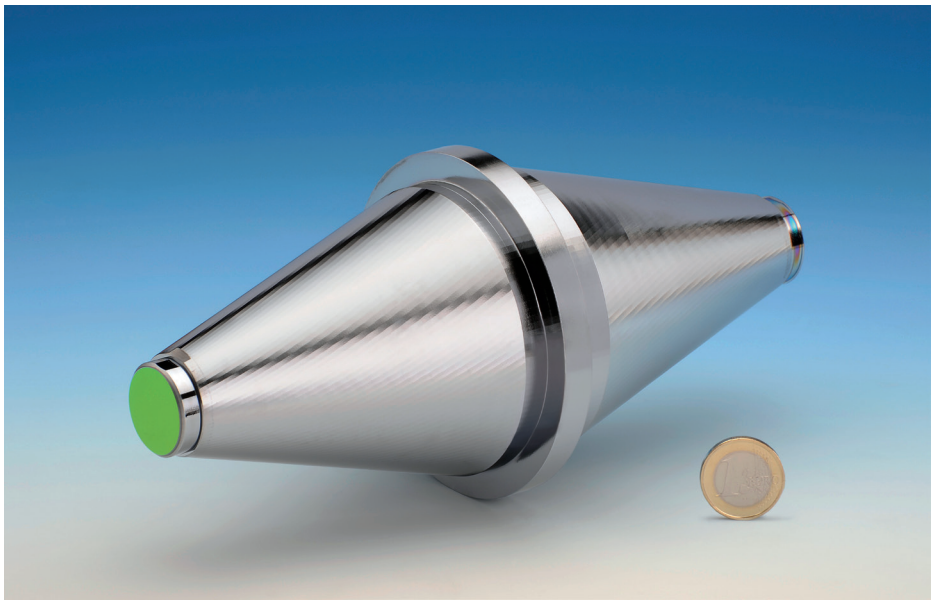
PTB NEWS
THE SCIENTIFIC NEWSLETTER OF PTB
3
2012

The PTB-News is the scientific newsletter of the Physikalisch-Technische Bundesanstalt (PTB). It is addressed to PTB's cooperative partners in economy, science and to all other interested readers.



The size of the new silicon resonator compared to the size of a coin

The most stable laser in the world

Silicon resonator with length stability unachieved so far – for improved optical atomic clocks

Especially interesting for
- developers of optical atomic clocks
- highest-resolution spectroscopy

A laser with a frequency stability so far unequalled: This is the result of a research cooperation of the Physikalisch-Technische Bundesanstalt (PTB) within the scope of the Excellence Cluster QUEST (Centre for Quantum Engineering and Space-Time Research) with colleagues from the American NIST (National Institute of Standards and Technology)/JILA Institue of NIST (National Institute of Standards and Technology) in Boulder/Colorado. Their development is important for optical spectroscopy with highest resolution, e. g. of ultra-cold atoms. But, above all, an even more stable interrogation laser is now available for use in optical atomic clocks.

Optical atomic clocks require laser sources that irradiate light with an extremely constant frequency. For this purpose, commercial laser systems have to be stabilized, by means of, e. g., optical resonators. They are composed of two highly reflecting mirrors which are kept at a fixed distance by means of a spacer. In analogy to an organ pipe, the resonator length determines the oscillation frequency. Consequently, a resonator with a high lengthstability is required for a stable laser, i.e. the distance between the mirrors must be kept as constant as possible.

Modern resonator-stabilized laser systems have meanwhile been technically developed to such an extent that their stability is only limited by the thermal noise of the resonators. Similar to the Brownian motion of molecules, the atoms in the resonator are constantly moving and are, thus, limiting its length stability. Up to

now, resonators have been made of glass, whose disordered and "soft" material structure, however, rather shows strong movements. For the new resonator, the research group has used single-crystal silicon, a particularly "stiff" and thus low-noise material. In addition, cooled down to a temperature of 124 K (-149 °C), silicon exhibits an extremely small thermal expansion; the low temperature contributes to additionally reducing the thermal noise. To operate the resonator at this temperature, the researchers had to develop, first of all, a suitable low-vibration cryostat. Comparison measurements with two glass resonators allowed the scientists to demonstrate a frequency stability so far unequalled of $1 \cdot 10^{-16}$ for the laser stabilized to the silicon resonator, which corresponds to twice the stability of the best laser in the world.

The "pendulum" of an optical clock, i. e. the swinging system of an optical clock, is a narrow optical absorption line in an atom or ion, whose transition frequency is read out by a laser. The linewidth of these transitions typically amounts to a few millihertz. This value could not be reached by glass resonators due to their limited length stability.

The laser to which the silicon resonator is stabilized reaches a linewidth of less than 40 mHz and can, thus, read out significantly narrower atomic lines. This contributes to improving the stability and accuracy of optical atomic clocks – the objective being an order of magnitude each. And also optical precision spectroscopy, another focal point of research of the Excellence Cluster QUEST, can receive decisive impetus.

For the future, there is still room to improve the optical mirrors whose thermal noise limits the achievable stability. Therefore, the researchers will in future go down to even lower temperatures close to absolute zero and use novel highly reflecting structures to improve the frequency stability by another order of magnitude. ■

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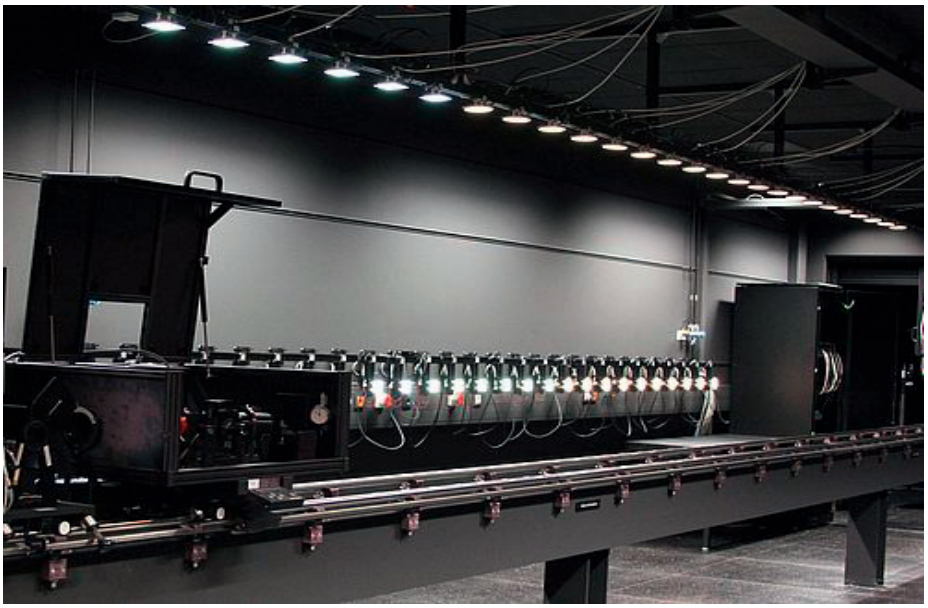
Scientific publication
Kessler, T.; Hagemann, C.; Grebing, C.; Legero, T.; Sterr, U.; Riehle, F.; Martin, M.J.; Chen, L.; Ye, J.: A sub-40-mHz-linewidth laser based on a silicon single-crystal optical cavity. Nature Photonics 6 (2012) 687–692

How long can OLEDs glow?

Measuring facility for the evaluation of the service life of organic light-emitting diodes

Especially interesting for
- manufacturers of OLEDs

Organic light-emitting diodes (OLEDs) are novel, large-scale light sources which are based on organic semi-conductors and have been commercially available for approx. 2 years. The particular properties of OLEDs require new measurement procedures to determine their service life. Within the scope of an extensive study, PTB has, in collaboration with all European OLED manufacturers, determined criteria that are relevant for the service life of OLEDs.



The new measuring facility allows numerous different measurands to be determined.

OLEDs consist of large-scale layers of organic materials – which are, however, only a few hundreds of nanometres thick – in which the light is generated. In the course of their operation, the organic layers wear out, and undesired effects may occur, such as, e. g., a change in the light colour. A clearly visible change in the light colour and decreasing brightness are both phenomena which are, by far, not desired by many consumers, so that these two criteria can be considered relevant for the estimation of the service life of an OLED.

Within the scope of a cooperation project, PTB has developed a measuring facility to assess the service life of OLEDs and used it to investigate 60 OLEDs manufactured by the companies Osram OS and Philips, as well as by the Fraunhofer Research Institution COMEDD. The measuring facility allows all optical, thermal and electrical measurands which are relevant for OLEDs to be monitored in-situ. Specifically, the mean and spatially resolved luminance, the spectral composition of the light, and, thus, its correlated colour temperature and colour rendering

for various viewing angles as well as the OLED supply voltage, operating current and temperature are recorded.

The service life of OLEDs amounts to several thousands of hours and is determined in practice by means of so-called "accelerated ageing procedures". Within the scope of the project, the new measuring facility was also used to validate accelerated ageing tests. Hereby, the project partners operated identical OLEDs, for example, at clearly elevated currents, so that their service life ended after only a few hundreds of hours of operation. A model for the determination of the service life under real-life conditions was developed and then validated.

Based on these results, a standardized measurement recommendation for OLEDs is now being elaborated in a Technical Committee at the International Commission on Illumination (CIE) under the leadership of PTB. Furthermore, a detailed description of the methods for the determination of the service life of OLEDs presented here will soon be published in the form of a book. ■

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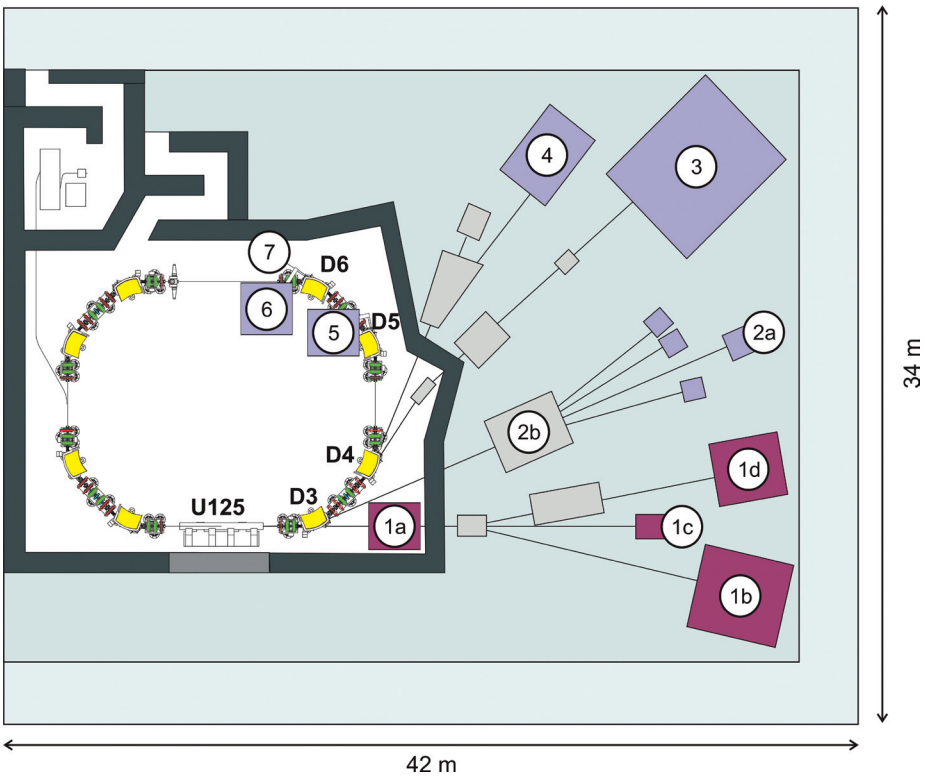
Scientific publication
Gerloff, T.; Sperling, A.; Lindner, D.; Meyer, M.; Pends, S.: In situ measurements of OLED lifetime. Proc. NEWRAD 86 (2011)

Cooperation with industry at the Metrology Light Source

Cooperation between Carl Zeiss and PTB on EUV lithography extended

Especially interesting for
- the semiconductor industry

PTB has extended its existing measurement capabilities at the Berlin synchrotron radiation facility BESSY II by an EUV beamline at PTB's own Metrology Light Source (MLS) in order to meet the growing need for high-end measurements for the at-wavelength characterization of lithography lens systems in the extreme UV (EUV, working wavelength: 13.5 nm). This measure has taken place in connection with new agreements in the field of EUV lithography (EUVL), in particular between Carl Zeiss SMT GmbH and PTB, with the objective of further intensifying the cooperation (which has been running since 1998) within the next four years. Carl Zeiss supplies the optical lens systems for ASML, the Dutch market leader when it comes to lithography machines used to manufacture computer chips and other microelectronic components.



The measuring stations at the Metrology Light Source (1 a-d: undulator radiation; 2 a-b: use of the MLS as a source standard; 3: EUV radiometry; 4: VUV radiometry; 5-6: IR and THz; 7: diagnostics for the storage ring)

The MLS is an electron storage ring located in the immediate vicinity of BESSY II, in Berlin-Adlershof; it belongs to PTB and has been in operation since 2008. The MLS supplies synchrotron radiation in the spectral range from 1 THz to the EUV range. In the EUV range, i. e. in the wavelength range around 13.5 nm, the measurement capabilities of the MLS and of BESSY II (where PTB also makes massive use of X-rays) overlap. The EUV beamline at the MLS (#3 in the figure) has been in operation since mid-2011 and – after a test phase – has, since the beginning of this year, been increasingly used for measurements within the scope of research cooperations, in particular for EUVL. In the near future, high-end computer chips will be manufactured using EUVL. For mid-2013, also PTB's large-scale EUV reflectometer is planned to be transferred from BESSY II to the MLS; an EUV scatterometer/ellipsometer – optimized for scattering measurements in

particular – is to take its place. With the new beamline, PTB has a total of approx. 6,000 hours of measurement time per year at its disposal for EUV metrology.

Also in other areas, PTB has clearly extended its measurement capabilities in the field of „metrology with synchrotron radiation“ over the course of the year by commissioning new beamlines at the MLS. Compared to its predecessor at BESSY II, a new measuring set-up for the calibration of radiation sources now also allows measurements to be carried out at wavelengths below 40 nm. Calibrated radiation sources in the vacuum-UV (VUV) and the EUV are, for example, of great importance for the characterization of space telescopes for solar and atmospheric research. A new undulator beamline provides monochromatized intensive and highly polarized radiation from the IR range up to the EUV range. At present, the first quantitative investigations of surfaces are being carried out by means of UV/VUV ellipsometry and electron spectroscopy together with partners from the research site Adlershof. Furthermore, a new near-field microscope has been put into operation at the infrared beamline of the MLS.

With the new beamlines and measuring stations at the MLS, PTB has, again, clearly extended the spectrum of measurement capabilities at its disposal for metrology with synchrotron radiation for EUV radiometry, source calibration, undulator radiation and IR near-field microscopy. ■

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Measuring with (razor-)thin layers

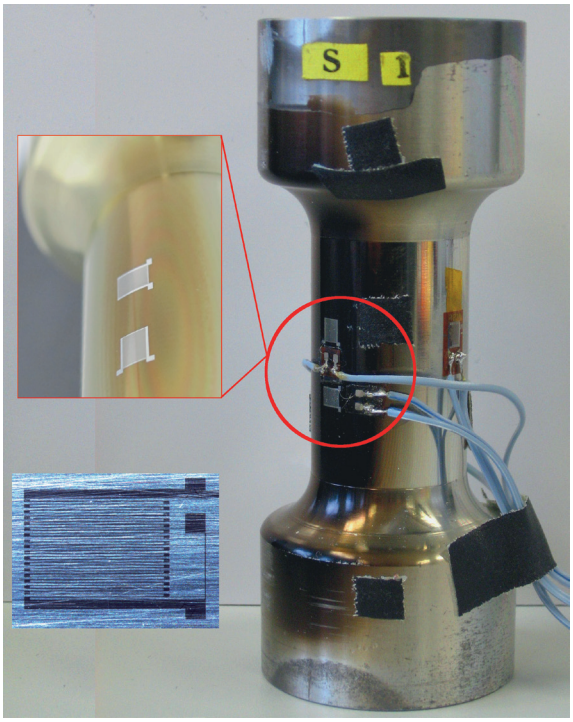
PTB develops thin-film sensors applied directly on uneven, rough workpiece surfaces

Especially interesting for
- metrology institutes
- sensor systems (mechanical and thermal measurands)

Thin-film sensors have a great potential in metrology. At PTB, such precise, micrometre-thick sensor layers can now be manufactured even on uneven and rough components.

Thin-film systems with thicknesses from a few 10 nanometres up to several micrometres have been successfully applied as sensors at PTB for several years. After using such layers successfully – e. g. as sensors for measuring the electrical resistance or conductance of aqueous solutions – PTB focused its developments on thin-film sensors which can be deposited on components with uneven, rough surfaces and on the most diverse materials.

These films are applied in vacuum by means of sputtering: atoms are ejected out of a suitable electrode material (target) by ions, and these atoms are deposited onto a substrate as a thin layer. Hereby, the layer has a very strong bond to the substrate, which, for sensors, is a great advantage compared to an adhesive film. Given the increasing need for high-precision sensors, further advantages are that measurements can be carried out with high resolution and, especially, directly at the working point and under the influence of harsh ambient conditions. Besides the development of 3D-compatible insulation layers, also the structuring procedures had to be adapted. After a few development steps, it is now possible to manufacture structures that are precise to the micrometre on such uneven, rough surfaces by exposing a photoresist to a UV laser and a 4-axis precision robot system. Hence, resistance strain gauges (e. g. for force sensor systems) were fixed directly onto a cylindrical test piece for force measurement of up to 10 kN. Compared to affixed strain gauges, the sputtered ones showed clear advantages with regard to sensitivity as well as with regard to the step function response of the sensor which emphasize the huge potential of this kind of bonding.



Direct comparison between sputtered (red) and (to the right of them) affixed strain gauges on a test piece for force measurements of up to 10 kN. The partial images on the left show close-up images of the sputtered strain-gauge structure in different image enlargements.

In future, this technique is to be further refined to be able to deposit a dense, flawless and well-bonded insulation layer onto various substrate materials and varying geometries. Hereby, particular attention will be paid to a perfect insulation layer between the electrically conducting substrate and the sensor layer.

This is the key to a universally applicable technology, since numerous applications are based on a metallic – and, thus, electrically conducting – uneven, rough body material. ■

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Scientific publication
Schmaljohann, F.; Hagedorn, D.; Buß, A.; Kumme, R.; Löffler, F.: Thin-film sensors with small structure size on flat and curved surfaces. Meas. Sci. Technol. 23 (2012) 074019

Improved diagnosis of rheumatism

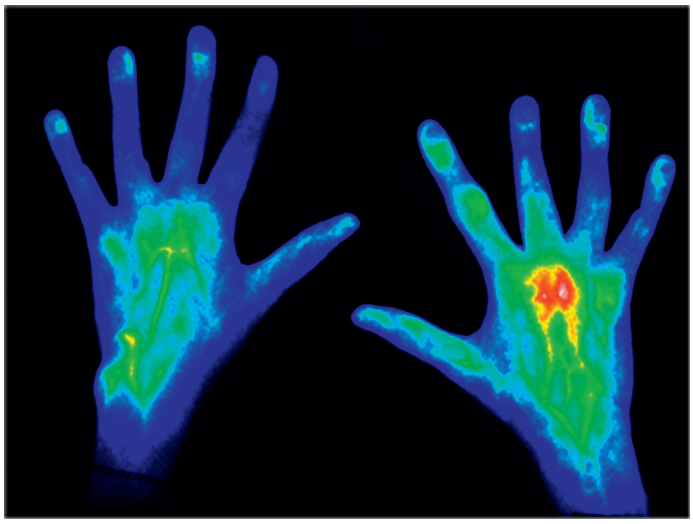
Patent granted for an optical procedure for the early diagnosis of rheumatism

Especially interesting for
- rheumatologists
- hospitals
- manufacturers of medical equipment

The European Patent Office has granted a patent for a novel diagnostic procedure which was developed at PTB, together with partners from the Charité Hospital in Berlin and industry. This diagnostic procedure could prove to be helpful in diagnosis and therapy control of rheumatoid arthritis.

Approximately 1 % of the population suffers from rheumatism. The earlier this disease is detected and treated, the more probable the chance of maintaining a good quality of life. The highly sensitive diagnostic procedure developed at PTB allows early stages of the disease to be detected. The patent describes the use of indocarbocyanine as a contrast agent in fluorescent imaging for the early detection of inflammatory diseases, in particular rheumatoid arthritis.

Fluorescent imaging is based on near-infrared light of the spectral range from 700 nm to 900 nm. The body's own dyes absorb less of near-infrared light, and it is less scattered by the tissue than visible light; similar to thermal radiation, it penetrates a few centimetres deep into the tissue. This makes the procedure interesting, in particular for the optical examination of finger joints. The near-infrared light excites the fluorescence of the contrast agent indocyanine green (ICG), which has previously been injected intravenously; this contrast agent is approved for invasive diagnostics. As increased vascularization occurs in inflamed joints, these fluoresce much stronger than healthy joints. The emitted fluorescence is detected by means of an extremely sensitive EMCCD (electron-multiplying charge-coupled device) camera in such a way that even small concentrations of the contrast agent can be made visible. For the assessment of the intensity distribution over the palm of the hand, PTB has developed software which, besides the visual assessment, also allows an objective quantitative comparison, similar to the assessment of an X-ray image. The measurement procedure was successfully tested within the scope of an exploratory study carried out on 60 patients and 30 test subjects.



Fluorescent image of the hands of a 51-year-old patient suffering from rheumatism, recorded with PTB's certified device two minutes after the injection of the contrast agent. False-colour representation where the colours red to white stand for the highest intensity. An inflammation is visible on the right hand, not on the left. (Fig.: Helios-Klinikum Berlin-Buch, Prof. Michael N. Berliner)

Within the scope of technology transfer, PTB has been working in close collaboration with the mivenion company as a partner for the exploitation of the procedure for some time. At an early stage, the company purchased the licence for the utilization of the now patented trade mark rights and commercialized a device (Xiralite®) based on this principle; several hospitals and practising physicians are already using this device for the early detection of rheumatoid arthritis. The trade mark rights which have now been granted are a significant milestone for the further exploitation of the new diagnostic procedure – and, thus, its establishment. PTB's European patent (Optical imaging of rheumatoid arthritis, EP No. 1 931 391 B1), which was granted on 21 December 2011, has now been transposed into the national phase of the European countries. ■

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Scientific publication
Dziekan, T.; Weissbach, C.; Voigt, J; Ebert, B.; Macdonald, R.; Bahner, M.L.; Mahler, M.; Schirner, M.; Berliner, M.; Berliner, B.; Osel, J.; Osel, I.: Detection of rheumatoid arthritis by evaluation of normalized variances of fluorescence time correlation functions. J. Biomed. Opt. 16 (2011) 076015

Music from the ear

PTB investigations have shown how an objective audiometric test can become even more reliable

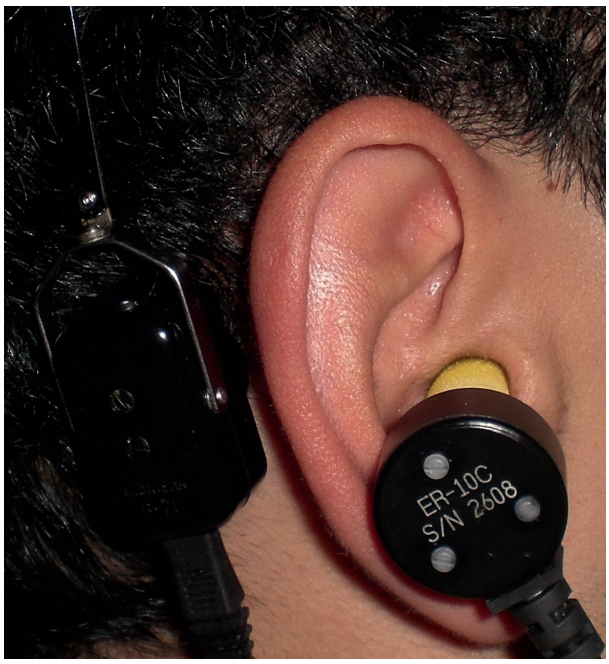
Especially interesting for
- otologists and hospitals
- manufacturers of medical devices

Not only can the human ear detect sounds, it can also generate them. If the ear hears the two upper tones of a major triad, it produces the fundamental of the chord which can then be measured. This phenomenon, called „otoacoustic emission“ (OAE), is used by otologists for objective audiometric tests, e. g. in newborns. PTB investigations have shown that an OAE audiometric test becomes even more reliable if the two sounds are transmitted to the ear not via a loudspeaker, but by bone conduction.

Regardless of where people come from, whether they are Europeans or Asians, the human ear is always tuned to a major scale. If the ear hears the two upper tones of a major triad, the ear itself produces the third, lowest, tone of the chord. This tone is called „distortion product otoacoustic emission (OAE)“ and is generated due to anatomic and physical laws: if the hair cells in the inner ear are healthy and sound, they are stimulated by the two matching tones to vibrate at a third frequency. This lower tone comes out of the ear again and can be measured by means of a highly sensitive microphone. With the aid of this phenomenon, it is possible to check objectively whether the hearing of newborns or infants is intact.

Such a test used to be performed using two tiny loudspeakers, each of which emitted a tone into the ear, as well as a miniaturized microphone, which recorded the third tone (if it came out at all). If this tone is not generated, physicians have a first inkling that the baby might need therapy or a hearing aid. However, it may be that the ear is healthy but does not „hum“. This can be caused by a badly calibrated loudspeaker, or due to the fact that the loudspeakers which are placed close to each other emit standing waves into the auditory canal which weaken one of the two tones.

To preclude such malfunctions, alternative tone generation methods have been investigated at PTB within the scope of a DFG project: so-called „bone vibrators“ which, in analogy to a tuning fork set onto the bone, convey the tone directly to the bone located behind the ear. The results have shown that both with two bone vibrators and in combination with a loudspeaker, correct otoacoustic emissions are generated. This not only allowed calibration errors to be reduced, but also provided physicians with improved differential diagnosis possibilities, since with the new procedure, they can test the function of the inner ear without a doubt and, potential damage of the middle ear, thus, has less influence. Clinical studies should follow. ■



Combined stimulation of otoacoustic emissions: the first tone is transmitted via air conduction (probe speaker in the ear), the second tone is conveyed via bone conduction (bone vibrator behind the ear).

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Scientific publication
Zebian, M.: Calibration of Distortion Product Otoacoustic Emission Probes. Dissertation, TU Braunschweig (2012)

Measuring instrument for ultrasonic cleaning efficiency

Especially interesting for
- users of ultrasonic cleaning devices
- medical engineering, doctors, and hospitals

Licence partner wanted

Ultrasonic cleaning baths are used for many applications – both for industrial and for domestic use. PTB has developed a new measurement procedure which allows the cleaning efficiency of such ultrasonic baths to be determined objectively for the first time. Hereby, a defined quantity of impurity is applied onto an oscillating quartz crystal; the quantity abraded is determined using a new measurement principle. This simple procedure, which has been developed at PTB and can be standardized, can be used directly in operating ultrasonic baths and serves the purposes of quality assurance, e. g., in medical engineering. ■

Measuring instrument for the cleaning efficiency of ultrasonic baths in operation

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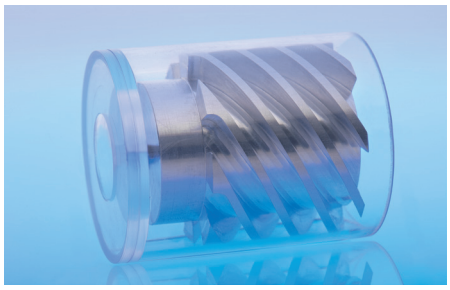
Measuring facility for small gas flowrates

Especially interesting for
- process technology (e. g. bioreactors)
- testing laboratories

Licence partner wanted

It is hardly possible to measure small gas flowrates using mechanical flow meters if, simultaneously, a low measurement uncertainty is to be attained. The novel drum design of a miniaturized drum-type gas meter of PTB allows gas flows of down to 1 ml/min to be determined simply and accurately, independent of the type of gas used. The novel design of the drum allows more accuracy than traditional designs and is not expensive. The novel drum-type gas meter can also be used where small gas quantities have to be accumulated over longer periods of time (creep flow volumes). ■



Model of the drum-type gas meter

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Dynamic measurements now fit for calibration

Especially interesting for
- component and materials testing
- the automotive industry (e. g. crash tests, engine performance tests)

Licence partner wanted

The dynamic bridge standard simulates the dynamic behaviour of DMS bridges and is used for the calibration of measuring amplifiers.

To measure dynamic mechanical quantities such as force or pressure, (mostly strain-gauge-based) sensors are used. The signal provided by these sensors is amplified, filtered, linearized, and normalized by measuring amplifiers, in short: processed. Not only the sensors, but also the corresponding measuring amplifiers have to be calibrated with regard to their amplification factors, linearity, and phase. For dynamic (temporally variable) operation, PTB has developed a bridge standard which allows these measuring amplifiers to be calibrated for practically any temporally variable voltage curve up into the kHz range. ■

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Awards

Philipp Hönicke
This staff member from the Department 7.2 Cryophysics and Spectrometry received the 2011 Norman Hackerman Young Author Award of the Electrochemical Society (ECS) at the ECS Meeting in Honolulu (Hawaii) on 8 October 2012 for his contribution to the work "Towards passivation of Ge(100) surfaces by sulfur adsorption from a $(NH_4)_2S$ solution: A combined NEXAFS, STM and LEED study".

Manfred Fuchs and Hermann Scholl
Prof. Dott. Ing. h. c. Manfred Fuchs (photo: left), head of the German aerospace company OHB AG, and Prof. Dr.-Ing. Hermann Scholl (photo: right), who has led the company Robert Bosch GmbH for many years, received the Werner von Siemens Ring on 13 December 2012, one of the most important German prizes in the field of technology and engineering sciences. Prof. Dr. Joachim Ullrich (photo: centre), PTB President and Chairman of the Foundation Council of the Werner von Siemens Ring Foundation, awarded the prize at the Representative Office of the Robert Bosch Stiftung in Berlin.

Dates

19.–20. March 2013: Berechnung der Messunsicherheit – Empfehlungen für die Praxis (Calculation of Measurement Uncertainty – Recommendations for Practical Applications)
PTB Seminar. PTB Berlin Institute.
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25.–26. April 2013: NanoScale 2013
10th Seminar on Quantitative Microscopy (QM) and 6th Seminar on Nanoscale Calibration Standards and Methods. Paris, France.
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29. May–2. June 2013: Annual Conference of the German Branch of the European Optical Society
PTB Braunschweig.
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5.–6. June 2013 TEMPERATUR 2013
Procedures and instruments in the measurement of temperature and humidity. Hermann von Helmholtz Building, Lecture Hall, PTB Berlin Institute.
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Steffen Rudtsch
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PTB at trade shows and conferences

12.–14.3.2013: DPG Spring Conference
PTB presents itself as an employer at the largest of the DPG's spring conferences, Solid-state Physics Section. Regensburg.
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15.–16. April: Zählen – Messen – Prüfen (Metering – Measuring – Testing)
Trade convention for intelligent and innovative measuring systems and processes. Messe Leipzig (Leipzig Trade Fair).
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13.–16.05. 2013: LASER World of PHOTONICS
PTB presents its latest developments in the field of optics, laser and measurement technologies in the scope of the Kompetenzzentrum für Ultrapräzise Oberflächenbearbeitung e.V. (Competence Centre "Ultraprecise Surface Figuring" – CC UPOB). Messe München (Munich Trade Fair), Hall B2.
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14.–17. May 2013: Control
PTB shows the latest developments from the field of geometric and precision engineering at the leading international trade fair for quality assurance and metrology. Messe Stuttgart (Stuttgart Exhibition Centre), Hall 1.
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Publications

PTB-Mitteilungen Vol. 3/2012
Focus: Metrology Through the Ages

PTB-Mitteilungen Vol. 4/2012
Focus: Metrology for the Future

For further information:
www.ptb.de > English Version > Publications

Imprint
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