PTB news
Metrology
Technology
Services
Cooperation
03.1

Avogadro constant re-measured

Measurements on silicon composed of its natural isotopes have been completed as part of PTB's "Avogadro-Project". The project itself is to contribute to the redefinition of the unit of mass, the kilogram. In a follow-up project with enriched ^{28}Si, the measurement uncertainty is to be further reduced.

The kilogram is the last quantity of the International System of Units, SI, still defined by a macroscopic body – the prototype kilogram. All other units are traced back to atomic processes, molecular properties or fundamental constants. By counting the atoms in a silicon crystal (mass: 1 kg) with highest precision, the definition of the unit of mass could be linked to the fundamental Avogadro constant.

The investigations were carried out on a high purity silicon single crystal produced by Wacker-Siltronic. The molar mass of the crystal was determined by mass spectrometry at the Institute for Reference Materials and Measurements of the European Union (IRMM) in Geel (relative measurement uncertainty: 4 µg/mol). At PTB, the following measurements were carried out: the lattice parameter of the material was linked to PTB's crystal length standard (measurement uncertainty: 0,012 fm); the mass of the 1 kg silicon sphere – manufactured at the Australian metrology institute – was compared with the national mass standard (measurement uncertainty: 19 µg); the diameter of the sphere (92 mm) and thus the sphere volume were determined through several individual measurements (measurement uncertainty: 2,7 nm) with a spherical Fizeau interferometer especially developed for this application; and the thickness of the oxide layer was determined by ellipsometry (measurement uncertainty: 0,36 nm). Special attention was paid to determine the type and number of defects in the crystal material: several external research institutions supported the PTB investigations.

The measurement results have been filed in the Avogadro Database of the BIPM. Based on the determined values of density, molar mass, impurities and lattice parameter, the following value is obtained for the Avogadro constant:
$N_A = 6{,}022\,135\,4\,(16) \cdot 10^{23}\ \text{mol}^{-1}$.

This value is in agreement with all previously published results obtained by this method. However, the new value is nearly $1 \cdot 10^{-6}$ lower than the 1998 CODATA value based on the results of the watt balance experiment performed at the National Institute of Standards and Technology (NIST). This discrepancy still must be clarified before the unit of kilogram is finally defined.

A serious problem for the measurements on silicon of natural isotopic composition is the relative inaccuracy in the value for the molar mass. This is reflected in the overall measurement uncertainty of $2{,}7 \cdot 10^{-7}$. In an international project with enriched ^{28}Si, the measurement uncertainty is to be reduced by a factor 10.

Stamp of the Italian Post, issued in 1956 on the 100th anniversary of the death of Amedeo Avogadro. The text on the stamp is a definition of the base unit mole for a gas.

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Semiconductor islands: From the artificial atom to the molecule

Semiconductor structures with two stacked islands of a few nanometres in size have been produced for the first time. The island structures were grown by a self-organizing process and the electrical transport properties were characterized. These structures are so tiny that they display atom-like (quantum) properties. Their realization represents a step forward in the direction of a new quantum standard for the unit ampere.

In the foreseeable future, progress in modern electronics which involves ever smaller circuits must overcome one boundary: as the structures are reduced to only a few nanometres in size, the laws of classical physics become invalid and quantum mechanics must be applied. Such tiny circuit components are also of great importance for precision metrology. In these components, single electrons can be manipulated to a greater extent than before. This is a prerequisite for a new standard for the unit of current, the ampere, based on counting electrons.

Growing semiconductor crystals utilizes a self-organizing effect which produces tiny "islands" of

(Continued on Page 2)

Semiconductor islands: From the artificial atom to the molecule

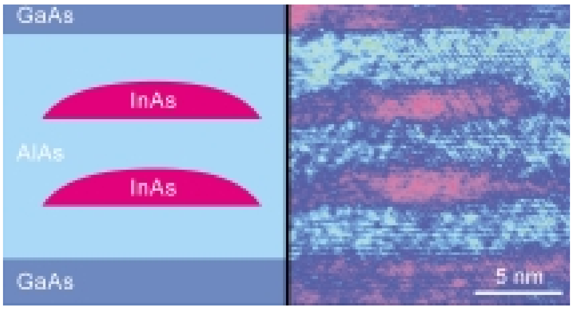
(Continued from Page 1)

a few nanometres in size. By exploiting this effect skilfully one can even stack the islands vertically. In experiments they then show new interesting properties. For this purpose, the self-organized semiconductor islands are embedded in the barrier of a resonant tunnel diode. Resonant tunnel diodes with two stacked nano-islands of indium-arsenide have been created for the first time. Their properties were observed during electrical transport experiments.

In the structures, the two islands in a way represent the emitter and the collector of a transistor. Sequential quantum-mechanical tunnelling processes determine the dependence of an electrical current flow as a function of the applied voltage. Just like atoms the quantum islands have typical discrete energy states for 0-dimensional systems and sharp current peaks are observed instead of steps as the voltage is changed. The sharp peaks hardly show any broadening with an increase in temperature, as they do not depend on the distribution of the most energetic electrons in the highly doped three-dimensional regions.

A new degree of freedom occurs by coupling the two nano-islands. By decreasing the thickness of the separating interlayer the two nano-islands become increasingly coupled and in the extreme case the electron wave functions of the two islands superimpose. Virtually speaking, a nano-island molecule is created. This was demonstrated by the influence of a modified spin splitting of the electrons in a magnetic field in cooperation with the "Nanostructures" Division of the University of Hannover.

The investigations also open up prospects for structures with more than two vertically coupled quantum dots. A practical advantage, viz. the reduction of an undesired background current through the barrier which is now thicker, could already be achieved.



Schematic section of two stacked semiconductor quantum dots (left) and transmission electron micrograph of a resonant tunnel diode (right).

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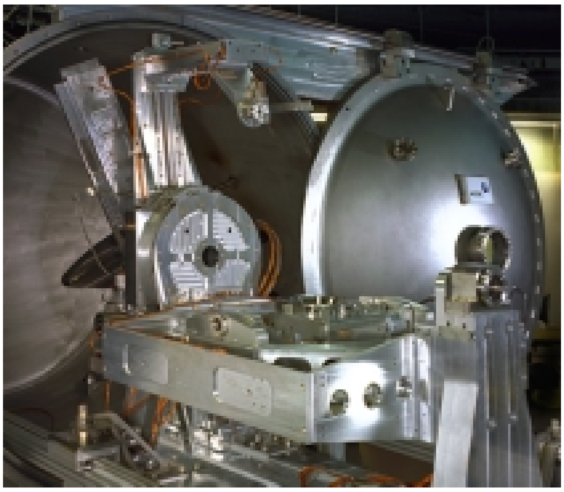
Large reflectometer for extreme ultraviolet lithography

PTB now operates a reflectometer to characterize mirrors of up to 50 cm for extreme ultraviolet lithography (EUVL) systems. The reflectometer is the only one of its kind in the world today.

Photolithographic generation of ever smaller structures for semiconductor processing requires the use of radiation with ever shorter wavelengths. By the year 2007 the semiconductor industry plans to use lithography systems with EUV radiation at a wavelength of 13,5 nm to manufacture components with structural widths below 50 nm.

Within the last decade PTB has become the European centre for EUV radiometry with its synchrotron radiation laboratory at the Berlin electron storage ring BESSY II. Today, PTB supports industry in the development of components and measurement procedures required for EUVL. At its beam line for soft X-rays, PTB performs "at-wavelength" measurements with 13 nm radiation necessary to characterize mirrors, mask blanks and masks for the European EUVL program. The PTB measurements are of oustanding accuracy. For example, a relative uncertainty of 0,14 percent was achieved in measurements of the reflectivity of Mo/Si multilayer mirrors, as has been confirmed recently through comparison measurements.

Carl Zeiss SMT AG, a close cooperation partner of the PTB, is presently preparing the production of EUVL mirrors as components of an industrial "α-Tool". To characterize these mirrors which can have dimensions of up to 50 cm and can weigh up to 50 kg PTB took a new reflectometer into operation in November 2002. The set-up was done in cooperation with Carl Zeiss and supported by the German Federal Ministry for Economy and Labour (BMWA). The unique measuring station will serve to characterize EUVL mirrors, mask blanks and masks with respect to their reflectivity and the homogeneity of the multilayer thickness – the latter with a relative target uncertainty of $1 \cdot 10^{-4}$.



The new EUV reflectometer. In the background, the open door of the vacuum tank 2 m in diameter can be seen, and in the foreground – on a mounting frame – the mechanical system for mirror positioning. The device allows also large and heavy mirrors to be aligned with accuracies of 10 µm and 0,01°.

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Precise generation of ultra-low currents

A new PTB device generates lowest currents with high precision and over long periods of time. The device allows to calibrate measuring instruments in the picoampere region with significantly improved accuracy.

Measuring lowest currents is becoming increasingly important in many fields such as, for example, semiconductor or medical instrumentation industries. In medical radiation diagnostics and therapy, for example, dosimeters based on the ionization of an irradiated gas are typically used to determine radiation levels. These devices measure very low currents in the pA region. The accuracy of the measurement is decisive for a precise dose determination. Testing and calibration of such dosimeters and other picoamperemeters require electrical current source standards that can produce constant low currents with high accuracy over long periods of time. An important method to generate extremely low currents is based on charging and discharging a capacitor. Applying a voltage that increases or decreases linearly in time (dU/dt = constant) to a capacitor of capacitance C, generates a constant current $I = C \cdot dU/dt$. By measuring the capacitance and the time derivative of the voltage the generated current to be traced back to the units of capacitance, voltage and time.

A new current source of this type for highest specificities has been developed at PTB. In order to calibrate measuring instruments that average over long periods of time as well, a voltage ramp generator was developed based on an electronic integrator. Continuous and variable measuring times of up to 35 minutes can be realized. The relative fluctuation of the voltage ramp remains below $1 \cdot 10^{-5}$.

The new device produces user-defined currents of 10 pA to 0,1 fA. At 1 pA, the uncertainty amounts to only 0,01 percent. At 10 fA it is still 0,05 percent.

This calibration technique is currently being transferred to a renowned German manufacturer of dosimeters. The method can gain even greater economic importance for the semiconductor industry. Here, the capability to measure lowest currents in the fA region is one of the typical requirements for modern picoamperemeters, as spurious leakage currents in the cells of modern memory devices are of the same order of magnitude. The new procedure has made the calibration of such devices more reliable and accurate.

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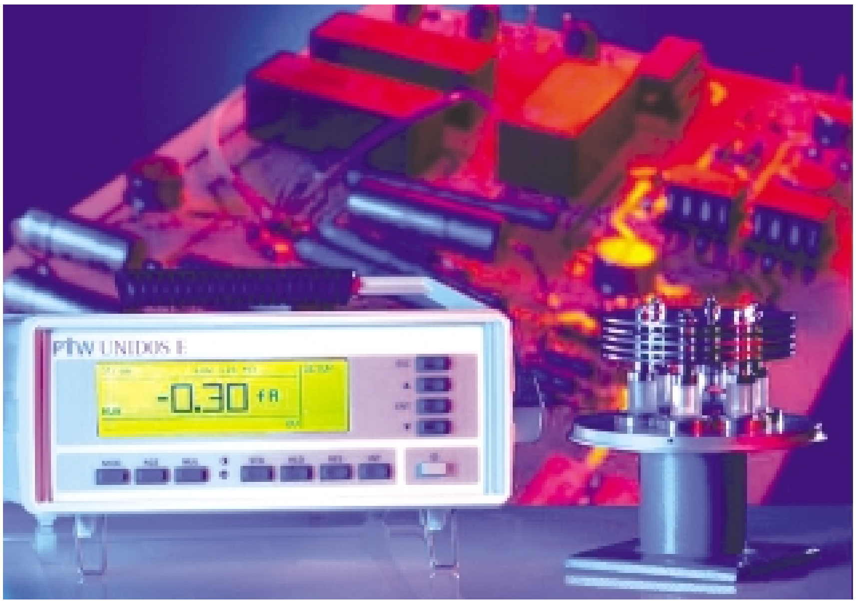

Measuring instruments for extremely low currents can be calibrated with the aid of an air capacitor and an electronic ramp generator.

New measurement station for wafer quality control

In the PTB laboratory at the Berlin synchrotron radiation facility BESSY II, a new measuring station has been put into operation. The set-up can determine smallest impurities on silicon wafers by means of X-ray fluorescence analysis. The station can analyse the composition of nanometre-thick layers as well.

Energy-dispersive X-ray fluorescence analysis (XRF) is a physical method for non-destructive elemental analysis. Several elements can be detected simultaneously. For investigations, the sample is irradiated with X-rays and the resulting element-specific fluorescence radiation is recorded with an energy-dispersive semiconductor detector. Besides other applications this method is also used for chemical ultra-trace analysis in the quality control of drinking water, foodstuffs and semiconductor surfaces.

Total reflection XRF (TXRF) is applied as a routine procedure in the semiconductor industry to control the quality of wafer surfaces. In this procedure, X-rays strike the sample under a grazing incident angle so small that they are totally reflected at the surface. By using monochromatic undulator radiation as generated at the synchrotron radiation source BESSY II in Berlin, even elements of low atomic numbers (C to Al) can be verified down to a limit of one picogram. This corresponds to a detection limit of approximately 10^8 foreign atoms per cm^2 in the case of 200 mm diameter silicon wafers treated by a VPD (vapor phase decomposition) procedure.

Clean room conditions are necessary for this ultra-trace analysis to ensure that the wafer samples are not additionally contaminated when introduced in the analysis chamber. The new measuring arrangement for (T)XRF operates without reference

(Continued on Page 4)

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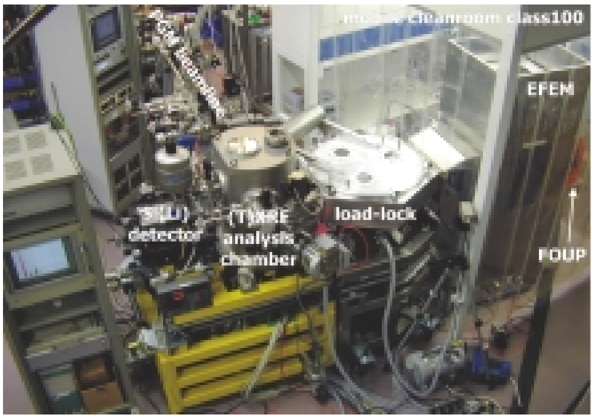
New measurement station for wafer quality control

(Continued from Page 3)

samples. It was taken into operation at the PTB laboratory at BESSY II in late 2002. The station can handle and position wafers of up to 300 mm in size automatically at a low additional contamination risk. The method has been developed in cooperation with the semiconductor industry. It is to be implemented for quality control of new wafer cleaning procedures.

In addition, the measuring station will serve the analysis of multi-elemental multi-layered systems with layer thicknesses in the nanometre region. The fine structure of the X-ray absorption edge can be investigated by variation of the excitation energy. That allows speciating compounds of light elements on wafer surfaces found only in small amounts in the nanogram region.



The new measuring station at PTB's plane grating monochromator beam line at BESSY II. A robot system (EFEM) transports the wafers from their standard transport containers (SMIF or, as in this case, FOUP) into a load lock from where a vacuum robot takes them to the (T)XRF analysis chamber.

Excellent ratings for PTB

As a departmental research institute of the German Federal Government, PTB was subjected to a comprehensive evaluation – with an excellent result.

On December 16, 2002, the Final Report of the Evaluation Commission – composed of several international experts – was submitted to the Federal Minister of Economics and Labour and presented to the public by the chairman of the Commission, Prof. Hartmut Weule, Karlsruhe University.

After one year of work, the Commission certified that PTB pursues and complies with its central mission to guarantee progress and reliability in metrology for society, economy and science, to the best advantage for German economy and society. Future PTB activities should therefore also be aligned consistently in terms of this mission. In summary, technical competence and quality of the work of the PTB staff members were assessed as excellent.

According to the Commission's findings PTB's technical orientation is widely conform to the present requirements. The Commission recommends restructuring and expanding two fields in view of their increasing importance: "metrology in chemistry" and "metrological information technologies." Patent applications and technology transfer should be consequently advanced to enhance exploitation of research results.

In respect to the sovereign duties and the neutrality of PTB, the legal form as a central national institute and the basic financing should be maintained as well as the scheme to dedicate 60 percent of the resources to research and development. The changes the Commission calls for – in the sense of increasing the direct responsibilities of PTB – are the implementation of a global budget and an increase in the budgets for materials and equipment, the adoption of the managerial organization structure well-tried and tested in business (Board of Directors and Executive Committee) and an end to the annual staff reduction by 1,5 percent.

Systematic and on-going analysis of customer requirements and of long-term developments in economy and society as well as regular technical assessments of all scientific activities by international experts are considered to be indispensable for the strategic planning of PTB tasks. PTB's scientific integration into the national, European and international environment is found to be very good. The establishment of an international network of cooperating national metrology institutes should be accelerated.

At the end of the year, the Commission will check the implementation of its recommendations.

The complete evaluation report can be obtained from the PTB Press Office. It is also available on the websites of PTB and of the German Federal Ministry for Economy and Labour (BMWA).
(http://www.ptb.de/ –cf. "What's new")
(http://www.bmwa.bund.de/)

Prof. Hartmut Weule (on the right) hands the Final Report of the Evaluation Commission to Minister Wolfgang Clement. On the left: Prof. Ernst O. Göbel, President of the PTB.

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