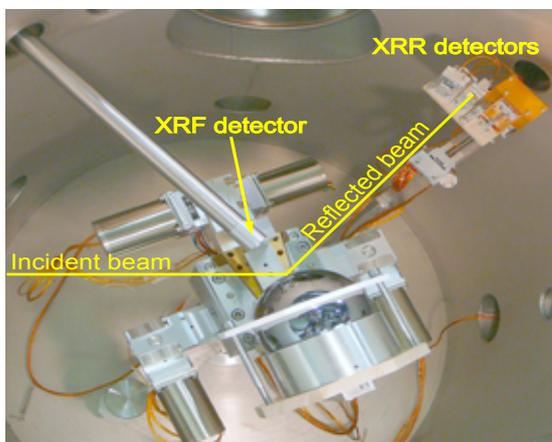


Avogadro Project: layer thickness of SiO₂

In the scope of the international Avogadro Project, an almost perfect silicon sphere is to be characterised with highest accuracy. In addition, the thickness of the unavoidable oxide layer of several nanometres on the surface of the silicon sphere has to be measured too. Measuring the thickness of the layer is based on X-ray reflectometry with synchrotron radiation of suitable photon energy as well as additional ellipsometric measurements.

Layer thicknesses in the nm range can be measured by means of X-ray reflectometry. Hereby, the fact is used that when varying the angle of incidence, in the reflected radiation, interference effects occur which result from the two reflections on the outer layer surface and on the inner interface. Commercial devices which use copper K α radiation with a photon energy of 8048 eV at almost grazing incidence are, however, less suitable here because at this photon energy, the reflection on the SiO₂/Si interface and therefore also the interference effects, are only weak. Clear interference effects appear, in contrast, in the area of the silicon K edge at 1840 eV and at the oxygen K edge at 540 eV, where strongly curved layers can also be measured with steep angles of incidence of up to 50°. Corresponding measurements were carried out with the UHV X-ray reflectometer at the undulator beamline in the PTB laboratory of the Berlin electron storage ring BESSY. Measurements at different photon energies



Silicon sphere with the detectors for the X-ray reflectometry (XRR) and X-ray fluorescence (XRF) measurements in the UHV reflectometer

in the environment of the oxygen K edge now allow the thicknesses of the SiO₂ layer as well as the carbonaceous contamination layer to be determined separately and the uncertainty of measurement of currently 0.2 nm to be reduced to the 0.1 nm demanded. The SiO₂ layer thicknesses determined by means of X-ray reflectometry at several points on the surface, will be used as reference values for ellipsometric measurements of the variations in layer thickness along great circles across the surface of the sphere.

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The most expensive spheres in the world

The international Avogadro Project coordinated by the PTB, is entering its last phase with two new, isotopically enriched silicon spheres.

The scientists hope that the new spheres will now bring about the crucial breakthrough in determining the Avogadro constant, i.e. the number of particles in one mole of amount of a substance. With

the spheres used till now, made of a silicon isotope mixture, the accuracy which would be necessary for the re-definition of the kilogram could not be reached.

On 10th April 2008 the two ²⁸Si spheres came to the PTB from the last stage of their production process in Australia. After the raw silicon from Russia had been processed into a cylindrical single crystal in the Institute for Crystal Growth (IKZ), Berlin, Australian colleagues at the National Measurement Institute (NMI) in Sydney had the last stage of production in their hands: to polish the silicon body into a perfect sphere. It is now the job of the PTB to determine the topography, the volume and the mass of the spheres with smallest measurement uncertainties. A measurement uncertainty of 0.1 nm (corresponding to the thickness of a monoatomic layer) is demanded for the radius of the sphere which is around 5 cm, and an uncertainty of 5 μ g, for the mass of the sphere weighing approximately 1 kilogram.

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Exact spectral emissivity measurements for radiation thermometry

Industry and research are increasingly relying on non-contact temperature measurements with the aid of heat radiation, for example, for the reliable and reproducible drying of car paint. In order to attain exact and reliable results, the emissivity of the measured surface has to be known. It can only be determined precisely in complex measuring facilities. The PTB has developed a modern emissivity measuring facility for industry-oriented calibrations.

Today, the accuracy of industrial temperature measurements carried out with contact-free radiation thermometers, is often no longer limited by the quality of the radiation thermometers, but rather by insufficient knowledge of the emissivity of the surface observed. Industrial radiation thermometers can furnish a resolution of up to 20 mK, with an uncertainty of 1 K for temperature measurements of 100 °C. In contrast to this, the directional spectral emissivities of surfaces can often only be specified with standard measurement uncertainties of 5 %. When measuring a temperature of 100 °C in the spectral range by around 10 μm , this corresponds to a temperature uncertainty of typically 5 K.

The emissivity is not a constant, but rather changes in general with changes of the surface (roughness, oxidation, impurities etc.), the observation angle, the observation wavelength as well as the temperature. Furthermore, it is often distributed inhomogeneously over the surface. Precise temperature measurements therefore demand exact knowledge of the emissivity. To determine the variety of dependencies of the emissivity on the above-mentioned parameters, complex measuring facilities are necessary.

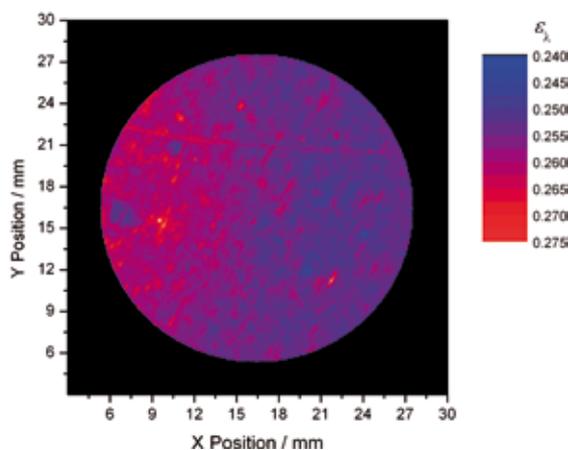
The spectral emissivity is measured in the PTB by comparing the radiances of a cavity radiator of high quality – resembling an almost ideal black body – with the sample to be investigated by means of a Fourier transform spectrometer, whereby the radiation of the environment and the inherent radiation of the spectrometer are taken into consideration. Holding the sample in a temperature-regulated hemisphere hereby guarantees a

constant radiation exchange with the environment. The apparatus allows the determination of the directional spectral emissivity as well as of the total emissivity of opaque samples under ambient conditions in a temperature range from 80 °C to 250 °C and a wavelength range from 4 μm to 40 μm under emission angles of 5° to 70° with a relative standard measurement uncertainty of better than 2 %. The extrapolation of the measured values of the directed spectral emissivity for emission angles above 70° then allows the hemispherical spectral emissivity, which is especially important for calculations of the net radiation exchange, as well as the total emissivity to be calculated. The homogeneity of the directional spectral emissivity at 4 μm is determined with the help of a thermography camera.

The results of the first orders from customers have served, for example, to optimise the paint drying process in the automobile industry, the thermal design of furnaces as well as the monitoring of glass forming processes.

Another measuring facility is currently being set up in the PTB which will allow emissivity measurements to be performed under vacuum conditions in an extended temperature and wavelength range – in particular for space applications.

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Local variation of the directed spectral emissivity ϵ_λ of a car paint sample at a wavelength of 4 μm , measured using a thermography camera.

Expansion of high tech materials

Industrial applications are ever more frequently demanding materials of highest thermal stability. A precision interferometer has been developed in the PTB to exactly measure this property. With this instrument, the change in length can be determined with highest accuracy in an absolute measurement as a function of temperature, time and – if necessary – ambient pressure.

Thermally stable materials play an important role in dimensional metrology and in precision manufacturing. The currently highest requirements on the thermal stability of critical components are made in EUV lithography of reflection masks and mirrors. These are, therefore, based on substrates made of high tech glass/ceramics which are to exhibit a very low thermal expansion coefficient α ($\alpha < 1 \cdot 10^{-8} \cdot \text{K}^{-1}$).

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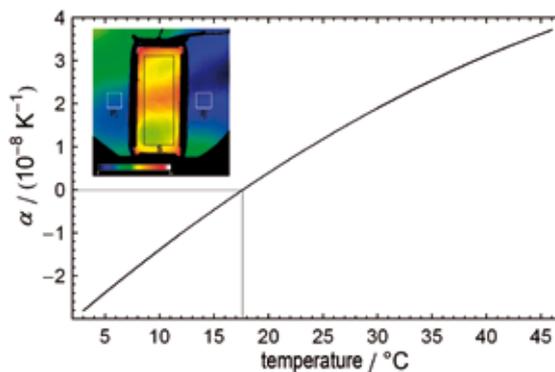
For the precise characterization of gauge-block-shaped measuring objects made of high tech materials, a precision interferometer was developed with the aim of measuring samples of up to 400 mm length with uncertainties in the sub-nanometer range. From such exact measurements of length, it is possible to calculate the thermal expansion coefficient as a function of the temperature with uncertainties of up to $2 \cdot 10^{-10} \cdot \text{K}^{-1}$. Furthermore, it is possible to get quantitative statements regarding the homogeneity of the thermal expansion, compressibility, length relaxations and also the long-term stability of samples.

Length measurements with sub-nm uncertainties demand, besides the application of frequency-stabilized lasers, the consideration of influences whose uncertainty contributions are difficult to minimize. For this purpose, various methods have been developed in the PTB in the last few years and these have been integrated into the measuring process. A new autocollimation process is cited as an example and this ensures that the lightwaves reach the surfaces of the measuring objects exactly perpendicularly. The so-called cosine error is hereby lowered to under $10^{-11} \cdot L$. Furthermore during the electronic evaluation of the interference pattern, the exact assignment of the sample position to the camera pixel coordinates is considered. This is particularly important when it comes to measuring objects whose end faces are non-parallel and when the influence of small temperature-induced changes of the lateral sample position can be corrected. By taking the temperature-related influence of the deflection of the end plate wrung to the back into consideration, the precision could be increased further.

When taking thermal expansion measurements on typical samples, length measurement uncertainties of 0.25 nm are now achieved.

In a recently completed international comparison measurement, the leading position of the PTB in the determination of thermal expansion coefficients was confirmed. The new possibilities for the precise characterization of high tech materials are already being used intensively by companies working in the fields of optics and precision manufacturing.

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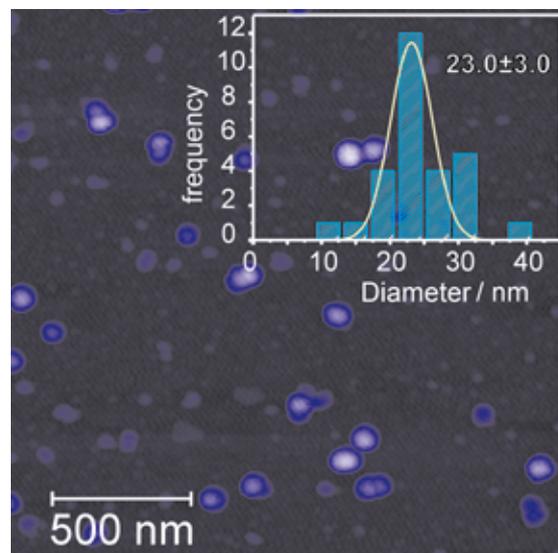


Thermal expansion coefficient (α) of a special glass with extremely low expansion. The progress of α shown here derives from a series of interferometrically measured lengths of a sample with parallel end surfaces as a function of temperature. In this example, there is a point of reversal at approximately 17.5 °C at which the thermal expansion disappears. Below this point, α fall in temperature causes an expansion of the material. The figure (upper left) shows a typical topography of the interference phase, which includes the front surface of the body and a wrung end plate. The averaged phase values within the rectangular areas on the end plate (left/right) and on the front surface of the body (middle), are the basis for the interferometrical length measurement.

Magnetic nanoparticles: suitable for cancer therapy?

A measuring procedure developed in the PTB can help to investigate in some detail the behaviour of magnetic nanoparticles which are used for cancer therapy.

Magnetic nanoparticles (with a size of some few to several hundred nanometres) are a new, promising means of fighting cancer. The particles serve as a carrier for drugs: "loaded" with the drugs, the nanoparticles are released into the blood stream, where they move until they come under the influence of a targeting magnetic field which holds them on to the tumour – until the drug has released its active agent. Besides this pharmaceutical effect, also a physical action can be applied: an electromagnetic a.c. field heats up the accumulated particles so much that they destroy the tumour. Both therapeutic concepts have the advantage of largely avoiding undesired side effects on the healthy tissue.



Atomic force microscopic image of magnetic nanoparticles of cobalt ferrite. The diagram shows the size distribution.

(Continued on page 4)

These procedures have already been successfully applied in the animal model and have, in part, already been tested on patients. Here it is important to know before application whether the particles tend to aggregate and thus might occlude blood vessels. Information about this can be gained by magnetorelaxometry developed at the PTB. In this procedure, the particles are shortly magnetised by a strong magnetic field in order to measure their relaxation after the switch-off of the field by means of superconducting quantum interferometers, so-called "SQUIDs". Conclusions on their aggregation behaviour in these media can be drawn from measurements of suspensions of nanoparticles in the serum or in whole blood. As

an example, it could be shown in this way that certain nanoparticles in the blood serum form clusters with a diameter of up to 200 nm – a clear indication of aggregation, so that these nanoparticles do not appear to be suitable for therapy.

At present, the high technical effort connected with the use of helium-cooled magnetic field sensors is still standing in the way of using this method routinely in practice. In a joint project with Braunschweig Technical University supported by the Ministry of Education and Research (BMBF), the procedure is currently being transferred to a simpler technology based on fluxgate magnetometers.

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Direct-reading personal dosimeters

How high is the current radiation exposure for the crew of the ISS? In the future, direct-reading dosimeters might answer this question clearly. To date the astronauts' exposure has only been measured using passive dosimeters. Evaluation takes place subsequently on Earth. The personal dosimeters developed at the PTB can, however, directly display radiation exposure. One of these dosimeters was patented and a prototype has been transferred to industry. A new prototype for possible use in space is being tested at present.

The radiation dose amounts to around 0.5 mSv per day on the International Space Station (ISS), in Low Earth Orbit at a height of approx. 400 km. The astronauts receive half of this dose in the few minutes during which the ISS crosses the radiation belt of the South Atlantic Anomaly several times a day. In view of this high dose received in a very short-time, carrying a direct-reading dosimeter with an alarm function is desirable. The astronauts could then retreat to better shielded areas of the space station when the dose increases.

The prototype dosimeter developed in the PTB for mixed radiation fields (neutrons and photons) is a handy device with a display which can be read directly and an alarm function. Whereas up till now several semiconductor detectors have always been used for constructing a neutron dosimeter, this dosimeter manages with a single detector (40 μm effective layer thickness and 1 cm^2 area), which is surrounded by several thin absorption layers. An industrially manufactured dosimeter based on the PTB's technology is already in use in the nuclear industry. For radiation fields with high neutron energies between 1 MeV and 100 MeV, as they appear at high-energy accelerators and also in space, the dose equivalent response, however, varies by more than one factor of ten.

In the meantime, a new neutron dosimeter has been developed at the PTB which is better suited for radiation fields with high energies. The new prototype essentially differs from the earlier one through the use of a thinner and smaller semiconductor detector (5.6 μm effective layer thickness, 0.25 cm^2 area) and the integration of pulses with a smaller pulse height (200 keV to 1 MeV). The dose equivalent response for neutrons only varies by $\pm 40\%$ in the neutron energy range between 1 MeV and 100 MeV.

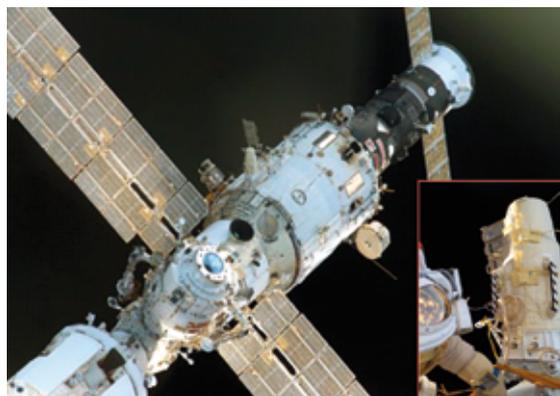
Apart from measurements, Monte-Carlo calculations for the response were also carried out. The results agree well with the measurements in view of the large energy range, the complex computation models and the construction of the dosimeter which has been simplified for the calculation. The dosimeter also seems suitable for use on the ISS. It is being tested for possible use in space in the scope of a project supported by ESA.

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A "phantom" (called Matroshka) similar to a person on the outside of the ISS wearing a waistcoat with various dosimeters for test purposes. (Photos: ESA)