

## Ultraprecise flatness measurements of optical surfaces

**A new method allows ultraprecise measurement of the topography of plane and weakly curved optical surfaces. With the new apparatus shape deviations from an ideal plane surface can be determined with a measurement uncertainty of a few atomic diameters. The measurements do not rely on an external flatness standard.**

Highest quality plane surfaces are needed in several fields of industry and research. For instance, well-characterized and highly accurate flat surfaces are used to calibrate interferometers. Until now, the actual deviation of a surface from flatness is determined with the aid of a liquid mercury mirror which at present serves as the national flatness standard but slightly deviates from the ideal plane due to the spherical shape of the earth and the surface tension.

To meet the industrial demand for improved accuracy PTB has developed a new device to perform highly accurate and traceable measurements of the topography of plane and weakly curved optical surfaces. The method is based on angle measurements. The reflection angles of a light beam are successively measured at different spots on the surface of the specimen and the differences between the angle measurements are determined. The spot positions are separated by a constant lateral displacement (shear) of a few millimetres to centimetres. The reflection angles are determined with a highly accurate electronic measuring device (an autocollimator). When repeated measurements of scans of 130 mm length across plane surfaces are performed the topography values typically deviate by merely one atomic diameter, i.e.: 0,1 nm to

0,2 nm. The measurement uncertainty of the new facility is 1 nanometre or better.

The method represents a novel approach to measure flatness deviations as it does not require an external flatness standard. Instead, the method utilizes the straight propagation of the light beam as a reference. The measurements, from which the topography is derived by application of mathematical algorithms, can be directly traced to the SI units of angle and length.

The facility has the potential to become a highly precise primary standard for straightness and flatness with a measurement uncertainty in the sub-nanometre range and to replace the liquid mercury mirror.

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*The new apparatus can measure specimens of up to 500 mm in diameter.*

## Length measurements with nanometre-accuracy

**Ever smaller manufacturing tolerances of precision components require measurement devices of increasing accuracy to assure the product quality. In cooperation with two German companies in the fields of machine tool control and coordinate measuring devices PTB has developed a length comparator. The new device significantly reduces the hitherto achievable measurement uncertainties for measurement lengths of 2 mm to 610 mm.**

Until now world-wide the spacing of structures (as SI-traceable length measurements) can only be measured with an uncertainty of roughly 20 nm. Yet today highly precise position control circuits and ever more compact integrated circuits already

require length measurements with smaller measurement uncertainties. The new nanometre comparator allows length measuring systems as used in precision processing machines to be characterized with the small measurement uncertainty required. Objects up to 450 mm in width can be handled by the comparator so that one-dimensional structure distances on two-dimensional specimens such as photomasks of the semiconductor industry can be determined.

Due to the high requirements on accuracy the comparator was constructed and installed with great expenditure and effort. For instance, the sliding carriage to move the specimen is fitted on air

*Continued on Page 2*

### Length measurements with nanometre-accuracy

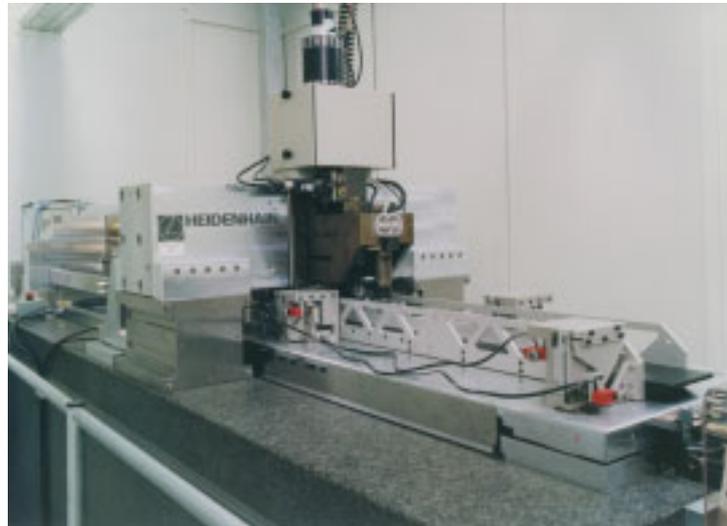
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cushion bearings and equipped with piezo-actuators to compensate the guide deviations of the bearings detected by the measurement system. The displacement is measured with a special in-vacuum interferometer set-up, in which the interferometer translation is encapsulated in a metal bellows. This practically eliminates the influence of the refractive index of air on the measurement result – which otherwise would dominate the measurement uncertainties. The interferometer itself consists of three reference beams in an arrangement for differentiation measurements with reflectors on the rigid frame of the comparator which compensate the mechanically and thermally induced shifts between the microscope bridge and the beam-splitter of the interferometer.

First comparative measurements with other length comparators from PTB and external sources using 200 mm line scale standards demonstrate agreements better than 30 nm which correspond roughly to the measurement uncertainty of these comparators. In the near future calibration services with measurement uncertainties below 10 nm are to be offered with the new nano-comparator. Further optimization of the comparator will aim to reduce the measurement uncertainty to below 5 nm.

For instance, due to the length of the measurement circles measurement uncertainties below 1 nm appear to be unrealistic even for small measurement lengths. Therefore PTB is preparing a further measurement facility for measurement lengths below 2 mm. First investigations with this facility are presented in the next contribution.

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The new nano-comparator.

## Contrast agents for magnetic resonance imaging

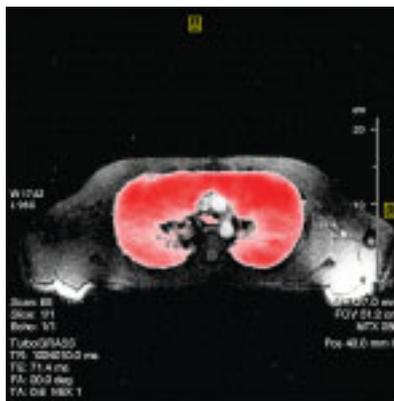
**PTB has developed an apparatus to produce hyper-polarized xenon for (nuclear spin) magnetic resonance imaging of the lungs, the brain, and potentially for further methods of medical diagnosis.**

Contrast agents are deployed in medical imaging techniques for anatomical presentations as well as for functional investigations to increase the significance of the image. For instance, imaging the lungs by conventional magnetic resonance imaging (MRI) is difficult because the proton signal from lung tissue declines swiftly over time and because the air contained in the lungs does not yield any MRI signal. The stable rare gas isotopes ( $^3\text{He}$ ,  $^{129}\text{Xe}$ ) can be (hyper-) polarized (Hp) via optical pumping with laser radiation, i.e.: the nuclear spins can be aligned to a high degree. Rare gases prepared in that way (HpHe; HpXe) can be deployed as contrast agents for magnetic resonance imaging of the lungs and for functional diagnostics of the brain.

Several research groups in Europe (e.g.: at the University of Mainz) and America have already successfully imaged the lungs with magnetic resonance techniques by letting patients inhale HpHe. The groups have also carried out dynamic studies of pulmonary functions with the new technique. As a by-product of tritium production  $^3\text{He}$  is not a naturally occurring isotope on earth.  $^{129}\text{Xe}$  on the other hand, has a natural abundance of 26 %. In addition, xenon is easily dissolved in blood and tissues, for instance, in brain tissue. This may open up further medical applications.

At PTB a new apparatus has been developed to produce sufficient amounts of HpXe (0,5 l/h) with polarization degrees of 10 % to 20 % (referred to  $^{129}\text{Xe}$ ) to image the lungs and to perform (time-resolved) spectroscopic imaging of the brain by means of MRI. Goals are to develop new fields of medical applications for HpXe as well as to compare quantitatively the images of lungs obtained with HpXe and HpHe. The latter activity aims at clarifying if HpXe can replace HpHe in lung imaging and distinguishing the respective medical applications. Furthermore, it is worthwhile to consider applications for HpHe and HpXe as contrast agents in SQUID measuring-techniques.

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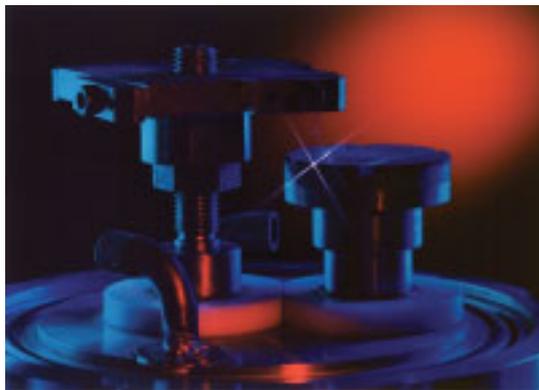


Superposition of  $^{129}\text{Xe}$  lung images (red, planar resolution  $4 \times 4 \text{ mm}^2$ ) and conventional proton images (white).

# Inherently safe despite higher pressure

The chemical industry can in future use electrical equipment which is also certified for operation under higher than atmospheric conditions. This is owed to the current investigations at PTB expanding the concept of intrinsic safety also to higher temperatures and pressures.

An electrical device is termed or intrinsically safe if it is impossible to ignite an explosion with the device despite the possibility of electrical sparks. In this regard intrinsically safe means the possible maximum energy in an electrical circuit is limited so that occurring sparks are not provided with enough energy to ignite an explosive mixture of flammable gases and air. Such gaseous mixtures are very customary in chemical and petrochemical industries.



In such a spark-test apparatus device tungsten wires scratch along a rotating cadmium disc and produce electrical sparks – until an explosion is ignited in the surrounding gaseous mixture.

Minimum ignition curves are crucial to the attribute intrinsically safe. Such curves mark the temperatures and ambient conditions at which an ignition probability of  $10^{-3}$  is established. In this context PTB has investigated the ignition behaviour of electrical sparks and the resulting explosions at higher temperatures and pressures. The relevant limiting curves for different gaseous mixtures have been determined with a spark-test apparatus device at ambient conditions at atmosphere and above. With the obtained results PTB can now test and legally certify intrinsically safe devices for temperatures up to  $100\text{ }^{\circ}\text{C}$  and pressures up to 5 bar.

The advantages for producers of electrical devices and subsequently for the chemical industry are unequivocal: producers could not guarantee the safe operation of their devices beyond the certified range and – in case the devices were deployed at all – the chemical industry was forced to take the liability at their own risk. The new results for inherent safety, however, lead to extended certifications and thus to a higher safety standard in the chemical industry.

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# Combined scanning tunnelling microscope and x-ray interferometer

PTB has combined a scanning tunnelling microscope (as the actual measurement instrument) and an x-ray interferometer (as a length standard) in one experimental set up. Standards – even containing atom size lateral structures – can be calibrated with such an apparatus.

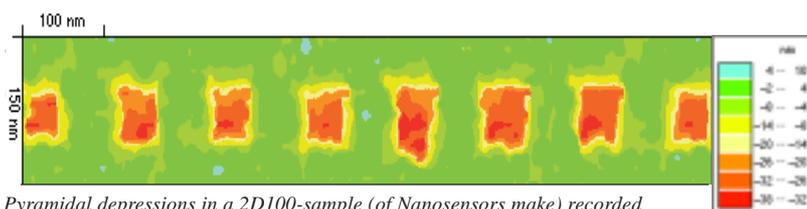
In nanotechnology atomic structures are commonly investigated with a scanning tunnelling microscope (STM). In the new experimental set up an STM is applied to determine the topography of the sample surface as well. An x-ray interferometer produced by PTB is used as a high precision adjusting mechanism. The interferometer is made of dislocation-free, single crystal silicon. The samples are adjusted in steps of  $0,192\text{ nm}$ , the spacing between the silicon (220) planes. In the last years this

spacing has been traced very accurately to the SI base unit one meter. A further advantage of the construction: the periodic sequence of the atomic planes is free of non-linearities, which have to be taken into account when measuring structures of a few fractions of the applied optical wavelength in size, for instance, as in case of a laser-based interferometer.

The experimental stand at PTB demonstrated an extremely high stability and, in correspondence, a very good reproducibility of results. First investigations were carried out on a two-dimensional grating with several smallpits etched into the surface with a nominal lateral spacing of  $100\text{ nm}$  (so-called 2D100-lattice). For this distance the calibration with the silicon grating scale produced a value of  $(100,12 \pm 0,36\text{ nm})$  over a measurement path of  $0,8\text{ }\mu\text{m}$ .

As a result of these investigations which were carried out in cooperation with the National Physical Laboratory (NPL), Teddington, U. K., PTB is planning the installation of a permanent in-house measurement station.

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Pyramidal depressions in a 2D100-sample (of Nanosensors make) recorded with the novel combination of measuring instruments.

# New primary standard flow liquid measurements

**The new hydrodynamic test field at PTB is operational. The currently achievable measurement uncertainties for flow and quantity measurements of flowing liquids are lower than half of their previous values. Therewith the facility is a worldwide inimitable primary standard for liquid measurements.**

Concerning the measurement of flow rate and totalized mass or volume of flowing liquids, industry and research as well as medicine, environment and consumer protection call for specificities which to be guaranteed demand PTB, in turn, to represent the appropriate units with expanded measurement uncertainties below 0,02 %. Internationally, the best current values lie between 0,04 % and 0,05 %.

The new hydrodynamic test field utilizes water as a test fluid. It will achieve the required uncertainty of 0,02 % in liquid flow in the flow range from 0,3 m<sup>3</sup>/h to 2100 m<sup>3</sup>/h. This follows from the projected results of the measurement uncer-

tainty budget as well as from the acceptance tests of the major components and parts. The crucial preconditions were already created with the measurement-technical concept of the facility. The facility combines two different-type reference systems based upon totally different measurement principles: a gravimetric reference comprising three weighing systems (for 30 t, 3 t and 300 kg, respectively, maximum load) and a geometrically verified volumetric reference (a 250-liter compact pipe prover), which are functionally interconnected via the water flow section. Through appropriate comparative measurements with both systems it is possible to recognize and evaluate the effects of hitherto hardly realistically assessable parameters on the measurement uncertainties. In this field such a concept has been realized for the first time. Furthermore various other innovative partial solutions have been implemented to achieve the aspired measurement parameters. Novel measurement-technical and design concepts were developed, e.g., for the weighing system, the flow diverters and the calibration procedure for the pipe prover.

Exact verification of the measurement uncertainties is currently one of the key activities of the scientific and experimental assignments at the hydrodynamic test field. Substantial research activities will also deal with integrating optical methods to measure velocities in the liquid measurements. Beyond that intensive investigations will concentrate on developing transfer methods and standards that are independent of the medium being measured.



Measurement section of the test field with the weighing system (in the rear: 30-t balance).

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## Events

### European Vacuum Congress Berlin 2003

(8<sup>th</sup> European Vacuum Conference in conjunction with the 2<sup>nd</sup> Annual Conference of the German Vacuum Society DVG)  
Rathaus Schöneberg, Berlin, 23 to 26 June 2003  
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### Temperatur 2003

Berlin, 8 and 9 September 2003  
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