PTB news

Metrology
Technology
Services
Cooperation

09.1

Towards 3D Nanometrology

At PTB, a purely metrological scanning probe microscope has been developed into a micro-/nano coordinate measuring machine. This now allows dimensional quantities with nanometer resolution also to be measured on three-dimensional objects in an extraordinarily large measurement range of 25 mm x 25 mm x 5 mm.

Today, components with structures in the micro- and nanometer range are integrated in many products of daily life – from the micromechanical motion sensor to the computer chip. Compliance with the geometric tolerances of these small structures is in many cases decisive for smooth functioning of the overall system. Dimensional metrology on such structures is, therefore, of increasing importance. To meet the increasing requirements for 3D measurements of micro- and nanostructures, 3D measuring probes newly developed at PTB were incorporated in a metrological scanning probe microscope based on a commercial nano-positioning system with integrated laser displacement sensors. On that basis, the device control was extended by a software interface (I++ DME) independent of manufacturer and a standard measurement and evaluation software (Quindos 7.0) for coordinate measuring machines (CMM).

The new functionalities given by measuring probe and software extend the scanning probe microscope to a metrological micro/nano coordinate measuring machine (CMM) which also allows 3D measurements conforming to standards to be performed on micro- and nanostructures.

International intercomparisons on step-height standards and lattice structures have shown that the measuring system is one of the most precise of its kind worldwide. For step heights, measurement uncertainties in the subnanometer range have been reached and for measurements of the mean structure spacing on extensive lattice standards, uncertainties even in the range of 10 picometers have been reached and confirmed in comparison with optical diffraction measurements.

The new measuring instrument is available for dimensional precision measurements with nm resolution on 3D micro- and nanostructures such as micro gears, micro balls, hardness indenters and nano lattice standards as well as for comparisons of measures; moreover, it serves as a platform for research and development tasks. It is an important link between nano-, micro- and macro coordinate metrology.

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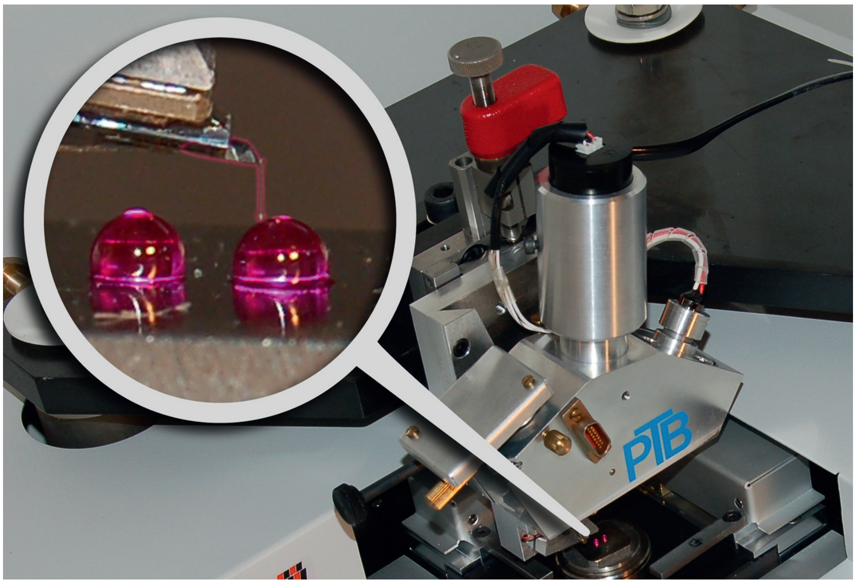

The micro probe used in the micro-nano CMM measures the form and the spacing of two reference spheres with diameters of two millimetres each. The figure shows a survey of the system and the proportions of measuring probe and measurement object in detail.

Reliable car sensors

Ob im Getriebe, inWhether in the gear, in the air-conditioning system or for the control of airbags – modern cars are no longer imaginable without sensors. The most important thing for the reliability of car sensors is their encapsulation. Although designed to protect the sensor against environmental impacts, it can for its part generate a considerable mechanical load which may cause a failure of the sensor functions. With a specially developed X-ray diffractometer, scientists of the Physikalisch-Technische Bundesanstalt (PTB) have now succeeded in performing a measurement of the mechanical stress condition of encapsulated silicon sensors. With immediate effect, this test device is now also available to customers from industry.

Today, more than 40 sensors, which control the safety systems, comfort functions and the energy budget, are already integrated into compact class vehicles. For protection against environmental impacts, the sensitive sensor element is closely wrapped with a die-cast housing of plastics. This encapsulation may, however, damage the sensor, as the silicon of the sensor and the plastic material of the encapsulation expand differently at different temperatures. The mechanical stresses caused by this in the sensor may lead to a premature failure of the electronic system. For a reliable and thus safe

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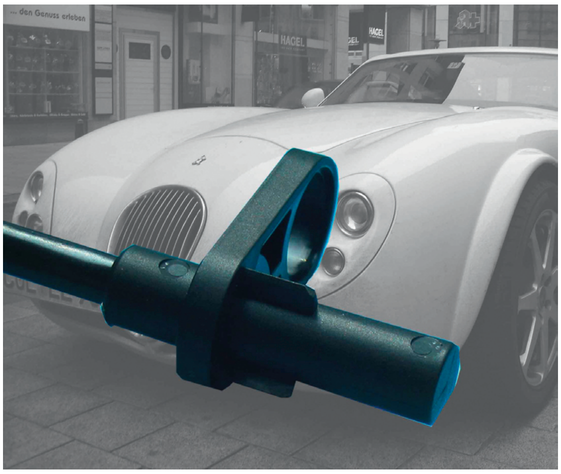
Reliable car sensors *(continued from page 1)*

automobile electronics, the encapsulation process must be improved with the aid of exact stress measurements.

For this purpose, an X-ray diffractometer, which can measure the stresses in a silicon sensor in a non-destructive way, has been developed at PTB. With the aid of high-energy X-radiation of more than 15 keV, the die cast packing of the sensors can be penetrated. In this way, stress values of up to – 200 MPa were determined on the encapsulated chip which indicates an enormous pressure load by the encapsulation. A special metrological challenge was the amplification of the weak measurement signal. It was met by combining a molybdenum fine focus source and an especially adapted glass capillary optics.

A parallel development of the company Bosch, in the case of which a force measuring chip passes through the encapsulation process instead of a car sensor and determines the mechanical stresses inside the encapsulation via an electric measurement, could be validated with the aid of PTB's X-ray diffractometer.



Wheel speed sensor in the die cast housing, application: ABS, ASR, ESP

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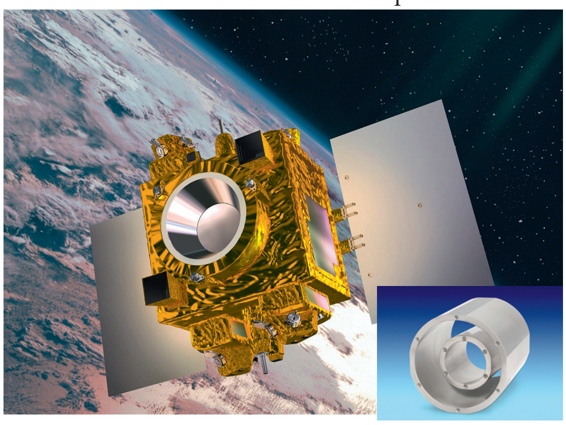
Equivalence principle in space test

Since Galileo Galilei and Newton, the assumption is valid that inert and heavy are equivalent. This is, however, questioned by new physical theories such as the String theory. Now, the equivalence principle is put to test with so far unachieved accuracy within the scope of the "Microscope" space project – a German-French cooperation. PTB has developed the manufacturing and measuring methods for the test masses which are required for the acceleration experiments in a near-Earth orbit, and has manufactured the first test pieces.

Space is the ideal place to check the equivalence of inert and heavy mass with an accuracy impossible under terrestrial conditions. This is why in 2013, the French Space Agency CNES (Centre National d'Etudes Spatiales) will bring a micro satellite in a near-Earth orbit and perform acceleration tests on different test masses. Main items of these tests are pairs of concentrically nested metal cylinders which flow in the satellite in equilibrium between the gravitational force of the Earth (which acts on the heavy mass of the cylinder) and the centrifugal force (which acts on the inert mass). If the satellite is, however, selectively accelerated, the equilibrium of forces is annulled.

The validity of these acceleration experiments decisively depends on the quality of the test masses used. Only if mass, form, density and thermal expansion of the cylinders are known with great accuracy, can the possibly very small differences between inert and heavy mass be observed anyway. PTB's Scientific Instrumentation Department has now succeeded in optimizing the manufacturing process for the test masses (made of a standard titan alloy and a very special platinum rhodium alloy) in such a way that the deviations in form and dimensions lie in all three space dimensions of the metal cylinders in the range of 1 µm. This precision represents an enormous technical challenge in which the theoretical production limits of the usable manufacturing machines were almost reached. This is why a comprehensive measuring technique had to be integrated into the processing station.

The prototypes manufactured so far were checked by the respective technical laboratories of PTB. They meet the accuracy aimed at and will be used in the Centre of Applied Space Technology and Microgravitation (ZARM) in Bremen – a cooperation partner in the project – for measurements in the drop tower which are performed before the orbital experiment is carried out. After evaluation of these measurements, PTB will manufacture the actual test masses for the satellite experiments.



The "Microscope" space project will check the equivalence principle of inert and heavy mass with so far unequalled accuracy. The test masses for the acceleration experiments have (in the case of the external cylinders) a length of 80 mm and an inner diameter of 60 mm. The surface roughness is smaller than 0.2 µm. (Photos: CNES/PTB)

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Fine vacuum - now measurable with larger accuracy

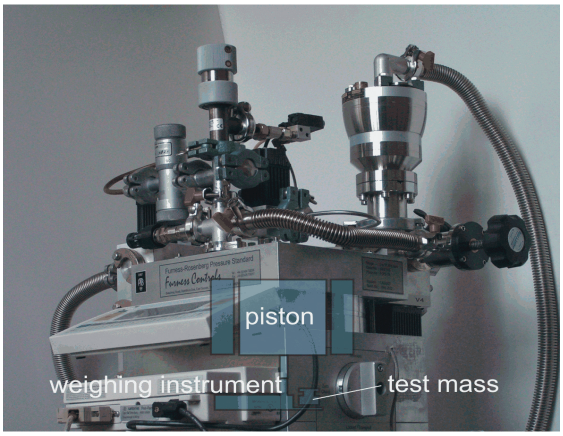
A new pressure balance now allows the uncertainty of pressure measurements in the range between 30 Pa and 1000 Pa to be improved by approx. one order of magnitude. Calibrations in this technologically important pressure range are demanded with particular frequency. Manufacturers of pressure gauges are thus informed with even more accuracy about the quality of their devices.

Industrially used pressure gauges control the gas pressure in production processes very exactly. The fine vacuum between 30 Pa and 1000 Pa is, for example, important in metallurgy when disturbing gases are eliminated from the liquid steel or in the chemical vapour separation of magnetic layers in the manufacture of hard disks.

For the calibration of industrial pressure gauges, PTB has evaluated a new pressure balance. It determines the pressure by measuring the force which a gas exerts on a piston. The "back pressure" can be neglected because the weighing instrument is in the vacuum. For exact measurements with a pressure balance, the effective cross-section of the piston must be known with great accuracy: It is determined with the aid of PTB's mercury manometer which measures the force on the piston at a known pressure. To rule out a significant influence of flow effects, the measurements were performed with different gases and under different pressure conditions.

In the pressure range between 30 Pa and 1000 Pa, the evaluation of the pressure balance furnished considerably smaller uncertainties of the realization as had been possible so far with the static expansion standard used up to now. At 1000 Pa, the relative expanded uncertainty now amounts to 0.0064 % instead of formerly 0.15 %.

At PTB, the pressure balance is already used parallel to the established national standard. Compared to the procedure so far applied, it turned out, however, that for recalibrations of high-quality capacitive membrane vacuum meters – which are used in industry – the long-term scatters were considerably reduced when the measurements were performed with the pressure gauge. Use of these devices will in future allow the manufacturers of vacuum meters to obtain considerably more exact information about the long-term stability and reproducibility of their devices.



The "heart" of the new pressure balance FRS5 is an Invar piston, centrically suspended by a parallelogram system, which presses on a weighing instrument. A test mass allows in-situ calibration of the balance. On the upper left: Standard deviation s in the case of recalibrations of four vacuum meters (CDG) over three years with the primary standard (SE2) so far used and the FRS5.

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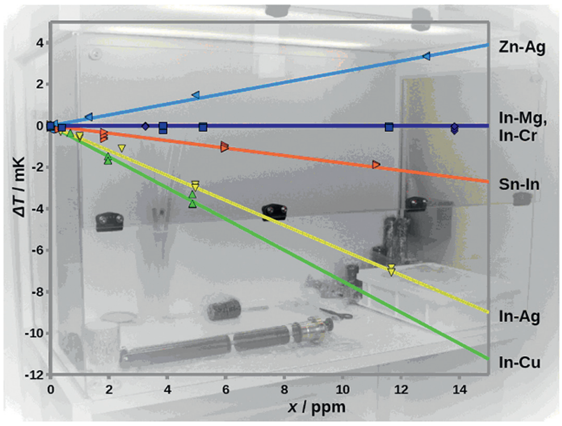
More reliable temperature fixed points

Almost all temperature measurements are finally based – via many intermediate steps – on the International Temperature Scale. In the temperature range between 20 °C and 1000 °C, which is important to industry, the fixed points of this scale are defined by the melting and freezing points of high-purity metals. Due to the contamination of the metals, uncertainties between 0.2 thousandths Kelvin and 2 thousandths Kelvin have so far been assumed. At PTB, the influence factors which have so far been theoretically assumed have now been replaced by concretely measured values so that the uncertainty can be reduced by factor 3.

The calculative correction developed by PTB is based on a chemical analysis. As no reliable data about the behaviour of solved substances are known in the range of extremely small contamination concentrations, these data must all be obtained by experiment. The applied method determines the amount of contaminations with uncertainties of a few ppb (this corresponds to 10^{-9} mol/mol) and determines its influence on the phase transition temperature of the metal.



Influence of different contaminations on the fixed point temperature of some high-purity metals. Whereas contaminations from magnesium or chromium, for example, do not affect indium fixed point cells, this does not apply to contaminations from silver or copper.

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More reliable temperature fixed points *(continued from page 3)*

To avoid use of the large fixed point cells the employed so far, special minimized cells have – on the basis of investigations of the heat transport in a fixed point cell – been developed which are selectively contaminated to observe the change of the melting temperature. The data obtained in this way on the fixed points of indium, tin and zinc are – as far as the uncertainty and amount at low costs are concerned – unequalled in the international comparison. The result of the investigations was surprising: For the first time it could be demonstrated that not all contaminations affect the fixed point as expected. A detailed analysis as well as calculations showed that elementary contaminations sometimes form insoluble compounds which do not have to be taken into account for the correction.

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Central certification body established

PTB's service offer in the field of certification of products and quality systems has now been bundled to form one single certification body. Previously, the responsibility for these activities had been distributed to three separate certification bodies.

The PTB performs conformity assessments within the scope of different certification systems and each year issues more than 2000 certificates for that purpose. With the fusion of the former three certification bodies (for non-automatic weighing instruments, explosion protection and measuring instruments) to one single body it pursues two objectives: uniformity and more effective quality assurance by consistent application of the four-eyes principle during conformity assessment. The latter is in compliance with the increased requirements of standard series DIN EN ISO 17000 which specifies fundamental requirements for modern certification bodies.

PTB has thus completed the step from an approving authority to a modern conformity assessment service provider. The range of activities of the certification body covers conformity assessments in the sector subject to legal control and in the voluntary sector – and that at the national and international level.

A survey of the current service offer can be found under: www.ptb.de/de/org/zs

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New 7 Tesla nuclear spin tomograph

A new nuclear spin tomograph (magnetic resonance tomograph, MRT) of the modern 7 Tesla generation has been put into operation in the Experimental and Clinical Research Center of the Max Delbrück Centre for Molecular Medicine in Berlin-Buch. It is the only 7 Tesla device wordwide in which a metrology institute is involved. As project partner, PTB is responsible for exploring the technical possibilities of the new device and for making it usable for clinical applications.

Inauguration of the new 7 Tesla MRT (from the left: Federal Research Minister Mrs. Annette Schavan, Dr. Bernd Ittermann, PTB, Prof. Dr. Walter Rosenthal, Scientific director of MDC).

In hospitals, MRT devices with magnetic fields of the strength 1.5 Tesla or 3 Tesla are usually used. The 7-tesla tomograph will at first be purely a research instrument in order to explore the possibilities of the ultra-high-field magnetic resonance tomography. Whereas most of the approx. 30 MRTs with such a high magnetic field which are at present available in the world are predominantly used for brain research, the new device in Berlin-Buch will be used for multiple purposes. As one of four in the world, the Berlin system is equipped with an 8-channel transmission system. By this, heart imaging by means of 7 Tesla MRT is, for the first time, the focus of attention.

New ground could also be broken in the case of the realization of molecular processes in the body, for example in the fight against tumours. Project partners are – in addition to the Max Delbrück Centre and PTB – the Berlin Charité, the Leibniz Institute for Molecular Pharmacology and the Siemens company.

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