PTBnews

Scientific Newsletter

1 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.

Recognizing nanostructures

Small-angle X-ray scattering on subminiature lithographic measuring fields

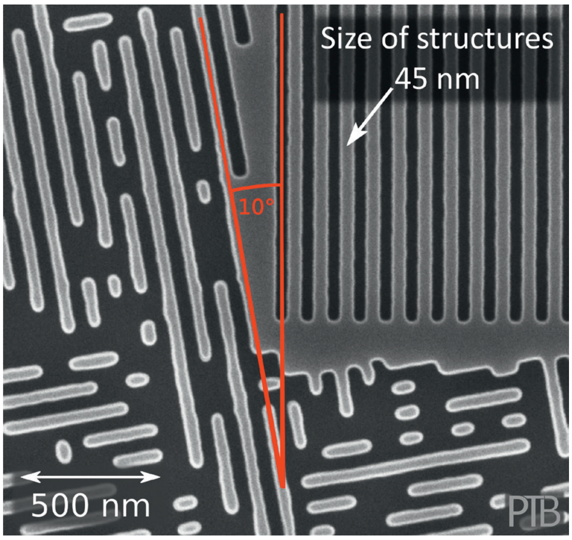
Especially interesting for
- nanometrology
- the semiconductor industry

Photolithography is used in industry to manufacture semiconductor elements. Hereby, the nanostructures of the logic gates of a photomask are imaged optically on the semiconductor wafer. For process control, measuring fields consisting of nanostructures in a periodical arrangement are distributed at different points over the entire photomask. The space and funds available being limited, these measuring fields are mostly no larger than 50 µm × 50 µm. Their lithographically processed image on the wafer can then be assessed using a microscope. Working towards quality control applications during the production process, PTB researchers at PTB's laboratory at the BESSY II electron storage ring in Berlin-Adlershof have succeeded in characterizing such measuring fields for the first time by means of small-angle X-ray scattering in a reflection geometry.

Since it is fast and provides high resolution, small-angle X-ray scattering (SAXS) is, in principle, an ideal measurement method to reconstruct periodically arranged nanostructures. It is thus considered when it comes to process-related metrology in the semiconductor industry. Due to X-ray absorption, measurements in a transmission geometry in which the incident X-ray beam penetrates the wafer are only possible with very thin wafers. It is possible to bypass this problem using SAXS in a reflection geometry under grazing incidence (GISAXS). However, due to the very small angles of incidence presupposed by this method, the projection of the incident X-ray beam onto the sample is prolonged in such a way that the scattering signals of the tiny measuring fields to be checked overlap with those from the surrounding nanostructured arrays. This is the reason why GISAXS has so far been rejected as an alternative.

However, the problem of the overlapping signals has now been bypassed by means of a simple work-around, namely by slightly rotating the symmetry axis of the measuring field with reference to the surrounding nanostructures. The scattering signals then occur in different solid angle ranges and can easily be distinguished spatially when identified by means of an area detector. In this way, it has been possible to measure fields with edge lengths of a few micrometers without overlapping interfering signals from their environment.

Corresponding test structures for the new procedure have been developed within the scope of a cooperation project with the Helmholtz-Zentrum Berlin



SEM image of a lithographic test structure: the periodical lines of the measuring field with dimensions of 4 µm × 4 µm (top right) are rotated by 10° with reference to the symmetry axis of the surrounding irregular nanostructures to be able to spatially separate the respective X-ray signals from each other.

and manufactured using electron-beam lithography. These structures have been successfully characterized at PTB by means of GISAXS. The new method's potential for industrial applications to photomask metrology in the manufacturing of semiconductors has also been demonstrated by scattering experiments with extreme ultraviolet radiation (EUV scatterometry), so that a patent for this procedure is pending. ■

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Scientific publication
M. Pflüger, V. Soltwisch, J. Probst, F. Scholze, M. Krumrey: Grazing-incidence small-angle X-ray scattering (GISAXS) on small periodic targets using large beams. IUCrJ 4, 431–438 (2017)

Thermometer for nanocircuits

Magnetic tunnel junctions enable absolute, time-resolved temperature measurements of nanocircuits

Especially interesting for
- manufacturers of magnetic sensors
- thermal investigations on nanocircuits

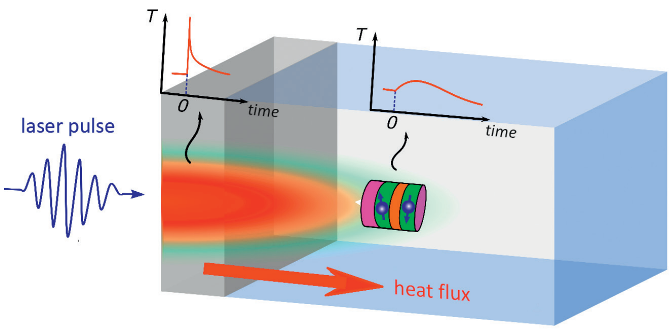
PTB has developed a method that can be used to manage the temperature of nanocircuits. The method is based on magnetic tunnel junctions and enables quantitative temperature measurements with a time resolution smaller than a nanosecond. Hereby, the tunnel junction acts as a calibrated thermometer by exploiting the temperature dependence of the tunnel resistance. The principle has been demonstrated at PTB for the time-resolved measurement of laser-induced increases in temperature; this principle can be used in numerous nanocircuits.

Given the rate at which technological advances have been progressing over the past few decades, many components containing nanocircuits have been created. Due to ever smaller dimensions and the associated high current densities, it is becoming increasingly important to monitor the temperature evolution in these components. Insufficient heat dissipation can modify or even destroy nanocircuits.

A procedure has been developed at PTB to measure the absolute temperature in nanostructures with a time resolution in the subnanosecond range. Hereby, the nanostructure is integrated into a magnetic tunnel junction. This junction is composed of two magnetic layers that are separated by a thin oxide layer. The tunnel resistance strongly depends on whether these layers are magnetized parallel or antiparallel to each other. A change in the magnetization direction can modify the resistance by more than 100 %. Due to complex physical effects, this change in resistance is temperature-dependent and decreases with increasing temperatures. The tunnel junction can therefore act as a fast thermometer by reading out its electric resistance.

To demonstrate this principle, a tunnel junction was integrated into a series of nanolayers and the temperature-induced change in the tunnel resistance was then calibrated first. For this purpose, a known temperature was adjusted by means of an electric heater. This calibration allowed the average change in the tunnel resistance, which occurred due to the heating of the nanolayers with a short laser pulse, to be converted into a temperature. In a layer lying more than 100 nm below the sample surface, a pulse train from a femtosecond laser with a pulse energy of 5 nJ and a repetition rate of 76 MHz leads to a mean temperature increase of 80 K. In addition, a very fast readout of the tunnel junction allowed the absolute temperature evolution to be determined with time resolution. This showed that in addition to the mean temperature increase mentioned, each laser pulse also caused a temperature peak. Approximately 4 ns after the laser pulse's impact on the sample surface, this fast temporal temperature increase attains its maximum of 2 K at the tunnel junction.

Such absolute, time-resolved temperature measurements in nanostructures that lie several 100 nm below the surface of a component could, in the future, be used to validate heat transport simulations, which makes them an important method to manage the temperature of nanocircuits. ■



Principle of absolute, time-resolved temperature measurement The optically induced increase in temperature is read out with subnanosecond time resolution by means of a magnetic tunnel junction which is located inside a nanostructure.

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Scientific publication
H. F. Yang, X. K. Hu, N. Liebing, T. Böhnert, J. D. Costa, M. Tarequzzaman, R. Ferreira, S. Sievers, M. Bieler, H. W. Schumacher: Electrical measurement of absolute temperature and temperature transients in a buried nanostructure under ultrafast optical heating. Appl. Phys. Lett. 110, 232403 (2017)

Detection limit of magnetic nanoparticles improved

A newly developed pick-up coil makes magnetic particle imaging even more efficient

Especially interesting for
- biomedical applications and research
- magnetic particle imaging (MPI) technology
- (magnetic) nanoparticle research

In cooperation with industrial partners, PTB has developed a pick-up coil for a novel imaging method called magnetic particle imaging (MPI). This coil has already been successfully implemented and tested at the MPI system in Berlin. The new coil considerably improves the sensitivity of the technique. Quantities of magnetic nanoparticles as small as a few nanograms can now be detected.

Magnetic nanoparticles exhibit unique magnetic properties which are utilized in a number of biomedical applications, both therapeutic and diagnostic. The tracking of cells or medicine inside the body is an example of this type of application. The particle surface can be modified in such a way that the particles bind to certain cells. These particles' magnetic properties allow them to interact in a non-invasive way; they can thus be used in various manners. One of these possibilities is magnetic particle imaging (MPI), a quantitative, radiation-free medical imaging method which exploits the physical properties of magnetic nanoparticles for diagnostics. After such nanoparticles have been introduced into the body, they can be excited by means of magnetic (alternating) fields. The distribution of the nanoparticles can eventually be reconstructed with millimeter precision from the measured signal. A great advantage of MPI compared to other imaging procedures is that not only are the nanoparticles represented, but they can also be quantified at each image point. Furthermore, the procedure enables dynamic images with a temporal resolution of up to 21 ms. These are two considerable advantages for functional biomedical applications.

The decisive aspect for most application fields is the sensitivity of the detection system to exceedingly small amounts of magnetic nanoparticles. In this context, the sensor plays a key role in realizing the full potential of the technique. In cooperation with Bruker, PTB in Berlin has developed a new pick-up coil for MPI which ensures enhanced signal acquisition. In order to characterize this coil, a prototype was installed and tested at the institute's own MPI scanner at the Rudolf Virchow Hospital in Berlin. Direct comparison with the previous transmit-receive hardware system shows that the new receive-only coil is four times as sensitive as the previous design, and offers enhanced suppression of interfering noise. This allows nanoparticle quantities as small as a few nanograms to be detected, i.e. the detection limit has been improved by one order of magnitude.

The findings obtained by PTB through this prototype have decisively supported the future potential of MPI to be used in preclinical research. ■

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Scientific publication
J. Wells, H. Paysen, O. Kosch, N. Löwa, F. Schmitzberger, M. Makowski, J. Franke, L. Trahms, F. Wiekhorst: Characterizing a preclinical magnetic particle imaging system with separate pickup coil. IEEE Transactions on Magnetics 53:1–5 (2017)

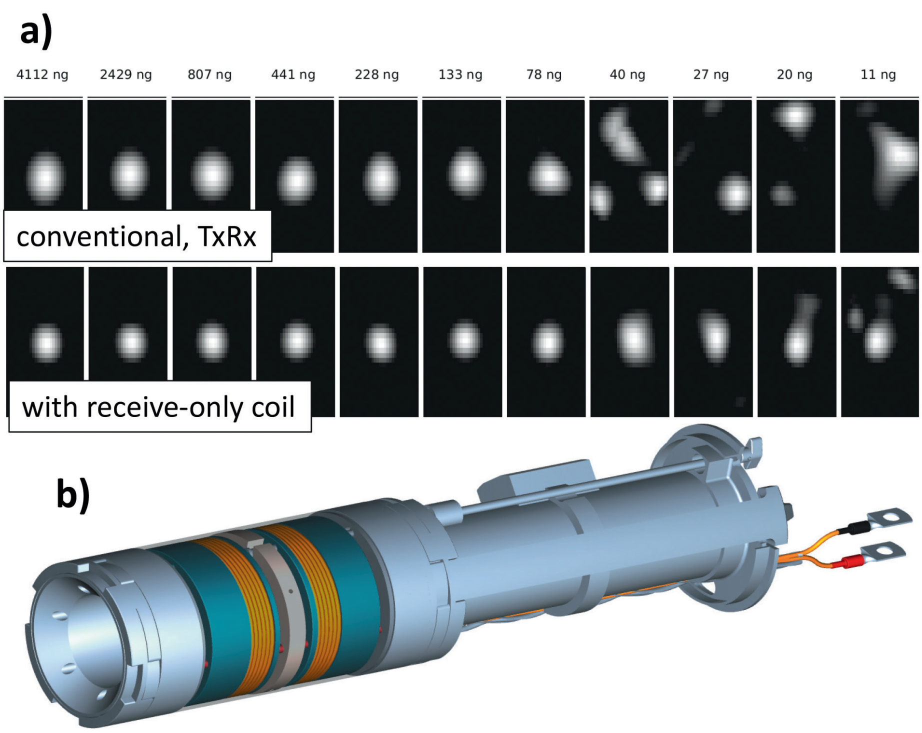


Figure: a) Reconstructions of varying nanoparticle quantities, each measured with a conventional transmit-receive coil system (top) and with the receive-only coil (bottom). The red dots represent the sample positions. b) Product layout of an optimized 1D receive-only coil

Experimental test of the fractional quantum Hall effect

Resistance quantization demonstrated with great accuracy

Especially interesting for
- resistance metrology
- solid-state physics

The "new SI", scheduled for May 2019, is based on fundamental constants which can be directly measured by means of macroscopic quantum effects. Verifying the theoretical fundaments of these effects experimentally with the greatest accuracy is one of the core tasks of metrology. In the case of the quantum Hall effect, this has now been successfully done for the first time for a state in which the current is no longer carried by electrons, but by quasi-particles of magnetic flux quanta and electrons.

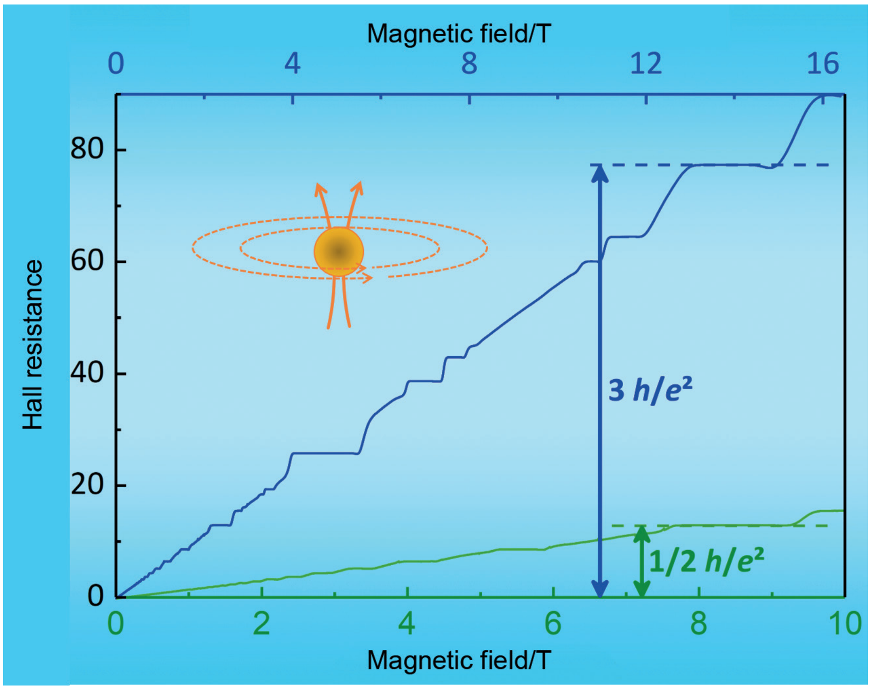
In the case of the quantum Hall effect (QHE), electric resistance only depends on the values of Planck's constant h and of the elementary charge e. In the case of the Josephson effect, these two constants (h and e) also play a decisive role – in this case, however, for the realization of electric voltages. Since h will also be the basis for the future definition of the kilogram, the two electric quantum effects play a key role in the new system of units (SI) which is based on fundamental constants.

The importance of the QHE is essentially due to the theoretically predicted universality with which resistance values are quantized in certain ranges of magnetic fields. In the case of the "regular" (integer) QHE, the quantized resistance values amount to $1/i\ R_K$, where i is an integer and the von Klitzing constant is $R_K = h/e^2$.

High-precision measurements were carried out at PTB for the first time in a special regime of the QHE where the current is no longer carried by electrons but by quasi-particles formed by a "cluster" of electrons and magnetic flux quanta. The charge of these quasi-particles designated as "composite fermions" corresponds to rational fractions of the elementary charge. In this exotic regime, the QHE exhibits quantized resistance values given by rational fractions of the von Klitzing constant and is therefore called fractional QHE.

Measuring this resistance accurately requires extremely pure semiconductor samples in which the composite particles form at very low temperatures of a few hundredths of degrees above absolute zero and at even higher magnetic fields than for the integer QHE. It has been possible to achieve these experimental conditions for several years. The limitation of the current used for the measurement to less than a hundredth of the usual current is much more critical. If the current is too high, the composite fermions will basically "melt" during the measurement; if the current is too low, the measurement uncertainties can become very high.

To solve this problem, PTB's improvement of electrical resistance bridges based on cryogenic current comparators (CCC) was crucial. Considerable improvements of the bridge now allow the achievement of relative measurement uncertainties of a few parts in 10^8 even at currents in the nanoampere range. The universality of resistance quantization could thus be demonstrated also in the fractional QHE regime with a relative uncertainty of $6.3 \cdot 10^{-8}$. ▪



In certain magnetic field ranges, fractional (blue curve) and integer QHE lead to resistance values that are only given by the fundamental constants h and e, and by a rational number. The ratio of 1 : 6, which had been predicted theoretically for the two resistance values characterized by the two arrows, has been experimentally confirmed for the first time with a measurement uncertainty of a few parts in 10^8. The inserted image is a schematic representation of the composite fermion in this fractional QHE regime. The quasi-particle consists of an electron to which two magnetic flux quanta are bound; it carries the charge $e/3$.

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Scientific publication
F. J. Ahlers, M. Götz, K. Pierz: Direct comparison of fractional and integer quantized Hall resistance. Metrologia 54, 516–523 (2017)

Atomic friction effects

Investigations with laser-cooled ions prove experimentally that topological defects help atomic chains slide more easily

Especially interesting for
- biology
- nanotechnology
- complex, self-organized systems

Investigations carried out at PTB's QUEST Institute in a model system consisting of ion chains sliding over each other have provided fundamental findings on friction in nanostructures and biomolecules. In the event of defects in the lattice structure, a transition toward superlubricity has been observed and explained.

Friction is encountered both in everyday life and in technical processes where its influence can be either positive (i.e. when it enables wheel drive) or negative (e.g. as a cause for wear). Atomic friction, as encountered in applications such as nanomachines or biomolecules, however, is very difficult to access, so that little is known about it. Contrary to macroscopic objects, which are atomically speaking rough-textured and only touch each other where irregularities occur, in the universe of miniature objects, smooth surfaces are superposed. It is therefore necessary to take the contact area into account within the scope of model computations. Such models predict new, fascinating phenomena – such as superlubricity – where static friction is nearly entirely absent.

To measure friction exactly, a powerful instrument already exists, namely the friction force microscope. In contrast, the dynamics of two frictional systems cannot be observed directly; instead, it requires a work-around in the form of a model system consisting of elements which behave as similarly as possible. Such a system has been developed at the QUEST Institute at PTB, in collaboration with the University of Sydney.

The core piece of this system consists of ytterbium ions that are trapped in an ion trap and are cooled down to a few millikelvins by means of lasers until they form a two-dimensional crystal composed of two superposed ion chains. These ion chains are coupled by the Coulomb interaction. If the ions are irradiated with laser light whose frequency is close to their resonant frequency, they start fluorescing and can be moved in a targeted way by means of light pressure. Using high-resolution imaging optics, the individual atomic particles can be observed in their motion.

If the periodicity of the chain arrangement is broken by a topological defect, a fascinating multi-particle effect occurs, leading to a phase transition at which static friction ceases to exist (see diagram). This so-called Aubry transition was predicted as early as in the 1980s but could not be experimentally measured for another 30 years. It has now been possible to observe atomic chains rubbing against each other for the first time with atomic-scale resolution.

The dynamics of the ion chains is comparable to that of molecule chains such as those occurring in DNA. In these chains, defects may cause the proteins to break apart. This new physical model system is generally suited for investigations of the complex, nonlinear dynamics of friction in one-, two- or three-dimensional systems with atomic-scale resolution. Further cooling levels will allow transport phenomena occurring in the quantum universe to be investigated. ■

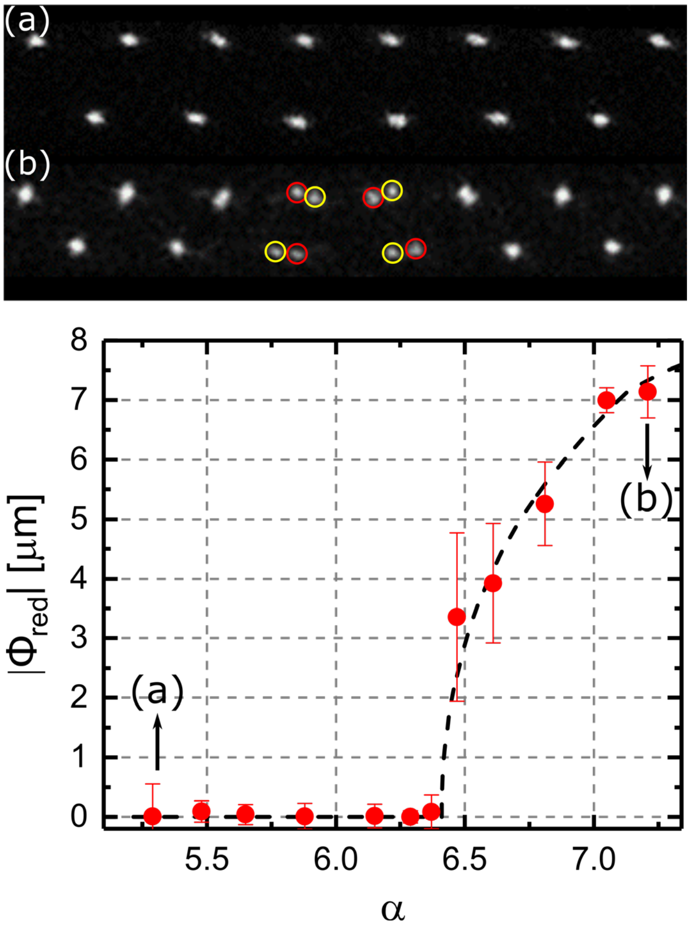

Image of the atoms in the ionic crystal when the interaction inside a chain is higher (a) or smaller (b) than between the two chains. A phase transition occurs between the two regimes; this phase transition leads to a break in the symmetry in finite systems (see red and yellow configurations). The graph below represents the measured curve of the order parameter Φ (a measure of the symmetry in the system). α is an experimental parameter which determines the distance between the two chains.

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Scientific publication
J. Kiethe, R. Nigmatullin, D. Kalincev, T. Schmirander, T. E. Mehlstäubler: Probing nanofriction and Aubry-type signatures
in a finite self-organized system.
Nat. Commun. 8 15364 (2017)

The case for ultrasonic measurements

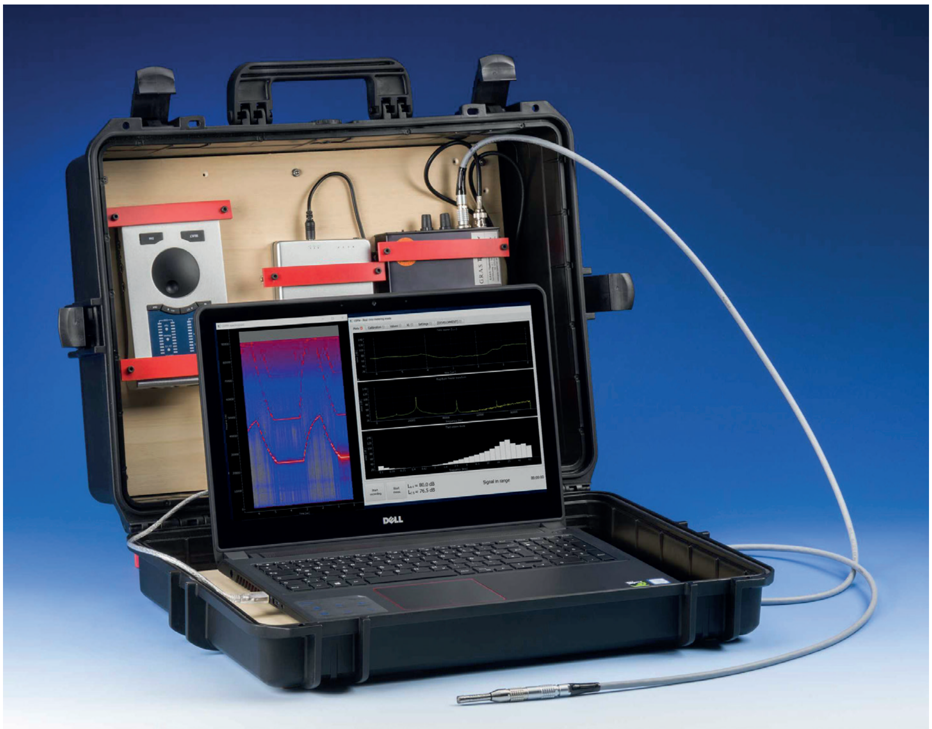
Mobile ultrasound level meter for practical use in occupational health and safety

Especially interesting for
- occupational health and safety activities in working environments involving ultrasonic technologies

In cooperation with the Institut für Arbeitsschutz (IFA) of the Deutsche Gesetzliche Unfallversicherung (DGUV), the prototype of a portable measuring system for airborne ultrasound has been developed at PTB. It will allow the determination and assessment of potential workplace risks represented by widespread ultrasonic machines which can generate very high sound pressure levels.

Ultrasound is an increasingly used technique in various areas such as welding, cleaning or cutting. When using ultrasound, the sound pressure levels can sometimes be very high. According to the German regulation on protection against noise and vibrations at the workplace (Lärm- und Vibrations-Arbeitsschutzverordnung), the risks also need to be assessed reliably at workplaces involving the use of ultrasound. Currently available sound level meters are, however, only suited to a certain extent to measuring sound in this frequency range. Consequently, entities such as professional associations cannot fully fulfill their tasks with regard to the risk assessment of exposed workplaces. We have solved this problem. Within the scope of transferring technology in the TransMeT program, PTB is currently developing a sound level measuring system that covers not only the range of audible sound, but also the ultrasonic frequency range. Our partner (and the direct user of the measuring system) is the Institut für Arbeitsschutz (IFA) of the Deutsche Gesetzliche Unfallversicherung (DGUV).

This development must take numerous requirements, such as those laid down by EN 61672, "Sound level meters", into account. The electromagnetic compatibility of the system must be guaranteed to ensure that the measurement is not biased by strong electromagnetic fields as are encountered at some industrial sites.



The prototype of the ultrasound level meter consists of a notebook, an analog-to-digital converter, a power module, a preamplifier and a 1/4-inch condenser microphone cartridge.

Moreover, the measuring range should be selected in such a way that the very high sound level pressures occurring locally at workplaces that are exposed to ultrasound can be detected. Due to the short wavelengths of ultrasound, even the smallest of obstacles can hinder its propagation. This must be taken into account when using microphone protection devices or fixtures to cause as little disturbance as possible to the sound field to be measured.

The first prototype of the measuring system has been developed. It can be transported in a case and can be powered by using rechargeable batteries. Tests are currently being carried out to find out whether the system can live up to the special requirements in practical application. A calibration procedure and a measurement method will then be provided.

In the short term, engineers working in occupational health and safety will be provided with a concrete tool to determine and assess the exposure due to airborne ultrasound at workplaces in accordance with the requirements of the relevant standards. In the long run, the findings obtained from this development can be used in standardization and for the development and future commercialization of improved systems. ■

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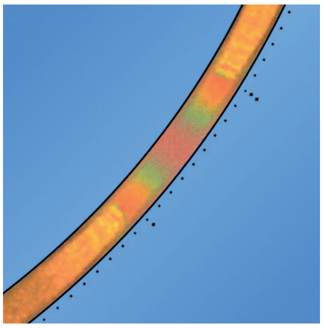
Standards and regulations
Verordnung zum Schutz der Beschäftigten vor Gefährdungen durch Lärm und Vibrationen (German Occupational Health and Safety Regulation on Noise and Vibrations – LärmVibrationsArbSchV)

EN 61672-1:2013: Electroacoustics – Sound level meters – Part 1: Specifications

Improved orientation of wafers

Especially interesting for
- manufacturers of microchips
- manufacturers of micromechanical components

For the exact processing of silicon wafers, it is necessary to identify the orientation of the crystal within the silicon wafer. An invention by PTB has improved the determination of the crystal orientation in <110>-wafers by additionally detecting incident light on special structures (see figure). These test structures are positioned on the surface and have enabled an automated determination process for the first time. This allows the crystal orientation to be determined with exceptional accuracy, so that the wafers can be oriented more precisely for the machine tools used. Moreover, this method can easily be integrated into existing facilities. (Technology Offer 0436) ■

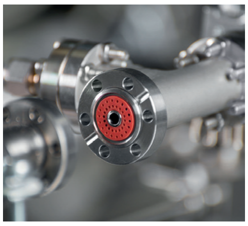


The figure shows rotationally symmetric grooves with a continuous color gradient on a wafer.

Advantages
- *allows the crystal orientation to be determined with excellent accuracy*
- *determination of the crystal orientation can be automated*
- *easy to integrate*

Mixing device for particulate matter

Especially interesting for
- the measurement of particulate matter and particles
- exhaust gas measuring techniques



The mixing device (red) surrounds a central flow channel and feeds a diluting gas into the channel to optimize the mixing process.

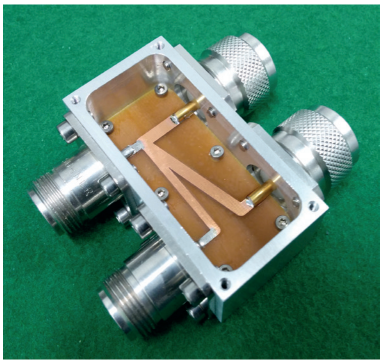
To date, two types of particulate matter or of fluid media (as encountered in exhaust gas measuring techniques) have been mixed by feeding one of the media laterally to the flowing direction of the other medium. These two media only mix homogeneously far way from the feeding point. A mixing module developed at PTB reduces the length of the section required for this process to take place, which can save costs in terms of material and space. Furthermore, this device enables a thorough mixing of two flows without the formation of interfaces. (Technology Offer 0472) ■

Advantages
- *mixing without interrupting the flow*
- *homogeneous mixing within a shorter section*
- *thorough mixing without formation of interfaces*

Calibration method for network analyzers

Especially interesting for
- manufacturers of network analyzers with more than two ports
- manufacturers of high-frequency measuring instruments

Network analyzers with more than two ports must be calibrated daily for use in electronics, communication engineering and high-frequency technology. This calibration can currently be very time consuming. A new calibration method from PTB mitigates this situation. Here, novel multi-port star circuit standards are characterized using a PTB reference calibration (see figure). The network analyzer is calibrated by means of such a calibrated standard applying the least-squares method. (Technology Offer 0456) ■



Example of a calibration standard of PTB in a star circuit

Advantages
- *little calibration effort*
- *low measurement uncertainty*
- *more robust in practice than con ventional procedures*

Contact person for questions about technology transfer
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Awards

Roman Schwartz

On 11 October 2017, PTB's Vice President was elected President of the International Committee of Legal Metrology (CIML) for a term of office of 6 years. He had previously already been CIML Vice President for a long time.

Uwe Arz

This staff member of Department 2.2, *High Frequency and Electromagnetic Fields*, has received the Best Oral Presentation Award of the 90th ARFTG Conference for his publication "Establishing Traceability for On-Wafer S-Parameter Measurements of Membrane Technology Devices up to 110 GHz".

Revolutionizing the System of Units

The International System of Units (SI) is being fundamentally revised. The foremost expert committee in the world of metrology, the International Committee for Weights and Measures (Comité international des poids et mesures, CIPM), issued its fundamental recommendation for this development at its recently concluded annual assembly. It is hoped that, at their general conference in November 2018, the international community of signatories to the Metre Convention will heed the experts' advice and provide the physical units with an especially firm foundation – one that consists of the set values of selected fundamental constants. The seven base units will thereby lose their prominent role. Instead, seven fundamental constants will be determined as defining reference entities from which all units can be derived.
The key date on which the new definitions are formally to enter into force is World Metrology Day (20 May), 2019. Nothing about our daily lives is expected to change. The kilogram will be just as heavy on 20 May as it was on 19 May. However, daily life in advanced technological fields will look somewhat different. There, the new, highly innovation-friendly definitions are sure to bring noticeable benefits.
You will find much more complementary information about this topic on our website at: PTB > Research & Development > Research on the new SI

An extension for the Willy Wien Laboratory

Topping-out ceremony for an extension to PTB's Willy Wien Laboratory:
The new building will provide laboratory and office space and will be directly connected to the neighboring electron-storage ring Metrology Light Source (MLS). With a gross floor area of approx. 1400 square meters, it will accommodate office, laboratory and seminar space as well as utility rooms. This extension has been implemented by the Federal Institute for Research on Building, Urban Affairs and Spatial Development (BBR); its constructional completion is planned for 2019.

New research building

First groundbreaking for a highly specialized research building: the new Walther Meißner building will provide laboratory space as well as measuring and clean rooms to measure temperature with the greatest possible accuracy and for research activities relating to all aspects of superconductor sensors. The new building was designed by architects from Rohdecan in Dresden who came out as the winners of the competition organized by the Federal Institute for Research on Building, Urban Affairs and Spatial Development (BBR). The new Walther Meißner building will give fresh impetus to two research fields, namely the development and manufacturing of SQUIDs based on supraconductor thin-film technology – a field in which PTB is the world leader – and the application of the best cryostat systems in thermometry, allowing PTB to calibrate thermometers for industry over a very wide temperature range with all related services coming from one provider.
Contact (for both the Willy Wien Laboratory and the new research building): Frank Melchert, +49 (0)30 3481-7446, frank.melchert@ptb.de

Consortium of European research light sources

Representatives from 16 European institutions operating synchrotron radiation sources and/or free-electron lasers met in Brussels for the LEAPS (League of European Accelerator-based Photon Sources) consortium on 13 November 2017. This concentrated load of scientific excellence is to help take up challenges in the fields of energy, transportation, health, food safety and environmental protection. As one among five partners from Germany, PTB operates a laboratory at the electron-storage ring BESSY II as well as its own storage ring, the Metrology Light Source (MLS). Synchrotron radiation from these facilities is used from the THz to the X-ray range to deal with fundamental and applied metrological tasks with a focus on radiometry, reflectometry, scatterometry and spectrometry.
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MetroSommer 2018

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 28 September 2018. You are welcome to submit your application for the voluntary internship taking place within the scope of MetroSommer 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. The remuneration amounts to € 500/month; days off may be granted if you need to take an exam. The application form and an overview of all projects offered within the scope of the internship are available at www.ptb.de/metrosommer (in German).
Application deadline: 30 April 2018
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Imprint
PTB News 1/2018, English edition,
Issue May 2017, 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)
Editorial Office: Press and Information Office, PTB, Bundesallee 100, D-38116 Braunschweig,
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Federal Ministry for Economic Affairs and Energy

The Physikalisch-Technische Bundesanstalt, Germany's national metrology institute, is a scientific and technical higher federal authority falling within the competence of the Federal Ministry for Economic Affairs and Energy.