basic properties



PTB scientist Johannes Thielking with the laser setup for measurements of the thorium-229 nuclear properties

such as the size and shape of the charge distribution to be measured in the excited state of the Th-229 nucleus. To this end, the Th-229 nuclei were not excited from their ground state (as will happen in the future in the clock); instead, in a device developed by LMU, they were obtained in the excited state from the alpha decay of uranium-233, slowed and stored as Th^{2+} ions in an ion trap. By means of laser systems developed at PTB for the spectroscopy of these ions, it was possible to measure transition frequencies in the electron shell accurately in order to derive information about the properties of the nucleus.

To date, models based solely on theory have not been able to predict the behavior of the structure of the Th-229 nucleus in this unusually low-energy transition. These models can now be refined by means of the experimental data obtained. Furthermore, because the structure of the electron shell is easier to measure using spectroscopy, it has become possible to use it to demonstrate a laser excitation of the nucleus. This, however, does not mean that search for the optical resonant frequency of the Th-229 nucleus as the needle in the haystack has been completed, but now at least, we know what the needle we are looking for actually looks like. ■

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Scientific publication
J. Thielking, M. V. Okhapkin, P. Glowacki, D. M. Meier, L. v. d. Wense, B. Seiferle, C. E. Düllmann, P. G. Thirolf, E. Peik: Laser spectroscopic characterization of the nuclear clock isomer 229mTh. Nature 556, 321–325 (2018)

Dynamic calibration of force transducers

Validating the calibration methodology with sine and shock force excitation

Especially interesting for
- manufacturers of force transducers
- calibration laboratories
- industrial force measuring techniques

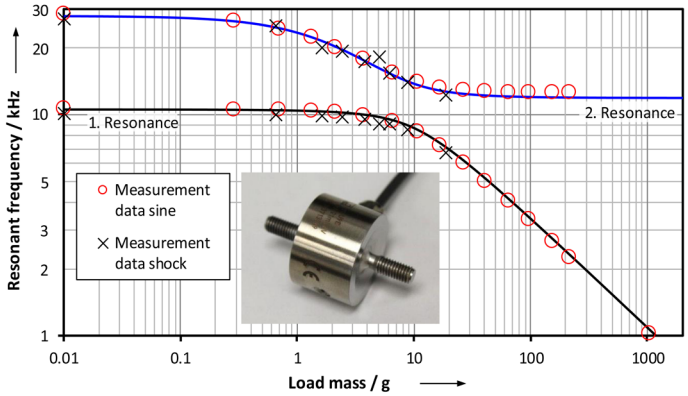
Good agreement between the results of different calibration methods (shock or sine) has been achieved for the first time. This was made possible by a mathematical model of a measuring setup with the force transducers elastically coupled on both sides.

The reliable measurement of dynamic forces plays an important role in industry. To provide the metrological infrastructure required, PTB has been pursuing an approach that describes the dynamic behavior of the force transducer by means of a mathematical model. The transducer and the calibration device are modeled as a series arrangement of mass-spring-damper elements. The mass, the stiffness and the damping parameters of the force transducer are identified applying the model equation to dynamic measurement data. The goal is the general characterization of the dynamic behavior, irrespective of the particular measuring application or the type of force excitation. Calibrations with shock or sine excitations should provide consistent parameters.

Earlier tests using a high-bandwidth force transducer did not give consistent results. A new model offers a thorough explanation. The force transducer used as a case study (measuring range ±1 kN, force introduction via two threaded rods) was subjected to shock or sine forces. Intended variations of the dynamic behavior could be realized by using additional load masses. The pulse duration ranged from 0.1 ms to 1 ms. The maximum excitation frequency was 30 kHz.

The force transducer has two dominant resonances whose characteristics depend on the size of the coupled mass. In the case of a typical shock excitation without load masses, the lowest resonant frequency is caused by the vibrating transducer housing, whereas it is due to the elastically coupled mass itself in the case of a sinusoidal excitation with high mass values. The new model of three elastically coupled masses considers a two-sided elastic coupling of the transducer. The resonant frequencies measured with the different measurement setups were compared with those of the model, and the stiffness parameters of the transducer were thus identified.

The improved model-based dynamic calibration now provides consistent parameters from measurement data with sinusoidal and pulse-shaped force excitation. This proves the suitability of this new calibration methodology. Complementary



Comparison of measured and modeled resonant frequencies of the force transducer investigated as a case study for dynamic calibrations with different load masses. The diagram shows the modeled resonant frequencies and the resonant frequencies determined experimentally with shock and sine excitation as a function of the load mass. The measurement point for 1 kg represents a typical sinusoidal calibration with large masses and excitations up to a few kHz. In addition, the resonances determined experimentally at shock excitations are also plotted.

investigations with finite-element methods have confirmed these results. The dynamic measurement behavior of the force transducer can therefore be transferred to a specific measurement application by extending the model correspondingly. ■

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Scientific publication
M. Kobusch, S. Eichstädt: A case study in model-based dynamic calibration of small strain gauge force transducers. ACTA IMEKO 6, 3–12 (2017)

Control of inorganic contaminants in pharmaceutical products traceable to the SI

High-accuracy monoelemental reference solutions for quality assurance in the pharmaceutical sector

Especially interesting for
- the pharmaceutical industry
- chemical analysis

According to a European quality guideline, elemental contaminations in medicinal products have to be adequately controlled. Consequently, the "European Directorate for the Quality of Medicines and HealthCare" (EDQM), the Joint Research Center (JRC) Geel, the Federal Institute for Materials Research and Testing (BAM) and the PTB have prepared high-accuracy reference solutions for four particularly toxic elements: lead, cadmium, mercury, and arsenic. Since January 2018, these solutions have been commercially available at the EDQM.

In the context of expanding globalization, it is becoming increasingly important to adequately control elemental impurities in medicinal products in order to avoid hazards to patients caused by toxic substances in medicines. Therefore, the European Medicines Agency (EMA) has adopted the internationally harmonized quality guideline ICH Q3D (EMA/CHMP/ICH/353369/2013). Since June 2016, new medicines have had to be checked for elemental impurities using instrumental analytics; as of December 2017, the same requirement has applied to products that have already been authorized.

In the past, the simplest accepted proof of elemental contaminants in medicines was obtained by using an unspecific sulfidic precipitation followed by a comparison of the chromaticity with that of a reference. The ICH guideline has been rendered legally binding in the 38 countries where the European Pharmacopoeia (Ph. Eur.) applies by incorporating it into the Ph. Eur. Conducting a risk-assessment-based quantification of contamination in a way that is traceable to the SI system is now required. This can be done, for instance, with analytical methods such as mass and optical emission spectrometry with inductively coupled plasma (ICP-MS or ICP OES). Traceability to the SI units means that the methods described above are calibrated by means of reference solutions whose mass fraction of the elements to be determined is traceable to the SI.

In 2016 and 2017, reference solutions with four particularly toxic elements, namely lead, cadmium, mercury, and arsenic, were produced within the framework of this cooperation project. PTB and BAM are able to prepare and characterize elemental solutions at an exceptionally high accuracy level by drawing on the expertise they have acquired over two decades. When characterizing the mass fraction of lead in the solution by means of ICP OES, PTB was, for example, able to achieve measurement uncertainties one hundred times smaller than those usually obtained in routine measurements.

The partners have agreed on the continuation of their successful collaboration. ■

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The European Guideline
EMA/CHMP/ICH/353369/2013: http://www.ema.europa.eu/docs/en_GB/document_library/Scientific_guideline/2015/01/WC500180284.pdf

Source of supply
https://crs.edqm.eu/

Reliable intelligent power grids

Procedures to model grid behavior and for safe data transmission within smart grids

Especially interesting for
- digitalization of the energy transition
- digital sensor networks

The more power that is generated by regenerative energy sources, the more complex the power grids become. This makes it ever more important to set up intelligent power grids where producers, consumers and measurement points communicate with each other. Within the scope of an EU project, mathematical modeling procedures have been developed to make the behavior of power grids more predictable and to create secure cryptographic methods.

The large number of suppliers and the fluctuating power supply from regenerative sources have transformed power grids into excessively complex systems. It has thus become ever more important to create "intelligent" power grids. In a so-called smart grid, intelligent power meters and other communicating elements ensure a constant exchange of information. It is the only way for suppliers and consumers to adapt their behavior to the current situation on the power market if need be. With the increasing levels of communication, it has become more and more important to ensure secure data transfer.

Owners of small-sized wind power, photovoltaic or CHP facilities do not often feed the power they produce into the high-voltage power system; instead, they feed it into the lower-level power grid, namely into the medium- or low-voltage grid. Too few measurement points are however available there to monitor grid behavior with sufficient accuracy.

Within the scope of the GridSens EU project, PTB, with several partners, has developed new mathematical methods to enable the current, the voltage and the phase angle to be predicted in medium- and low-voltage power grids. Both the time-dependent progress of the measured values and the fact that the characteristic of the current conductors might not be sufficiently known were taken into account. Applied to real grid data, it has been possible to demonstrate that the procedure could be used in practice.

To find out whether and how this procedure for the secure transmission of measurement and operating data could possibly influence the response speed of the measurement points, a test network including all the necessary infrastructure for secure data transmission was set up at the *Energieforschungszentrum Niedersachsen* (EFZN – Lower Saxony research center on energy). A newly developed generic model for the end-to-end protection of the data transfer has been implemented in the prototype of an intelligent measurement sensor. It has shown that the protection goals consisting of integrity, authenticity and privacy are achievable without the response speed being significantly influenced.

More reliable methods for the reconstruction of the grid state for typical medium- and low-voltage grids and validated procedures for the secure transmission of measurement and operating data are thus available. ■

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Scientific publication
S. Eichstädt, N. Makarava, C. Elster: On the evaluation of uncertainties for state estimation with the Kalman filter. Meas. Sci. Technol. 27, 125009 (2016)

Searching for sentinel lymph nodes

New optical procedure can replace nuclear medical method

Especially interesting for
- manufacturers of fluorescence-based medical equipment
- hospitals with a surgical oncology department

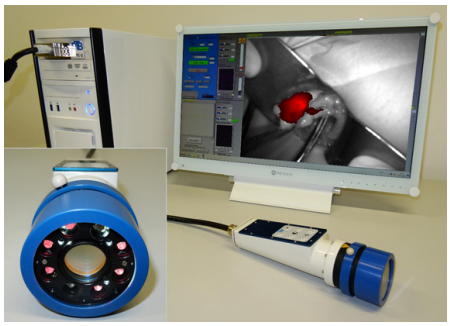
PTB has developed a camera system for sentinel lymph node mapping using the fluorescent contrast agent indocyanine green. The system has been successfully tested in collaboration with the Department of Gynecology of the Charité university hospital in Berlin.

When cancer starts spreading to other parts of the body via the lymphatic system, the cancerous cells first pass through the lymph nodes closest to the carcinoma. These nodes are therefore called sentinel lymph nodes. For various types of cancer, while the tumor is being surgically removed, the sentinel lymph nodes are also checked for cancerous cells. These lymph nodes are therefore removed and undergo microscopic examination. If no cancerous cells are found, lymph nodes located further from the carcinoma may remain in the body, so that the lymphatic system can carry on functioning to a large extent.

To date, a nuclear medical technique has been used in routine clinical practice to find the sentinel lymph nodes. Medical research has meanwhile shown that it is possible to use the fluorescent contrast agent indocyanine green instead, which could help to reduce the application of radioactive contrast agents. Detecting the fluorescence in the tissue requires a sensitive camera system.

Within the scope of a project funded by the Federal Ministry of Education and

Research (FKZ 03V0270), PTB has developed a camera system based on a small camera that does not necessitate cooling. This device can be handheld and can therefore be taken directly to the tissue to be examined. The fluorescence in the tissue is excited by means of light-emitting diodes (LEDs) arranged in a circle around the camera lens. Together with the fluorescence image, an anatomic image is taken in which the fluorescent area is highlighted in pseudo colors to show the physician where the sentinel lymph nodes are located.



Fluorescence camera system with camera head, electronic plug-in unit in the PC and control software showing a tissue section with fluorescent sentinel lymph nodes (highlighted in red). Detail screen bottom left: Arrangement of the LEDs in the camera head.

This camera system has been successfully tested within the scope of a feasibility study at the Department of Gynecology of the Charité university hospital, Campus Virchow, in Berlin. This study involved the examination of 16 female patients with carcinomas of the following types: vulvar, cervical, endometrial and ovarian cancer. It was demonstrated that the sensitivity of the camera system was sufficient to image both the lymph nodes themselves and the lymphatic vessels. ■

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Scientific publication
L. Szyc, S. Bonifer, A. Walter, U. Jagemann, D. Grosenick, R. Macdonald: Development of a handheld fluorescence imaging camera for intraoperative sentinel lymph node mapping, J. Biomed. Opt. 20, 51025 (2015)

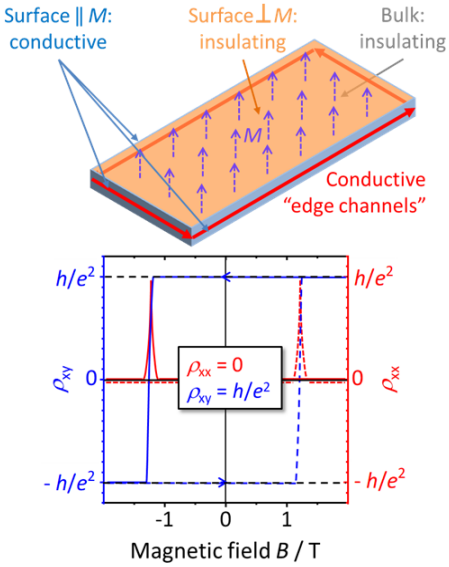
Resistance quantization at zero magnetic field

First precision measurement of the quantum anomalous Hall effect

Especially interesting for
- resistance metrology
- solid-state physics

In collaboration with the University of Würzburg, PTB has succeeded in measuring the quantum anomalous Hall effect with a relative measurement uncertainty of 2.5 · 10^{-7}. This represents an improvement by nearly three orders of magnitude. It brings the on-chip integration of quantum Hall resistance standards with Josephson voltage standards one step closer to reality, which would be a breakthrough for the practical use of electrical quantum standards.

The quantum anomalous Hall effect occurs in topological insulators. These are novel solid-state materials which were just discovered a few years ago and are conductive only at the surface. In the form of ferromagnetic thin layers, they exhibit a Hall resistance which is quantized at low temperatures without an external magnetic field; this Hall resistance has the value of the von Klitzing constant h/e^2. For the investigations carried out by PTB, the topological insulator used was a ferromagnetic vanadium-$(Bi_xSb_{1-x})_2Te_3$ mixed crystal which had been manufactured by molecular beam epitaxy (MBE) at the University of Würzburg. The anomalous Hall effect in this and similar materials and its theoretically predicted quantization without an external magnetic field had already been demonstrated for some time, but only with relative measurement uncertainties of approximately 10^{-4}. If the effect were truly accurate, it could lead to future components in electrical quantum metrology in which quantum Hall resistance standards could be combined on one chip with Josephson voltage standards which only operate at zero magnetic field. However, manufacturing these crystals by MBE is so difficult that worldwide only very few research groups have mastered it. Moreover, the ferromagnetic state has been so sensitive up to now that, contrary



A topological insulator insulates in the bulk but is conductive at the surface. Ferromagnetic doping also makes the surface perpendicular to the direction of magnetization M insulating. One-dimensional "edge channels", i.e. electric ring currents, run at the edge of the film. In this state, the Hall resistance ρ_{xy} is quantized at zero magnetic field and takes the value of the von Klitzing constant h/e^2, while at the same time, the longitudinal conductivity ρ_{xx} becomes zero.

to theoretical estimates, quantization occurs only at extremely low temperatures in the millikelvin range and at very small measuring currents of a few nanoamperes.

In cooperation with the Würzburg research group, quantization at zero magnetic field has now been demonstrated at currents of a few nanoamperes with a measurement uncertainty of $2.5 \cdot 10^{-7}$; this uncertainty is lower by nearly three orders of magnitude than previously obtained values. The measurements were made with the cryogenic current comparator bridge developed at PTB, which had already proven its value during the demonstration of the precise quantization of the fractional quantum Hall effect (see PTB-News, issue 1.2018).

The result obtained underpins the hope that the quantum anomalous Hall effect at zero magnetic field may in the future lead to electrical quantum standards that are simpler to operate, despite all the details of the effect still not being completely understood. ■

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Scientific publication
M. Götz, K. M. Fijalkowski, E. Pesel, M. Hartl, S. Schreyeck, M. Winnerlein, S. Grauer, H. Scherer, K. Brunner, Ch. Gould, F. J. Ahlers, L. W. Molenkamp: Precision measurement of the quantized anomalous Hall resistance at zero magnetic field. Appl. Phys. Lett. 112, *072102 (2018)*

Calibration certificates go digital

Considerable added value through easy use of all calibration data at a later stage

Especially interesting for
- the fundamentals of digital metrology
- calibration laboratories
- quality management and certification

Conventional calibration certificates could soon become history. To furnish proof that a measuring instrument has been calibrated and of how this has been done, metrology institutes throughout the world could, in the near future, use digital calibration certificates (DCCs) rather than their analog version. Especially the fact that they are machine-readable speaks in favour of DCCs for production and quality monitoring processes in which digitalization is becoming ever more important. The ultimate goal consists in developing universal DCC exchange formats valid for all areas of metrology.

In industrial production, the quality of products can only be guaranteed if the measuring equipment used is calibrated at regular intervals – i.e. if it is traced directly or indirectly to the national standards. Ensuring that this is possible is one of the central tasks of national metrology institutes. In this context, calibration certificates play a decisive role in each metrological quality management system.

But digital calibration certificates can do more than just serve as a proof of metrological traceability. Since the newly developed DCC is based on the internationally accepted and approved exchange format XML (Extensible Markup Language), it is machine-readable; moreover all indications, including the numerical calibration curves, can be directly and automatically transferred into all digitally supported processes. At the same time, cryptographic signatures are used as security procedures to guarantee that the integrity and the authenticity of a calibration certificate is still ensured. The cryptographic procedures used for DCCs have proven their worth in other areas such as the civil register office ("Standesamt"), waste management or in the purchasing department of the German federal administration.

The digital calibration certificate is already being further developed: a digital twin could contain even more data and software and would thus enable the measurement process to be simulated. Physical weights already have such digital twins which have been successfully tested. This "digital weight" contains both information from calibrations and estimations concerning the expected behaviour of the weight under certain ambient conditions. ■

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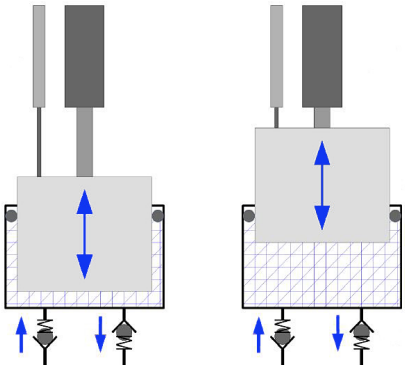
Scientific publication
S. Hackel, F. Härtig, J. Hornig, T. Wiedenhöfer: The digital calibration certificate. PTB-Mitteilungen 127 *(4), 75–81 (2017)*

Pulsation-free fluid flow

Especially interesting for
- the chemical and pharmaceutical industry
- flow rate test rigs in testing and calibration laboratories

Flow rate measuring technology requires exact fluid delivery for testing and calibration rigs. These systems should be able to continuously deliver constant volume flow rates. PTB has developed a new device consisting of two synchronized piston systems which can generate a constant and pulsation-free fluid flow.



Two pistons generate a constant volume rate by alternately pushing the fluid out of the respective cylinder.

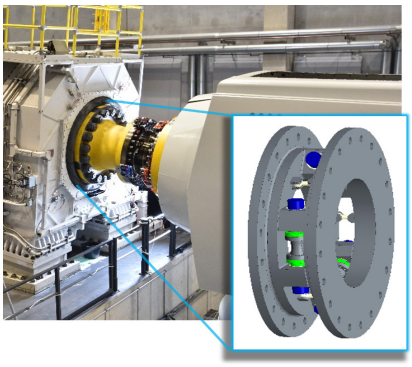
In addition, the flow rate can be varied quickly. Optical positioning provides exact knowledge of the flow rate at any time. Furthermore, the required operating pressure can be achieved without control valves. This requires less assembly space and thus saves costs and space. (Technology Offer 0470) ■

Advantages
- *exact flow rate known at any time*
- *continuous flow rate that can be varied quickly*
- *reduced assembly space and thus cost-effective*

Force lever system

Especially interesting for
- operators of torque measuring facilities
- operators of nacelle test stands

The traceable measurement of torques of more than 2 MN · m is of paramount importance in areas such as wind energy. An invention developed at PTB enables traceability by segmenting the torque into several force signals that are measured over a defined lever length. In addition, the torque sensor can decouple most of the mechanical disturbing components.



Flange with support to accommodate force sensors. Background: typical nacelle test stand. PTB's new force lever system can be mounted here.

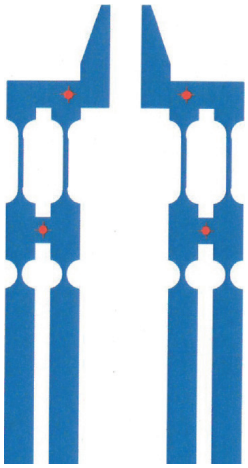
The measurement uncertainty amounts to a maximum of 1 %. (Technology Offer 0470) ■

Advantages
- *decoupled design to prevent disturbances due to shearing forces or bending moments*
- *Improved measurement uncertainty of max. 1 %*
- *traceable measuring chain*

Measuring gripping force

Especially interesting for
- medical research for cell diagnostics and cell manipulation
- keyhole surgery, in vitro fertilization

The handling and exact controlling of microgrippers requires high precision. The measuring devices used to date are characterized by great manufacturing effort and only allow a limited selection of materials to be used. PTB's concept simplifies the measurement of the gripping force: the grippers are segmented and interconnected by means of spring elements to reduce their stiffness. Markers are applied to both segments; they are then optically recorded by a camera. This allows the gripping force to be measured accurately without applying strain gauges or microelectromechanical systems (MEMS). (Technology Offer 0436) ■



Segmented gripper jaw with markers (red) to measure the gripping force

Advantages
- *gripping force measurable without strain gauges or MEMS*
- *optical image assessment*
- *low manufacturing effort*

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Awards

Dietmar Drung, Hansjörg Scherer, Martin Götz, Eckart Pesel
These staff members of departments 7.2, *Cryophysics and Spectrometry,* (D. Drung) and 2.6, *Electrical Quantum Metrology,* (H. Scherer, M. Götz, E. Pesel) have won this year's Helmholtz Prize in the category "Precision measurements in applied metrology". They have been given this award for the development of a new current amplifier, the Ultrastable Low-Noise Current Amplifier (ULCA) and the associated current comparator which enables the traceable and highly accurate calibration of the ULCA. The Helmholtz Prize is the most significant German metrology prize; it is awarded for exceptional work in the field of high-accuracy measurement in physics, chemistry and medicine.

Ekkehard Peik

The head of department 4.4, *Time and Frequency*, was awarded the European Frequency and Time Award 2018 at the 32nd European Frequency and Time Forum EFTF 2018 in Turin because of his "seminal contributions to single-ion optical frequency standards and high-precision spectroscopy thereby establishing most stringent limits on possible variations of fundamental constants".

Raul Garcia Diez

This staff member of the Helmholtz-Zentrum Berlin (and a former doctoral candidate at PTB) was awarded the Adlershof Dissertation Prize 2017 by the Humboldt-Universität zu Berlin, IGAFA (the scientific network representing nine non-university research institutions located in the technology park Berlin-Adlershof), and WISTA-MANAGEMENT GMBH for his doctoral dissertation which he produced at PTB's BESSY II laboratory. Raul Garcia Diez defended his thesis, "Characterization of nanoparticles by continuous contrast variation in small-angle X-ray scattering", which received a summa cum laude distinction at the Technische Universität Berlin in April 2017.

Jan-Hendrik Hagemann

This member of staff of department 4.2, *Imaging and Wave Optics*, has won the first Young Scientists Award given by the Competence Center Ultrapräzise Oberflächenbearbeitung (image: CC UPOB e.V.). The prize was awarded for the first time at this year's 9th High-Level Expert Meeting Asphere Metrology 2018.

PTB Annual Report relaunched

The PTB Annual Report has been fully revamped: its recently published 2017 issue is briefer, more topical, brighter and more informative. You can find it on PTB's website at "Press & What's new" > "Journals & magazines".
We can send you a free print version if you like; all you have to do is place an order by e-mailing presse@ptb.de.

PTB-Mitteilungen now available as an open-access magazine

PTB's journal, "PTB-Mitteilungen", is changing too. What used to be a professional publication that was paid for, has now become an open-access publication which is available to anyone interested.
You can find the PTB-Mitteilungen on PTB's website at "Press & What's new" > "Journals & magazines". Digitalization has changed the delivery form, but the release schedule should remain unchanged (at the end of each quarter).
We would be pleased to inform you about newly published issues by e-mail if you register for our newsletter. Just send a short e-mail to presse@ptb.de with "PTB-Mitteilungen" as the subject line.

World Metrology Day: the countdown has begun

This year's World Metrology Day (on 20 May) marks the start of the countdown to the introduction of the new International System of Units (SI). The new – or rather revised – SI, in which all units are traced to fundamental constants, will in all likelihood be adopted by the CGPM in November 2018. The revised SI will then officially come into force on next year's World Metrology Day (20 May 2019).
This "revolution in the system of units" is a huge challenge for all communicators. PTB's Press & Information Office has therefore given the new SI a warm welcome with a short animation showing the way from "natural feet" to "natural constants". You can watch this on PTB's Youtube channel: www.youtube.ptb.de

Imprint
PTB News 2/2018, English edition, Issue July 2018, ISSN 1611-163X
The PTB News is published three times each year in a German as well as in an English edition and can be subscribed to free of charge.
Subscription form: www.ptb.de > English Version > Publications > PTB News > Subscribe the PTB News
Publisher: Physikalisch-Technische Bundesanstalt (PTB), Braunschweig and Berlin
Editors: Andreas Barthel, Ludger Koenders, Christoph Kolbisch, Christian Lisdat, Mathias Richter, Hansjörg Scherer, Erika Schow, Jens Simon (responsible)
Layout: Volker Großmann, Alberto Parra del Riego (concept)
Translation: PTB Translation Office (Cécile Charvieux, Monika Hohlrüther)
Editorial Office: Press and Information Office, PTB, Bundesallee 100, D-38116 Braunschweig,
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Federal Ministry for Economic Affairs and Energy

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