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Sirius – Brazil’s bright synchrotron future 9

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Printed in the UK by Warners (Midlands) plc, The Maltings, West Street, Bourne, Lincolnshire PE10 9PH

IOP Publishing

Physics World
 Temple Circus, Temple Way, Bristol BS1 6HG, UK
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 Web: physicsworld.com

Focus on: **Vacuum technology**

Welcome to this focus issue on vacuum science and technology, in which you can find out how researchers are developing a better definition of the pascal and why a three-year European project – ending this month – is setting new vacuum standards for industrial processing. We report on the importance of “non-evaporable getter” coatings at Brazil’s new synchrotron X-ray source, and hear from Agilent about the latest pumping technology. You can also find out about the recent merger between Atlas Copco and Edwards, while our cover story looks at an experiment at CERN that’s shedding light on how clouds form.

Matin Durrani, Editor, *Physics World*

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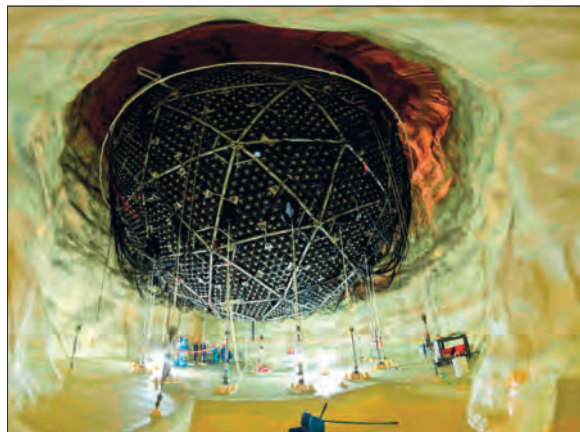
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Vacuum boosts neutrino detection

Vacuum technology is playing a key role in a new experiment being built at the SNOLAB facility in Canada, which will make some of the best measurements yet of the mysterious properties of neutrinos. Located 2km underground at the Vale Creighton Mine near Sudbury in Ontario, the SNO+ detector will measure neutrinos emitted by the Sun, the Earth and from nuclear reactors in Canada. In particular, it will let researchers study an exotic nuclear process called neutrinoless double beta decay. Based on modifications to the original SNO experiment, which ran from 1999 to 2006, SNO+ is expected to begin operation next year.

Since the discovery 15 years ago that neutrinos can oscillate between their three different types, which showed that neutrinos have a small mass, several major experiments have honed in on the properties of these enigmatic particles. Neutrinos are so unreactive that they require very large detectors to maximize the probability of a strike. SNO+ contains a 12 m-diameter acrylic sphere filled with 800 tonnes of liquid scintillator called linear alkyl-benzene, which



emits ultraviolet light in response to ionizing radiation caused by a passing neutrino. A highly sensitive array of 9500 photomultiplier tubes surrounding the vessel will allow physicists to detect and analyse the signals.

According to SNO+ member Steve Biller, who is a particle physicist at the University of Oxford in the UK, vacuum is critical in order to remove trace heavy metal contaminants from the scintillator. "There is a lot of piping and since we are sensitive to very low levels of contamination, especially from radon gas, the liquid

Scintillating science

The SNO+ detector requires an ultraclean environment to study rare processes such as neutrinoless double beta decay.

handling system has to be brought down to vacuum pressures so that we can be sure of the reliability of our results," he explains.

To this end, the SNO+ team has worked with vacuum manufacturers Busch to develop pumps that were recently installed at the experiment's detector purification plant. A COBRA dry-screw pump controls ultra-high purity multi-stage distillation at pressures of 20 mbar and temperatures of 238 °C, while the equipment will also strip the scintillator under vacuum at 200 mbar and 100 °C to remove radon, krypton, argon and oxygen, and to control the scintillator humidity level.

After-condensers installed at the discharge outlet on the COBRA pump also help cool the exhaust gases and thereby enable process vapours to condense back into liquid for safe recovery, says the firm. "The team from Busch Canada worked with Busch USA, which designed the original system, to supply and install the vacuum equipment for this unusual and prestigious application," said the company in a statement.

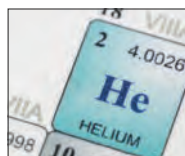
Matthew Chalmers

Research

Physicists bid to reduce helium costs

A pilot programme aimed at providing helium at affordable prices for US researchers who need only small amounts of the element has been launched by the American Physical Society (APS). The plan will see the Defense Logistics Agency (DLA), which buys helium on behalf of the US Department of Defense, negotiate the cost for helium with suppliers on behalf of researchers who are funded by US government grants. "The hope is that some universities in a poor position to negotiate with particular vendors will have access to helium," says Moses Chan, a low-temperature physicist at Penn State University in Pennsylvania.

Helium gas is essential for customized, vacuum-leak detection systems, and is used across science, industry and medicine for cooling and manufacturing. But with growing demand



Short supply

Despite being the second most abundant element in the universe, terrestrial helium is rare and demand for the inert gas is higher than ever.

pushing up the price of helium, shortages of the finite and non-renewable commodity have occurred regularly in recent years. While large laboratories can negotiate a good price for helium, smaller users buying in the region of 100l at a time find that they are paying more. Users at the end of a supplier's delivery route can also lose up to a quarter of their purchase due to evaporation.

The APS team is currently looking for research groups to participate in the programme, according to its policy analyst Mark Elssesser, who will serve as a liaison between researchers and the DLA. "Then we'll look at where users are located, where supply needs are and other issues," he says. The consortium expects to review the helium-purchasing plan in December and, if successful, it will be rolled out in 2016.

Pfeiffer Vacuum, meanwhile, has recently launched helium-recovery units that make it possible to recycle the helium that is used in leak detection. Designed as stand-alone systems that work regardless of the leak-detection manufacturer, the units allow up to 98% of gas to be recovered.

In anticipation of dwindling global supplies, some firms are even eliminating their need for helium altogether. Gigaphoton, which provides advanced lasers for lithography in the semiconductor sector, estimates that 100 million litres of helium are used worldwide every year as a purge gas to prevent optical components from being damaged. The company has therefore recently developed the first purge process to use nitrogen. "We will strive to maintain a stable production environment and stay active to be conscious of the global environment," says Gigaphoton chief executive Hitoshi Tomaru.

Peter Gwynne and Matthew Chalmers



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Fusion

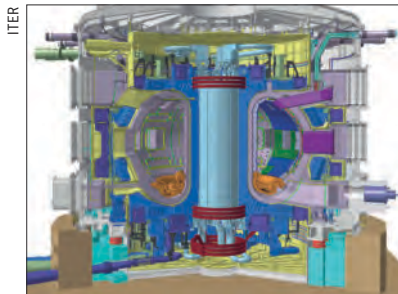
Pfeiffer wins ITER vacuum contract

German pump manufacturer Pfeiffer Vacuum has been awarded an open-ended contract to develop bespoke vacuum pumps for the ITER fusion reactor, which is currently under construction in Cadarache, France. ITER, which is expected to be operational in 2020, is a giant international experiment designed to demonstrate the feasibility of fusion power. Based around a 23 000 tonne doughnut-shaped chamber in which a plasma will be heated to temperatures of 150 million degrees, the hope is that hydrogen nuclei will fuse into helium with the release of more energy than was put in.

An ultra-high vacuum is vital to keep the chamber free of impurities, and strong magnetic fields prevent elements from coming in contact with the reactor walls. Pfeiffer will supply ITER with innovative vacuum pumps designed from stainless steel, which have an extremely high external leak-tightness. A new technology that has been used for the first time in vacuum pumps will also prevent

Leak tight

The ITER tokamak will be the largest ever built, with a plasma volume of 840 m³.



contamination between pump-gear lubrication and ITER's fusion fuel.

The new pumps will also be used to evacuate chambers through which high-powered neutral particles will be injected in order to heat the plasma, and the firm will also deploy its products for pressure measurements and leak detection. "The leak-tightness of welds and flange connections must be individually checked and ensured before the various chambers can be evacuated, demanding portable leak detectors in regions that are difficult to access," says the firm in a statement.

Matthew Chalmers

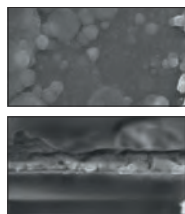
Instrumentation

Vacuum lamp sets record

Researchers in Japan have developed a solid-state lamp that sets new standards in vacuum-UV (VUV) emission. Producing high-energy electromagnetic radiation with a wavelength of 140–220 nm – the shortest ever recorded for such a device – the lamp will provide a powerful tool for sensing, material processing and the decomposition of chemical materials (*APL Materials* 2 046110).

VUV light is so named because although it can propagate in a vacuum it is quickly absorbed by oxygen in the air. This property makes VUV light useful for industrial applications such as sterilizing medical devices or cleaning semiconductor substrates because when it strikes a surface, the light generates highly reactive oxygen radicals that can destroy any microbes that are present.

Existing VUV lamps tend to be bulky and expensive, plus they use a lot of power and contain toxic gasses. The new lamp avoids these issues, because it is fabricated with a solid-

**Invisible emission**

A KMgF₃ thin-film prepared by pulsed laser deposition forms the basis of the new solid-state VUV lamp.

state phosphor made from a thin film of potassium-manganese-fluoride (KMgF₃). Solid-state phosphor is relatively easy to make, avoids the use of toxic gasses and does not require expensive rare earth elements.

"Our lamp is a promising light source in terms of lifetime, size, heat conduction and stability," says team leader Shingo Ono of the Nagoya Institute of Technology in Japan. "It has the potential to be an excellent alternate light source to low-pressure mercury lamps, excimer lamps and deuterium lamps."

The key challenge for Ono's team was to find a way to safely fabricate the phosphor thin films using fluoride, which itself is a toxic and corrosive chemical. Instead of using a conventional inflow of gaseous fluoride to coat the surface of the KMgF₃ film, the researchers discovered a safer fabrication route based on pulsed laser deposition. According to the team, solid-state phosphor also promises to make future UV lamps much smaller and longer lasting – mirroring the advances made in general solid-state lighting.

Matthew Chalmers

Technology briefs

Edwards expands in steel

Edwards Vacuum has joined forces with two major steel contractors in the Czech Republic and the Middle East to supply dry-pumping technology for vacuum degassing, which is used across the industry to remove impurities. The firm has delivered two major orders for standard steel degassing modules that will let manufacturers produce high-quality steel in a cheaper and more environmentally friendly way. The systems are capable of degassing 200-tonne batches of liquid steel, making them among the largest in the world using dry pump technology. Mechanical dry pumps have lower running costs, higher pumping speeds and minimal maintenance compared to traditional steam ejectors.

Raptor keeps cameras cool

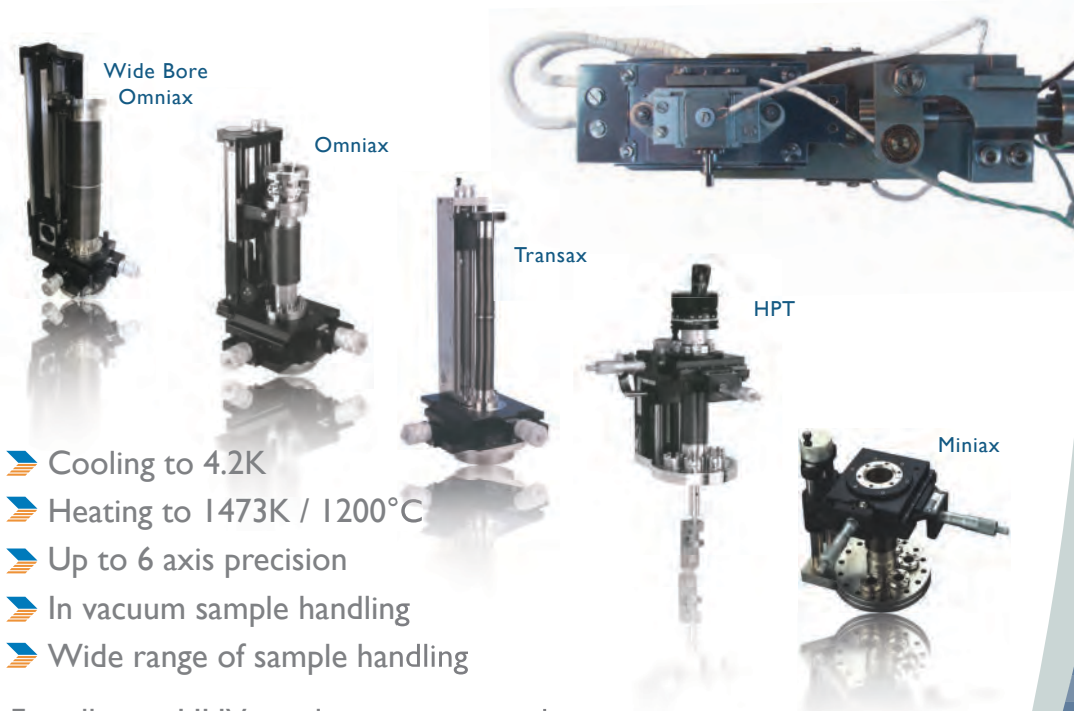
High-performance digital camera manufacturers Raptor Photonics has developed advanced vacuum technology that reduces dark current and minimizes blemishes during longer exposure times. The PentaVac technology, which consists of a small vacuum chamber enclosed in a lightweight package that houses an image sensor, also ensures a longer camera lifetime. Its main role is to allow the sensor to be cooled to low temperatures, thereby minimizing the effects of thermal fluctuations on charge-coupled device (CCD) sensors, but the vacuum also protects the sensor's quality and integrity. Proprietary sealing methods and sophisticated multistage getters assembled in a class-100 clean room environment ensure a lifetime vacuum guarantee with a mean time between failures of 100 years.

Oerlikon wins design award

Oerlikon Leybold Vacuum has received the Red Dot Product Design Award for its PHOENIX L500i leak detector. According to the firm, the PHOENIX L500i is the fastest leak detector on the market, and combines the sophistication of a very high-tech piece of equipment with an aesthetically pleasing and functional appearance that marks it out from its competitors. The device is also compact, making it easy to manoeuvre into tight workspaces, and boasts an iPad interface and simplified controls that have been optimized ergonomically. The Red Dot Design Award claims to represent the best in design and business and provide an international indicator for product design.



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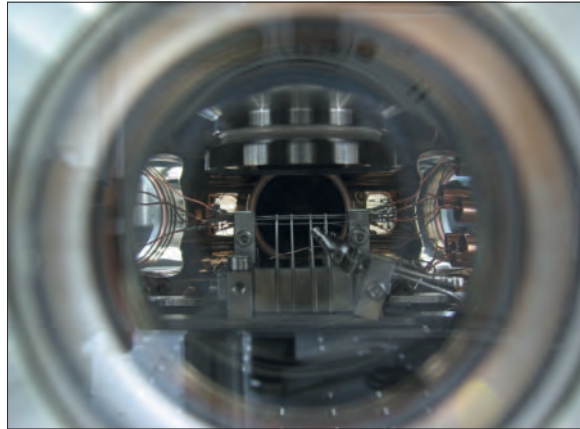


Research

Vacuum aids magnetic measurement

Vacuum technology has helped an international team of physicists to measure the extremely weak magnetic interaction between two individual electrons. Using experimental techniques first developed for quantum-information and ion-trapping technologies, the team made its measurement despite the presence of magnetic noise, which is a million times stronger than the signal it was seeking. Apart from measuring magnetism at the shortest length scale thus far, the researchers say that their technique could be applied to other measurement scenarios where noise is a dominant factor, such as for quantum error corrections.

Physicists have long known that the electron has an intrinsic angular momentum and an associated magnetic moment – known as its spin magnetic moment. Although the magnetic field of an individual electron has been accurately measured before, the magnetic interactions between two electrons have proved much harder to observe. The problem is that when two electrons are very close together, the magnetic interactions are drowned out by the effects of Pauli's exclusion principle



Courtesy: Yoni Dalia

and Coulomb electrical repulsion. Although these two effects weaken as the electrons move further apart, so does the magnetic interaction, which is then almost completely obscured by ambient magnetic noise.

One way of coping with this noise is to completely isolate the electrons from the environment – a technique that is often used in quantum-information processing. This is the concept that Shlomi Kotler, of the Weizmann Institute of Science, Israel, and colleagues adopted to make their exquisite measurements on two strontium-88 ions in a vac-

Where the action is

Viewed through the vacuum-chamber window, this linear Paul trap hold pairs of strontium ions that are used to measure the interaction between pairs of electrons.

uum chamber at a fixed distance of $2\mu\text{m}$ from one another, held using a Paul ion trap. Each ion has a single ground-state, spin-1/2 valence electron and no nuclear spin. Using lasers tuned to the atomic transition of the ions, the team prepared the electrons in an initial state where the north pole of one electron is facing the north pole of the other. Like a regular bar magnet, the like poles repel each other and rotate, thereby interacting. But as the magnets in this case are electrons, the electrons become entangled in what Kotler describes as “both a north–north and south–south facing state”.

Even more surprising is that this naturally created entanglement lasts for 15 s – a very long time for a system to remain in a coherent, quantum state. After that time, the researchers use laser pulses to detect whether the ions' north poles are facing or anti-facing each other. By varying the separation between the two ions, the researchers were able to measure the strength of the magnetic interaction as a function of distance – confirming the expected inverse-cubic dependence of the interaction.

Tushna Commissariat

Devices

NASA targets classic vacuum tubes

Researchers at NASA's Ames Research Center in the US are trying to turn back the clock 50 years and breathe new life into classic vacuum tubes. These tubes, which are big, bulky and soak up lots of power, were once routinely found in radio and TV sets but have almost entirely been replaced by transistor technology. Yet Jin-Woo Han and Meyya Meyyappan – writing in the July issue of *IEEE Spectrum* – believe that vacuum-tube technology could make its way back into computers.

Traditional vacuum tubes consist of two electrodes separated by a grid that controls the flow of current between them, with all three components housed inside an evacuated glass tube. The vacuum is needed to

Vacuum-tube technology could make its way back into computers

prevent the electrons from colliding with air molecules, which would otherwise ionize the gas, damaging both the cathode and letting uncontrolled currents flow. Vacuum-channel transistors also consist of two electrodes, but this time the electrodes are separated not by millimetres, but by just nanometres.

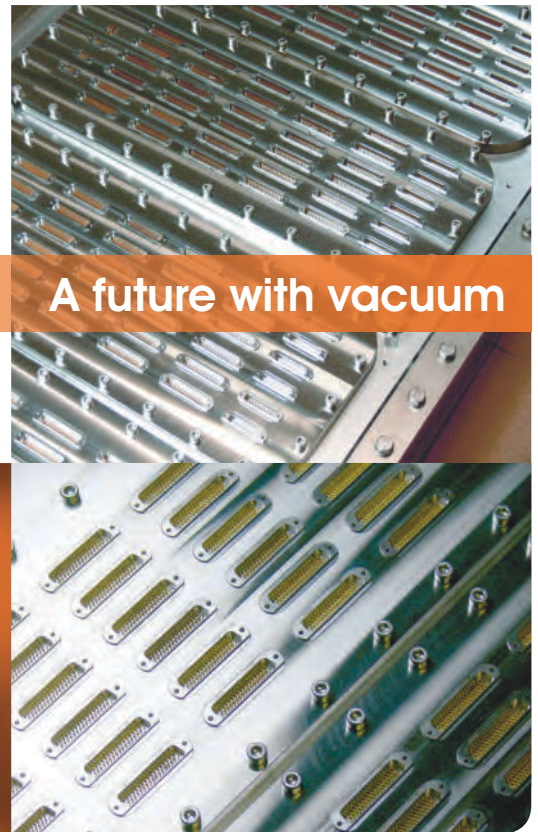
As the gap is so small, the chance of an electron colliding with any gas molecules is tiny enough that a vacuum is not actually needed – in fact, NASA's vacuum-channel transistor uses helium at atmospheric pressure in the gap. Moreover, the cathode does not have to be heated up to emit electrons using a power-hungry heating element. Instead, electrons are emitted simply by placing the cath-

ode in a strong static electric field, which thereby reduces power costs.

The other benefit of the technique is that the switching speed of a transistor depends on how fast electrons flow through it. As the electrons in the new NASA devices travel through helium at atmospheric pressure, they move much more quickly than in a solid-state device. In fact, NASA's prototype can switch at 460 GHz – meaning that vacuum-channel transistors could operate at terahertz frequencies, which is notoriously hard to do with conventional technology. Meyyappan, who is editor-in-chief of *Materials Research Express* from IOP Publishing, which also publishes *Physics World*, admits that his transistor is still at the lab stage. In particular, NASA will have to find ways of mass-producing the device and reducing the operating voltage from its present value of 10 V.

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The Sirius vacuum challenge

The cutting-edge vacuum technology under development at Brazil's new synchrotron, Sirius, will allow more stable and brighter X-ray beams for the study of matter at shorter length and timescales, describes **Rafael Molena Seraphim**

In the past few decades, synchrotron light sources have significantly advanced our knowledge of the structure and properties of materials. Based on electrons travelling around large storage rings, synchrotrons produce high-brilliance radiation ranging from the infrared to hard X-ray region of the spectrum. By tapping off this radiation and sending it along numerous beamlines that fan out tangentially from the storage ring, many different and unique experiments can be carried out simultaneously. In addition to advanced X-ray crystallography and imaging methods, synchrotrons allow scientists to link the atomic-scale structure of materials with their macroscopic properties and increasingly are able to characterize working devices such as solar cells in their native states.

Driving synchrotron science forward is the quest for ever-smaller beam “emittance” – a measure of the lateral spread of the electron beam. This results in a brighter and more collimated X-ray output, allowing the study of smaller features and faster processes. Around 50 synchrotrons of varying sizes and capabilities are in operation worldwide, and some of the top facilities are planning upgrades based on storage rings with very low emittance. The Advanced Photon Source (APS) in the US, the European Synchrotron Radiation Facility (ESRF) in France, and SPring-8 in Japan are all proposing ambitious upgrades, while other labs are building entirely new machines – namely MAX IV in Sweden and Sirius in Brazil.

Sirius will replace Brazil's existing, second-generation synchrotron with one of the brightest X-ray sources on the planet. Boasting a very low emittance of 0.28 nm rad, it will also be the first third-generation light source in Latin America. Ground works for the new facility are com-



High production Intertwisted titanium, zirconium and vanadium wires being assembled into Sirius vacuum chambers in the NEG-coating rig at the Brazilian Light Source Laboratory. These are then inserted into a solenoid where a DC magnetron sputtering process completes the coating process. When fully loaded, the rig can simultaneously coat six 3.2 m-long vacuum chambers.

plete and construction is scheduled to start at the end of this year, with engineering teams already deeply engaged in the design, prototyping and R&D of the machine's sub-systems. Commissioning is planned to start in mid-2016 and the first beam for users in mid-2017, which is a tight schedule.

Distributed environment

In all synchrotron light sources, an ultra-high-vacuum environment is essential in order to accelerate electrons and extract the radiation that they produce. The most demanding component of such a facility is the storage ring, which must operate at pressures below 10^{-9} mbar to minimize beam-gas scattering and other effects that reduce the lifetime of the electron beam. Since the cross section of the vacuum chamber is very small compared with its length (the Sirius storage ring will have a circumference of 518 m and a chamber diameter of 24 mm, for instance), these machines tend to be pumped in a discrete or distributed manner.

Most modern storage rings use discrete pumping, whereby hundreds of pumps are installed at intervals of around 1.5 m or less, which requires vacuum chambers with good conductance and sufficient longitudinal space in which to place the pumps. But this approach goes completely against the design of next-generation synchrotrons, which in order to reduce beam emittance

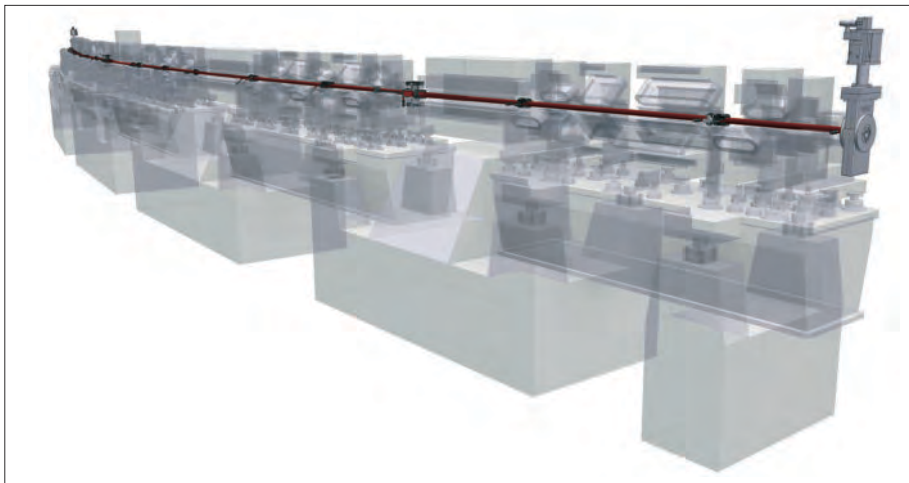
will guide electrons more gradually around the ring. Technically, Sirius is based on a “multi-bend achromat” design that involves many small-aperture magnets and leaves little space for other components. In total, the storage ring will comprise 20 “five-bend cells” and 20 straight sections.

Distributed pumping based on non-evaporable getter (NEG) technology offers a more effective option for such a compact “lattice”. NEG coatings, which were developed by the CERN particle-physics lab, are based on thin films that coat the inner surfaces of a vacuum chamber and have a chemical affinity for gas molecules. CERN has used the technology extensively in the beam pipes of the Large Hadron Collider, which requires extreme vacuum conditions. Today, NEG coatings are a proven industrialized technology used in many other settings. The first synchrotron to extensively use NEG coatings was France's national light source Soleil, with 56% of the machine exploiting the technology. Sirius and MAX IV will be the first synchrotron light sources to base vacuum pumping of the storage ring mainly on NEG coatings, with more than 95% of the vacuum chambers being coated.

The Sirius challenge

The highly compact lattice of the Sirius storage ring leaves very little space for components and therefore requires nar-

Synchrotrons



LNLS

currently in their final design stages, the coating procedure for the complex vacuum chambers is still under development.

Following the NEG coating process, the vacuum chambers will be filled with nitrogen and stored in batches according to their assembly in the storage ring. Since the coatings must be activated by heating them *in situ* (in a procedure called a bake-out), Sirius requires lots of bellows to accommodate the chamber's expansion and this can lead to a higher machine impedance. Also, the heating tapes wrapped around the chambers to heat them up must be very thin for those chambers inside the multipole and dipole magnets. For this reason, we have developed a customizable thin polyimide heating tape in conjunction with Brazilian company EXA-M Instrumentação do Nordeste, which is one of the first successful examples of LNLS's partnership with Brazilian companies to build Sirius.

Fine thread The Sirius vacuum chamber, inside which electrons will circulate while emitting synchrotron radiation, has to weave its way through a highly compact arrangement of magnets and other components (grey) and needs complex chamber shapes and more than 450 individual sections.

row vacuum chambers, which are harder to pump. Another challenge is that the vacuum chambers and components must provide a continuous electrical path to minimize the impedance of the machine, which can affect beam stability by introducing electromagnetic wake fields. The most common materials used in third-generation light sources are stainless steel and aluminium, but we have chosen to build the Sirius vacuum chamber from oxygen-free silver bearing (OFS) copper. The higher electrical conductivity of this material minimizes the machine's impedance, while its improved thermal conductivity makes it better at absorbing unused synchrotron radiation. OFS copper also has a higher annealing temperature, which is convenient for NEG coatings because vacuum chambers need to be heated to a temperature of at least 200°C to activate the coating. Most of the Sirius storage ring vacuum chambers will have a circular cross section with an inner diameter of 24 mm and a wall thickness of 1 mm.

Since the chambers also have to absorb unused synchrotron radiation, narrow copper cooling pipes will be attached to their outer side. But the machine's compact lattice means that other sections of the storage ring – especially where the X-rays are siphoned off and sent down various beamlines towards experimental targets – demand more complex chambers. This makes the NEG coating extremely challenging. Sirius will have around 450 chambers in total, and even the circular cross-section chambers inside the multipole and bending magnets (where there is no need to extract radiation for the beamlines) do not have a simple manufacturing process.

Three joining processes will be needed to manufacture every single chamber to ensure that the copper does not become annealed and distorted. Vacuum brazing

will be used to join short copper adapters to the stainless-steel vacuum flanges, while tungsten inert gas (TIG) welding will be used to weld these components to the copper vacuum chambers. A robotized TIG welding station gives us precise control and is suitable for complex geometries, but neither TIG welding nor vacuum brazing are suitable for attaching the copper cooling pipes to the vacuum chambers because of the potential for distortion or annealing, respectively. Here, vacuum soldering at a temperature of about 330°C is being developed to produce joints for thermal contact.

NEG coatings

Since all the Sirius vacuum chambers will have small vacuum conductance, on account of their narrow cross section, NEG coatings are paramount. The host lab of Sirius, the Brazilian Light Source Laboratory (LNLS), has signed a licence agreement with CERN to use the coating technology and also has its own NEG coating facility to produce the Sirius vacuum chambers. Built last year by the LNLS engineering teams at a cost of around R\$300 000 (about £80 000), this is one of the few places in the world where NEG coatings can be produced according to standards determined by CERN – offering the ability to coat vacuum chambers up to 3.2 m long and 450 mm in diameter (see image on page 9).

To ensure that the NEG coatings have good adhesion and pumping properties, the surfaces of the vacuum chambers must be completely free of contaminants. We therefore developed a special cleaning process based on a recirculation system where only the inner surfaces of the vacuum chambers are exposed to the etching solutions, which also reduces workers' exposure to harmful substances. Although NEG coatings for the simple circular vacuum chambers are

Global ambition

Further Brazilian firms are being encouraged to take part in Sirius, and collaborations between LNLS and other laboratories are in progress. One involves CERN, where there is a mutual interest between our vacuum teams in studying the behaviour of surfaces exposed to synchrotron radiation. These studies will allow a better understanding of the Sirius vacuum chambers as well as an opportunity for CERN to better understand the proposed new surface technologies for the LHC chambers.

Sirius will soon be one of the world's brightest synchrotrons, opening new frontiers for research across materials science and also serving as a stepping stone to a diffraction-limited storage ring – a so-called ultimate synchrotron. The design is pushing synchrotron technology to the limit, especially concerning the different and unique concepts proposed for the vacuum system. All of the vacuum chambers and components must be designed and manufactured according to tight requirements, not just in terms of vacuum-system specifications but also to maximize the scientific performance of Sirius.

There are still many challenges to overcome with the Sirius vacuum system, such as the manufacture and NEG-coating of the complex-shape chambers. But once operational, Sirius will provide research from Brazil and the rest of Latin America with the opportunity to develop cutting-edge science in many fields and put nations from this region on the scientific map.

Rafael Molena Seraphim is head of the vacuum group at the Laboratório Nacional de Luz Síncrotron (LNLS) and at Centro Nacional de Pesquisa em Energia e Materiais (CNPEM) in São Paulo, Brazil, e-mail rafael.seraphim@lnls.br

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Refraction redefines the pascal

Based on highly accurate optical interferometry and fundamental quantum calculations, researchers in the US are developing an improved definition of the SI unit for pressure that will consign the mercury manometer to history, explain **Jay Hendricks, Jacob Ricker, Patrick Egan and Gregory Strouse**

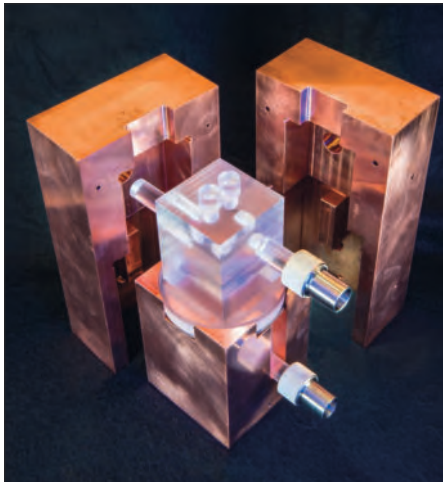
Many of us are unaware of the extent to which the international system of units (SI) touches our everyday lives, and this is especially true for pressure. Highly accurate, traceable measurements of pressure and vacuum conditions are vital in many manufacturing processes including that of semiconductor chips, where precise pressure measurements can improve control over features at the nanometre level. The routing of air traffic is another critical application: the minimum separation between aircraft as they approach busy airspace currently is about 600 m, but better pressure measurements could allow this distance to be cut in half – thereby reducing congestion and saving fuel.

Although pressure is one of the most widely measured units in everyday processes – and is key to vacuum science and technology – the standards that underpin it are very old. The SI unit for pressure, the pascal, is realized by mercury manometers that date back more than 300 years and we have now reached a point where the accuracy of such standards cannot be improved further. The same is true of temperature, the most widely measured unit, where the fundamental SI unit – the kelvin – is defined by the triple point of water.

A five-year project at the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland, US, aims to fundamentally change the way that the pascal is realized and disseminated. Based on ultra-precise measurements of the refractive index of gases using optical interferometry, it will raise pressure standards to the level of other SI units based on fundamental constants and also offer a brand new way to redefine the kelvin.

Mercury falling

The story of barometric pressure and early vacuum measurements is the story of mercury manometers, which were invented in 1643 by Italian physicist and mathematician Evangelista Torricelli. Some of the earliest barometric measurements were made by taking liquid in a glass–mercury manometer up a mountain and periodically observing the height of the mercury column along the way: the higher the altitude, the lower the mercury column height, indicating a



Raising standards A fixed-length optical cavity (FLOC) being developed at the National Institute of Standards and Technology, showing its copper temperature shield and a 15 cm dual-cavity prototype (one cavity under vacuum and one in nitrogen gas). The FLOC is a portable version of the primary optical standard based on a variable-length optical cavity.

lower barometric pressure. Named in Torricelli's honour, the torr (equal to 133.322 Pa) is nominally equal to 1 mm of mercury-column height and the unit is still in common use today on many vacuum gauges.

Since Torricelli's day, improvements in the mercury manometer have been incremental, based on resolving column heights with greater precision and accuracy. The lowest pressure uncertainties today come from a device called the ultrasonic interferometer manometer developed at NIST in 1975, which uses pulsed ultrasound to determine column heights in a 3-m-tall mercury liquid-column manometer to within 10 nm. But we have now reached the limit of such improvements. Furthermore, mercury's status as a neurotoxin and environmental hazard has led to a ban on the purchase of mercury products. Government laboratories, national laboratories and corporations have therefore been forced to get rid of their

We have reached the limits of improvements based on mercury manometers

mercury manometers, and as a result have seen their pressure-measurement capabilities downgraded. However, NIST still maintains its three mercury manometers as the national standard for the US – and will continue to do so until a high-accuracy, mercury-free alternative has been developed.

Since reference manometers are not portable (the NIST device is 3 m high and contains 250 kg of mercury), a parallel goal is to build a pressure standard that can deliver pressure measurements directly to the community. Several years ago, NIST researchers took an intermediate step towards this goal by developing a mercury-free “transfer standard package” (TSP) that enables high-quality measurements of NIST's mercury manometers to be transferred to industry, academia and national standards laboratories around the world. This device allows reliable pressure measurements ranging from high vacuum (10^{-2} Pa) to atmospheric conditions (10^5 Pa), where improper use or incorrect calibration of gauges can cost time and money. But even though it provides a bloodline that links vacuum gauges directly to the SI system, the TSP still requires NIST to maintain and operate a 3-m-high mercury manometer. Our new project aims to replace not only the mercury manometer, but also the TSP, and do it at higher accuracy with an even more compact device.

Pressure by refractive index

In search of a better, mercury-free way to realize pressure – and one based on fundamental physics rather than a physical object – NIST has embarked on an entirely new optical pressure standard that links the pascal to quantum calculations of helium's refractive index. Pressure and vacuum standards based on refractive index will significantly cut measurement uncertainties and improve accuracy in the aerospace, energy and advanced manufacturing sectors by between three- and 10-fold.

The refractive index of a gas depends on its density, which is a function of temperature and pressure. Quantum mechanics tells us the exact relationship between these variables, and calculations for atomic helium will allow us to relate pressure, temperature and refractive index with an accuracy significantly better than one part in

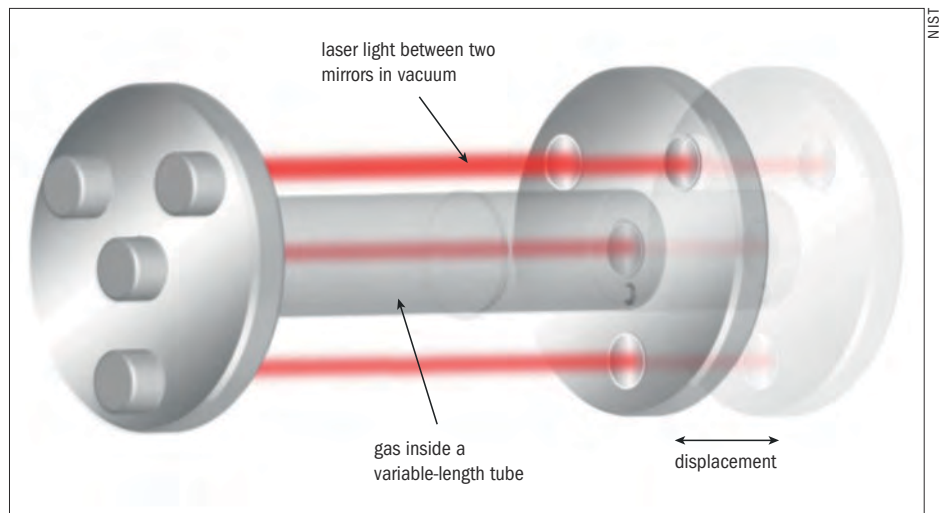
Standards

10^6 . By measuring temperature and refractive index to high accuracy, we can determine the pressure and turn the device into a primary pressure standard based on the atomic properties of helium. Conversely, if pressure can be measured accurately using alternative means, then thermodynamic temperature can be determined by measuring the refractive index.

The key to making this approach worthwhile is to measure refractive index with much higher accuracy than has previously been achievable, which is possible using laser interferometry. When measuring a length using laser interferometry, the presence of a gas such as helium causes the distance to look a little bit longer than would be observed in vacuum. If we were able to compare two identical lengths in helium and in vacuum, the apparent difference would therefore allow us to determine the refractive index. The challenge facing NIST is that this difference must be measured with an accuracy of a few picometres – about 1% of a typical atomic diameter. Although picometre precision is relatively straightforward using a Fabry–Pérot interferometer, in which light bounces back and forth many times in an optical cavity, we face the challenge of generating identical displacements in two optical cavities (one in vacuum and one in helium) both with picometre accuracy.

To this end, NIST is developing a variable-length optical cavity (VLOC) comprising four individual cavities: a central cavity in helium gas surrounded by three cavities in vacuum (see figure). The mirrors at each end of the apparatus are built on highly stable bases such that the displacement of one mirrored end-piece generates equal displacements in both the outer and inner interferometers. The reason for having three interferometers in vacuum is that it allows measurement and control of angular tilts as the end-piece is translated, which is necessary to avoid Abbe errors.

Building such a primary pressure standard at NIST is all well and good, but we also have to find a way to transfer its superior accuracy to locations outside the standards lab. We are therefore also developing a less complex fixed-length optical cavity (FLOC), which will offer enhanced sensitivity over the current TSP in a greatly simplified and more portable package. The transportable FLOC standard, a prototype of which is currently being built, will use nitrogen as a working medium because it has a higher refractivity than helium and is 100 times less sensitive to contaminants. Fixed-length cavities are attractive as pressure standards because they can be made from materials with small temporal instabilities, and will deliver an immedi-



Picometre precision Conceptual picture of the VLOC apparatus: a displacement of the end-piece on the right changes the lengths of the four optical cavities by equal amounts and the difference between the four cavities represents a measure of the refractivity of helium. Knowing the temperature, a new primary pressure standard can therefore be realized.

ate improvement by a factor of three in pressure-measurement uncertainties over existing commercial technologies for vacuum pressures down to 1 Pa. NIST plans to employ the FLOC and VLOC as optical pressure and vacuum standards for pressures up to 360 kPa, which will allow us to replace all mercury-based pressure standards within the next 5–10 years.

Branching out

The five-year-long project is currently in its second year. The design phase has been completed, custom parts are currently being fabricated and NIST plans to have a fully working prototype by the summer of 2015. Over the next three years, we will complete the VLOC and refine the quantum-mechanical calculations of helium's refractive index. This will also allow us to measure the refractive index of nitrogen to a level sufficient to make the FLOC a standalone pressure and vacuum standard. Finally, the optical standard will be compared with the existing NIST mercury manometers to close the final chapter on Torricelli's mercury pressure standard.

Since the new optical pressure standard does not require the long central column that mercury manometers do, there is essentially no limit to how small the device can get. While the first generation of FLOCs will be 15 cm in length (which is already 20 times smaller than NIST's mercury manometer), future versions may become still smaller and more compact to deliver better pressure measurements directly to the pressure and vacuum community. Even more exciting is that the unit for pressure will be quantum-based. Manometers have a direct link back to the SI through high-accuracy measurements of elemental

mercury's density that is traceable to the kilogram. However, the new standard will no longer be linked to the kilogram, which is an artefact-based standard, but based on the refractive index of helium and on fundamental constants that are the same everywhere in the universe.

The same technology can be used to determine thermodynamic temperature with uncertainties below what has been previously achieved, providing a definition of the kelvin that is independent of water's triple point. The NIST project also applies to dimensional metrology using laser interferometry, for which accuracy can be limited by uncertainty in the refractive index of air. With the FLOC cavity being open to the environment, it provides *in situ* measurements of the local refractive index and therefore allows real-time wavelength corrections to be made with a projected uncertainty at the level of three parts in 10^9 when measuring large distances, say on a factory floor.

NIST's new mercury-free optical pressure standard will benefit science, industry and defence applications, as well as secondary-measurement services. Indeed, the FLOC and VLOC devices will ultimately eliminate mercury from all NIST primary pressure standards and at other labs and industrial facilities. We expect the optical pressure standard to become a commercial device used across academia and industry at a fraction of the cost of a commercial pressure standard, while being cheaper to maintain and having a larger pressure range and lower uncertainty.

Jay Hendricks, Jacob Ricker, Patrick Egan and Gregory Strouse are at the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland, US, e-mail jay.hendricks@nist.gov

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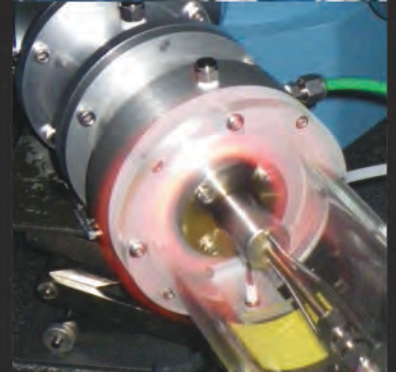
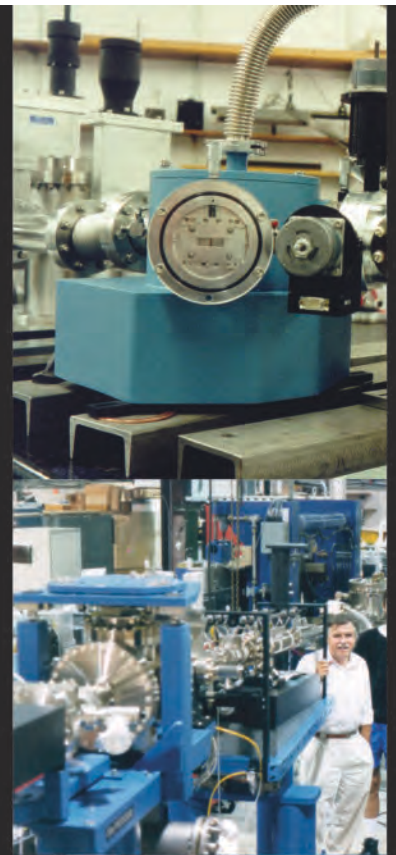
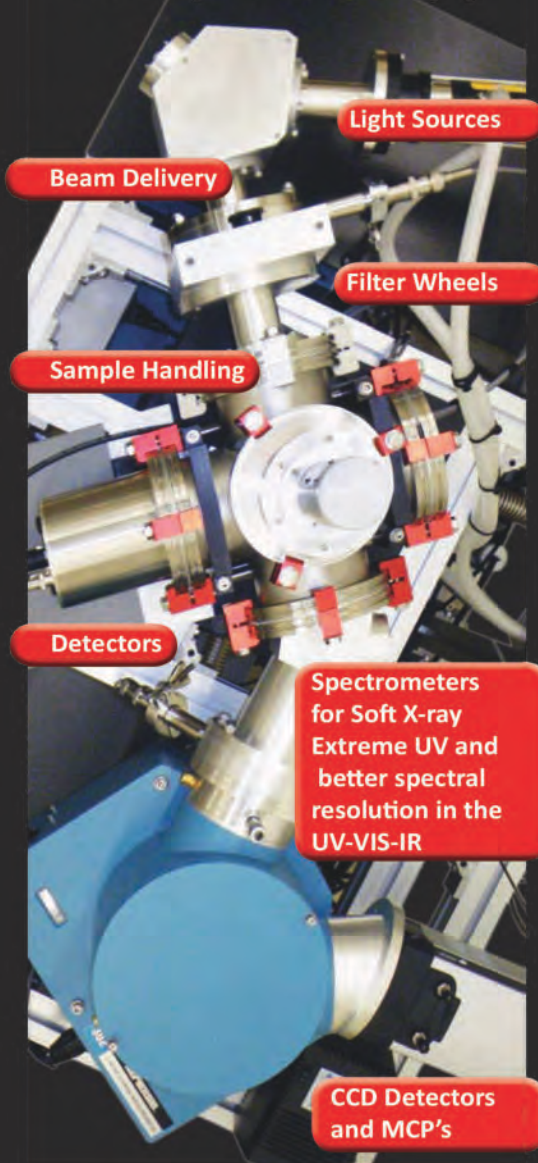
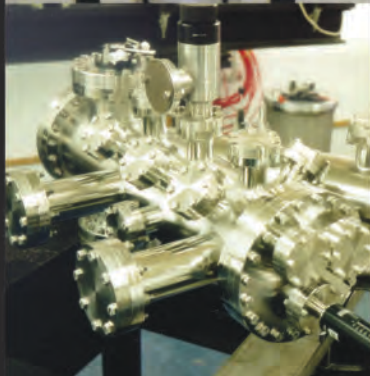
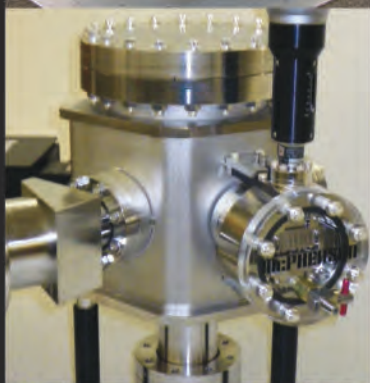
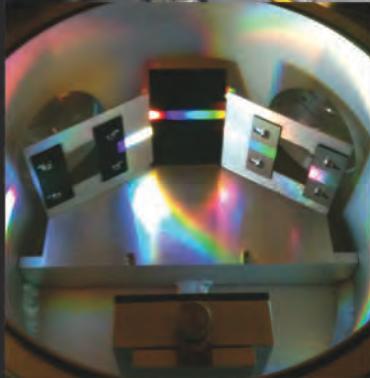
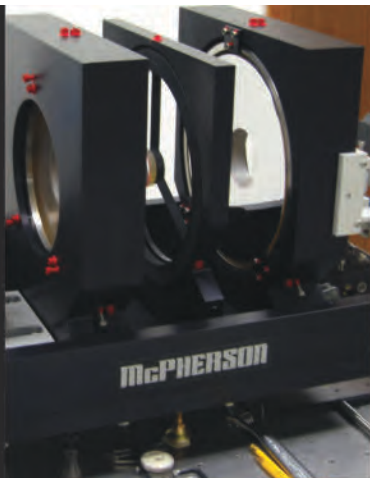
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Firms set new pump standards

The challenge for today's vacuum-pump designers lies in better energy efficiency, reduced noise and the use of new materials, says **Romina Gotta**

Vacuum is a key enabler for many industrial processes and scientific enquiries, ranging from chemical vapour deposition in the semiconductor industry to multibillion-dollar colliders that explore the fundamental constituents of the universe. At its heart are pumps that remove unwanted molecules from a chamber. While the first vacuum pumps were invented more than 400 years ago, it has only been since the 1950s that manufacturers have been producing pumps on an industrial scale.

Today, the vacuum industry is led by four major companies – Agilent, Pfeiffer, Edwards and Oerlikon Leybold – plus



Pumping profits Agilent's TwisTorr 304 FS removes gas from a chamber through a combination of turbine and molecular-drag technology.

several dozen other firms specializing in certain products or applications, with a combined global annual turnover of about \$4.5bn. When it bought Varian in 2010, Agilent acquired almost 60 years of pump design and manufacturing experience, immediately making it a key player in academia and large research centres.

Many vacuum-based processes require low pressures in the range known as high-vacuum (10^{-9} – 10^{-4} mbar) or even lower in the ultra-high vacuum (UHV) range. In order to achieve such low pressures, it is necessary to use two or more pumping technologies. When starting from atmospheric pressure, it is possible to efficiently displace much of the gas by comparatively simple mechanical means because the gas molecules under these conditions are plentiful and constantly colliding with one another. But high-vacuum pump technologies, such as turbomolecular pumps, by contrast, are optimized for pumping a smaller number of molecules



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Pumping

and require a low starting pressure. High-vacuum pumps would be choked in the primary vacuum range, and primary pumps cease to work at lower pressures.

Fundamental limit

The principal performance specifications are pumping speed and base pressure, which is the lowest pressure that a pump can normally produce and maintain. Cast in these terms, pump technology is now approaching its theoretical limit. Yet how well a pump actually performs depends on other factors such as the size and cleanliness of the vacuum chamber, the rate of pressure cycling and any additional gas-load introduced by the system.

The major focus of leading vacuum-pump manufacturers has now turned to managing, optimizing, reducing or even eliminating the impact that vacuum products have on the system in which they operate. For example, pump designers seek to reduce energy consumption, noise and vibration levels, space occupied, and the frequency or difficulty of maintenance – all while continuing to meet or exceed the pumping performance requirements of the application.

Examples of this design approach include Agilent's TwisTorr 304 FS turbomolecular

high-vacuum pump and its IDP-15 dry-scroll primary pump, which represent recent advances in pump design. Like most turbomolecular pumps produced today, the TwisTorr 304 FS removes gas from a chamber through a combination of turbine and molecular-drag technology. The patented technique dramatically improves the efficiency of pumping light gases such as hydrogen and helium, making it possible to reduce the size of the rotor and the energy required to maintain high-speed rotation.

Change of focus

Along with other leading pump manufacturers, Agilent is responding to a clear market need for turbomolecular pumps with higher compression profiles that permit the use of smaller, quieter and less-expensive primary pumps. Audible noise is increasingly an issue for vacuum users who use scroll pumps in a laboratory environment. Instrument companies have historically used sound-insulating boxes or cabinets to reduce the effect, but these are expensive, bulky and need to be designed carefully to be sure enough cooling air is delivered to the pump. There is also strong demand for quiet medium-vacuum pumps in the academic and industrial markets, since

researchers often need to work in close proximity to their apparatus and industry tends to employ a large number of vacuum pumps in a confined manufacturing space.

Agilent engineers isolated sources of noise by enclosing areas of the test pumps with sound-deadening material and comparing measurements with those of the test pump as a whole. Using spectrum analysis, we were able to identify the nature of several noise sources – including those from exhaust pulsation, mechanical movement and the required cooling fan – each of which was incorporated in the product design.

Although the idealized concept of full-range vacuum pumps remains out of reach, it has been approximated by systems that integrate primary and high-vacuum pump technologies in a single package that also includes vacuum gauges, control software and user-friendly interfaces. The challenge for today's vacuum-pump designers is to become expert in energy management, noise and vibration, use of new materials, tribology, control firm- and software, and many other disciplines. This will lead to new products that will make vacuum even more invisible to its end users.

Romina Gotta is in Agilent's Vacuum Product Division in Turin, Italy, e-mail romina.gotta@agilent.com

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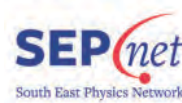


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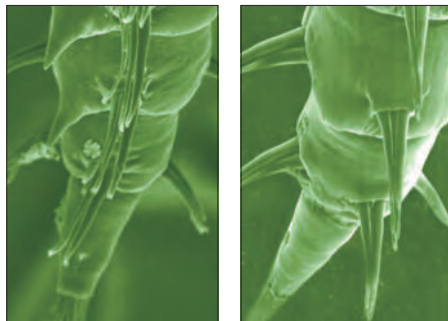
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Coated creatures survive SEM

Artificial polymer cases that keep creatures alive in the high-vacuum environment of an electron microscope are allowing biologists to observe creatures in their native states, writes **Matthew Chalmers**



Hamamatsu University School of Medicine

Up close SEM images of midge larvae prepared by traditional chemical fixing (left) and when protected by a nanosuit (right) reveal morphological differences, but the nanosuit appears to preserve the natural surface structure of living organisms.

Life on Earth can withstand an impressive range of conditions, from huge pressures in the deep ocean to extreme temperatures in volcanic vents. Put a creature in a vacuum, however, and things soon turn ugly. Water begins to boil off surfaces and internal gases rapidly expand, culminating in a loss of consciousness and death. For biologists, this severely limits the ability of electron microscopy to study living organisms.

The scanning electron microscope (SEM), which uses the short wavelength of

electrons to achieve magnifications as high as 10 million, has become an essential tool for observing biological materials at the microscopic level. But in order to prevent electrons scattering off molecules in the air, the sample chamber of an SEM must be

evacuated. Soft-bodied organisms therefore first have to be chemically fixed, dried and coated with an ultrathin layer of electrical conductor to prevent charge build up.

In 2012 researchers in Japan reported a way to get round such obstacles. Takahiko Hariyama of Hamamatsu University School of Medicine and colleagues found that bombardment by electrons in an SEM can equip certain multicellular organisms with a thin polymer membrane – dubbed a “nanosuit” – that keeps creatures alive for extended periods (*PNAS* 110 7631). Now the team has published the results of experiments with synthetic nanosuits and has also registered the NanoSuit trademark. “Honestly, I cannot explain why animals can survive such an extreme environment,” says Hariyama. “But we know that mosquito larvae that we have observed at high SEM magnification developed to adult mosquitos normally.”

It was a frustration with the lengthy process of preparing biological samples 40 years ago that led Hariyama to want to put

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Microscopy

live animals in an SEM. He says he wanted to see the real structure of a creature's surface, free from preparation artifacts, but was forbidden to do so by his supervisor in case he damaged the equipment. While recounting this story to his own students four years ago, Hariyama was encouraged to give the experiment another go.

The team placed numerous different species into an SEM chamber to see how long each would survive. Almost all suffered massive structural collapse and died rapidly, but to the team's surprise the larvae of fruit flies continued to move around for one hour inside the SEM and subsequently developed normally. When control larvae were placed under the same vacuum conditions but without electron-beam irradiation, by contrast, they were destroyed. Transmission electron microscopy revealed that an extra polymer layer 50–100 nm thick had formed on the irradiated larvae surfaces, suggesting that electron bombardment enhances the cross-linking of extracellular substances on fruit flies to provide a protective case that prevents the passage of gases and liquids.

To test this hypothesis, the team tried to mimic the extracellular substances using an amphiphilic surfactant called Tween 20, which polymerizes when irradiated by a

plasma or electron beam. Having dipped several species including flatworms and ants into dilute Tween 20 solution before they were imaged, the researchers found that all survived in the SEM. The team then irradiated animals with a plasma before putting them in the SEM and found that they survived in the high-vacuum environment without electron-beam irradiation. "This was the final breakthrough that allowed us to realize the existence of the nanosuit," says Hariyama.

Seeing is believing

The team has since tested its synthetic prefabricated nanosuits on a range of animals, including midge and mosquito larvae, and found that the creatures survive long enough to allow observations of movement without ill-effects – even at high magnifications (*Microscopy* 10.1093/jmicro/dfu015). Other groups have taken up the discovery, but some experts say that we can already image unprepared biological samples under low-vacuum conditions or by using a device called an environmental SEM (ESEM).

Developed in the 1980s, ESEMs use water vapour to create a tropical environment around a sample, allowing it to be imaged in its natural hydrated state. Around 10

years ago researchers at Carl Zeiss Microscopy used an ESEM to observe live cheese mites for up to three minutes. "It was a low-resolution study and the creatures eventually died from beam damage, but such observations don't need very high magnifications," says Veronika Kugler of Carl Zeiss Microscopy in the UK. "The nanosuit idea is a very interesting one, but it involves modifying the surface of a sample and it is therefore not clear exactly what is being observed and perhaps requires further investigation."

Hariyama is still trying to make sense of the new findings himself. "When we observe biological samples treated by traditional methods, we see some debris caused by electron-beam irradiation, yet the live animals coated by nanosuits don't show those artifacts," he says, adding that there are several R&D stages ahead to investigate whole living creatures and to develop appropriate engineering materials.

The nanosuit might even shed light on the origin of life, he adds, since it potentially allows simple life forms to travel through space. "The existence of nanosuits, and the fact that energetic electrons are known to occur commonly throughout the universe, offers a fresh view on the conditions under which life can survive," says Hariyama.

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Atlas Copco bets on vacuum

Following Atlas Copco's \$1.5bn takeover of Edwards Vacuum, the president of the firm's newly created vacuum-solutions division, **Geert Follens**, explains why he expects strong growth in the vacuum market

What is Atlas Copco's track record in the vacuum sector?

In 2009 Atlas Copco acquired Quincy Compressors, which gave us a range of oil-injected screw vacuum pumps. Then in April 2012 we added to this by launching a range of oil-injected vanes. These products were aimed at the rough-vacuum market, where the vanes are typical point-of-use products and the screws are used in centralized vacuum installations. The business was small, but showed that we saw vacuum as a natural extension of our compressor business.

Why did Atlas Copco buy Edwards?

Atlas Copco's core compressor business, which provides industrial air and gas compressors and management systems,



Atlas Copco

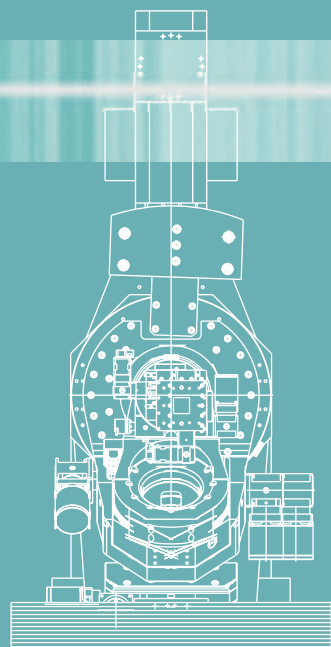
In the hot seat Geert Follens, who trained as an electrical engineer, joined Atlas Copco in 1995 as a purchasing manager and was made president of the firm's newly created vacuum solutions division last year after the acquisition of Edwards Vacuum. Atlas Copco now has around 40 000 staff in 170 countries.

has grown considerably in the past 10 years. But our high market share also means we cannot grow much further through acquisitions in this area, so we have been looking at adjacent parts of the business. Not only is vacuum one of the most attractive, with a market worth more than \$6bn and an expected growth rate of about 5% per annum, but its customer base and technology are similar to those in compressors. So, we decided to acquire Edwards, which is the number one technology leader in high vacuum.

How will Atlas Copco and Edwards benefit one another?

First and foremost, we will exchange technology. For example, we may be able to accelerate the development of some

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Interview: Geert Follens

of Edwards' high-end pumps by sharing technology from Atlas Copco, while learning from Edwards to help develop compressor technology. The technology for vacuum and compressors is closely related, so from an R&D point of view we can certainly complement each other. Another opportunity is cross-selling: Edwards has customers to whom we are not delivering compressors, and Atlas Copco has customers to whom Edwards does not deliver vacuum. In the rough-vacuum area, where air is used as a utility, Edwards does not really have a presence, so that's where we want to come in under the Atlas Copco brand.

What is the overlap between compressor and vacuum customers?

Quite big. For example, around 60% of customers in Atlas Copco's industrial air division, which I headed until recently, also use rough-vacuum products. A typical customer in that division is a small- to medium-sized manufacturing company, but many aren't really conscious that they rely on vacuum technology because very many rough-vacuum products are sold through original equipment manufacturers (OEMs). An example is printing-machine manufacturer Heidelberg, which uses a huge amount of vacuum in its machines. We also share customers in the semiconductor business, but Edwards has a much higher share there.

What has the merger process been like?

It's been a very positive experience, and Edwards people have been very professional and well prepared. It was different for Atlas Copco because most of the acquisitions we have made before were privately owned companies, whereas Edwards was owned by private equity and listed on the NASDAQ stock market. The business culture is very similar to Atlas Copco's:

we are both product driven and customer focused, and we both focus on the total cost of ownership and on the service business. But the operating model is very different because Edwards has a centralized set-up whereas Atlas Copco is more decentralized.

How do you see the vacuum market developing in the future?

We can divide the vacuum market roughly in three parts. First, there is the high-end vacuum process used in making semiconductors, microchips and flat panels. The global use of these hi-tech products will surely continue to increase, while production methods are becoming more effective with bigger wafers and bigger machines, which means more vacuum technology will be required. A second part of the vacuum business is what we call general vacuum, where vacuum is used as a utility – for example to move packages. This area is growing at the rate of the broader industry, and the global coverage of Atlas Copco means that we potentially can outperform the market. The third part concerns service.

What is vacuum's growth potential in different regions?

For high-end process vacuum, the growth looks to be mostly in Asia and North America. Increasing numbers of semiconductor factories are being built in China. At the same time, the American market is becoming more interesting for manufacturers, and several Asian firms are investigating whether they can build factories in the US. In Europe, you don't see huge activity in semiconductors, although the European Union is now taking some initiatives, but when it comes to general and rough vacuum, Europe has equal growth potential to that of Asia and North America.

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CLOUD aids climate predictions

The cleanest chamber ever built is allowing researchers at CERN to track the microscopic processes governing cloud formation, finds **Matthew Chalmers**

In its fifth assessment report published last year, the Intergovernmental Panel on Climate Change identified atmospheric aerosols and their influence on clouds as the largest source of uncertainty in current climate models. Hundreds of these tiny particles – ranging from a few nanometres to a few micrometres across – are present in every cubic centimetre of atmosphere, with higher concentrations in urban environments. In addition to cooling the climate by reflecting or absorbing solar radiation, atmospheric aerosols also provide the seeds for all cloud droplets. Some cloud seeds



Clean climate The CLOUD experiment is sensitive to contaminants at the level of parts per trillion.

enter the atmosphere directly from dust or sea spray, but around half are produced indirectly by the nucleation of trace vapours to form tiny molecular clusters that grow

under condensation.

This process is poorly understood, but an experiment called CLOUD at CERN, the particle-physics laboratory in Geneva, is now providing scientists with some unique perspectives on it. At its heart is a electropolished stainless-steel cylinder – with a volume of roughly 26 m³ – in which researchers recreate a portion of the atmosphere under precisely controlled conditions. Earlier this year, the 50-strong CLOUD collaboration reported its latest results, revealing that biogenic vapours emitted by trees have a significant impact on the formation of aerosols and clouds that helps cool the planet.

“The reason why it has taken so long to understand the vapours responsible for new particle formation is that they are present in minute amounts near one molecule per trillion air molecules,” explains CLOUD spokesperson Jasper Kirkby of CERN. “Reaching this level of cleanliness and control is at the limit of current technology, and CERN’s expertise in materials, gas systems

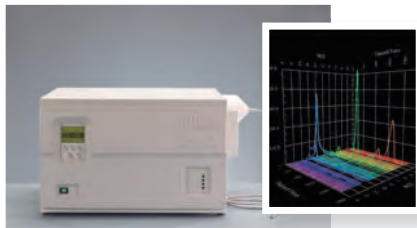
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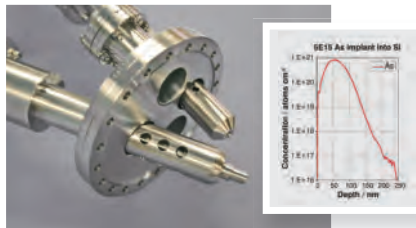
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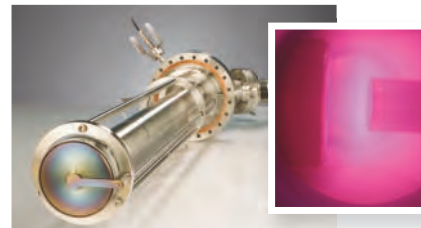
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Meteorology

and ultra-high-vacuum technologies has been crucial,” he says.

Cosmic origins

CLOUD, which stands for Cosmics Leaving Outdoor Droplets, is designed to explore the link between galactic cosmic rays and cloud formation. First proposed in the 1970s, the idea is that charged particles that strike the Earth’s atmosphere produce ions that help new aerosol particles nucleate. To mimic such processes the CLOUD chamber is bombarded by beams of charged pions from CERN’s Proton Synchrotron.

A bespoke gas system and strict use of clean materials in the chamber lets the CLOUD researchers get much lower concentrations of contaminants than in previous experiments. Indeed, the team even produces its own synthetic air from cryogenic liquid oxygen and nitrogen, since natural air cannot be purified sufficiently. The temperature of the chamber can be varied between +30 °C and –70 °C and kept steady to within 0.01 °C, allowing researchers to reproduce any region of the troposphere. In order to simulate sunlight, which is necessary for photolytic reactions, the chamber is fitted with ultraviolet lamps linked to a quartz fibre-optic system and 250 optical-

fibre vacuum feedthroughs.

In 2011 CLOUD reported its first results, revealing that trace sulphuric-acid and ammonia vapours at high altitudes can rapidly cause clusters to nucleate and become the seeds for clouds. The results also showed that ionization from cosmic rays can make nucleation occur up to 10 times faster. On the other hand, the data showed that these vapours could only account for 1000th of the rate of actual aerosol formation observed at low altitudes (*Nature* **476** 429).

Two years later, the collaboration showed that amines – derivatives of ammonia – in concentrations of a few parts per trillion can stabilize sulphuric-acid particles and better reproduce the observed particle formation rates (*Nature* **502** 359). Yet unlike sulphuric acid, which enters the atmosphere mainly through coal-fired power plants, amines are only found close to primary sources such as farms. This suggested that additional vapours must be at work.

Biogenic source

CLOUD’s latest results show that sulphuric-acid aerosols do indeed have a significant influence on the formation of clouds, but only if they are stabilized by

volatile biogenic vapours emitted by trees (*Science* **344** 717). These vapours, which give pine forests their characteristic smell, are rapidly oxidized in the atmosphere to produce low-volatility vapours that participate in the formation of new aerosol particles. When the researchers modelled the global impact of the new process, they were able to account for the observed seasonal variations in atmospheric aerosol concentrations for the first time.

According to Ian Ford, a condensed-matter theorist at University College London who is not involved in the experiment, the result represents a significant step forward in climate science. “CLOUD is one of the best experimental rigs in the world and the wide range of atmospheric conditions that it covers means that we can now better parameterize the impact of aerosols and clouds in global climate models,” he says.

The CLOUD team is now studying how nucleation rates vary under different conditions, vapour concentrations and particle-beam intensities, and the experiment will run for 10 more years. “We are confident that it will settle the link between cosmic rays and cloud formation, while also reducing uncertainties in anthropogenic aerosol-cloud changes,” says Kirkby.

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Vacuum measures up for industry

A European project led by national metrology institutes aims to develop better, faster and cheaper ways to measure vacuum pressure and trace leaks under industrial processing conditions, explain **Karl Jousten, Mercedes Bergoglio and Janez Setina**

For more than a century, vacuum technology has become increasingly vital to industry. Light bulbs were the first product to require good-quality vacuum, followed by radio tubes, X-ray tubes, television tubes and now the semiconductor industry. Computer chips, flat-panel displays, mobile phones and solar cells all rely on production steps carried out in a vacuum environment, with the semiconductor industry alone making up about 40% of the vacuum market.

Modern vacuum systems not only have to be robust and easy to operate, they also have to meet tough demands in terms of performance. Vacuum technology has matured a lot over the last 50 years, but there are still important challenges to overcome for industry applications. The “cleanliness” of a vacuum – or more technically the absence or sufficiently low partial pressure of specific gas species or vapours – is vital. Although oil-free vacuum pumps and other components have improved cleanliness considerably, components can still be a source of unwanted gases that get released or “outgassed” into the vacuum. Outgassing rates are therefore a vital quality-assurance figure in vacuum technology, but there is currently no established way to measure such rates and the results are not traceable to the SI system of units.

Leak detection is also crucial to ensure the safety of vacuum systems. Anyone who has a pacemaker installed in their body can be sure that the device has the lowest leak rate of any industrial product, while vacuum leak detection also stops refrigerants in air-conditioning systems from escaping. Industry demands faster and cheaper measurement methods in this and other areas, including the coating industry. Plastic bottles are coated to prevent the escape of carbon dioxide, for instance, and optical discs have to be metallized. These fast-vacuum



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processes demand vacuum gauges to be correct during cycling times lasting a couple of seconds.

National metrology institutes have established primary standards with which to calibrate vacuum gauges under clean, stable and equilibrium conditions. But to keep vacuum technology in step with the needs of industry, we need to extend such capabilities to fast-changing pressures, partial pressures or “rough-vacuum” environments. In fact, this is the goal of a €2.7m European Union project called IND12 that has been funded by the European Metrology Research Programme and involves researchers from seven national metrological institutes and five industrial partners. Ending in August 2014, the three-year project has set new standards for vacuum metrology in production environments.

Project structure

The IND12 project was divided into three technical work packages to address the most important challenges in industry. In the first, led by researchers at the Physikalisch-Technische Bundesanstalt (PTB) in Germany, the consortium built a calibration system that allows pressure to be changed by three orders of magnitude – from 100 kPa to 100 Pa – either in steps over a period of 20 ms or more slowly, but more predictably, over an entire second. This fast pressure change, which simulates what

may happen in a load lock, was achieved by allowing a gas to expand from a volume of about 0.1 l into a large evacuated volume of 185 l via a very fast-opening gate valve and an exchangeable duct. Knowing the mass flow rate from numerical simulations of the Navier–Stokes equations, the team was able to calculate how pressure varies with time in small volumes and therefore provide a reference against which vacuum gauges can be tested.

To ensure that the valve’s opening time is negligible compared with the duration of the pressure change, valve manufacturers VAT Vacuum developed a very fast gate valve that can be opened within 4.6 ms. Without any additional duct between the small and large volume, specially developed capacitance diaphragm gauges from project partner INFICON showed a time response (corresponding to a $1/e$ decay in pressure) of 1.3 ms. This is the shortest time response that can be measured, and the new standard will offer industry traceable pressure measurements on the millisecond scale with uncertainties of less than 5%. The work has also allowed INFICON to launch a new series of fast gauges.

The second IND12 work package, coordinated by the Istituto Nazionale di Ricerca Metrologica (INRIM) in Italy, concerns leak detection – specifically improvements in our knowledge of ducts on the micrometre or sub-micrometre scale for gas-flow metrology. Nowadays, the most sensitive and versatile method for leak detection is to flush a system with helium and then measure how much of it leaks into the vacuum. In industrial applications, however, leaks usually happen under different pressures, temperatures, gas species and mixtures – and can even involve liquid flows. The pure helium flow rate therefore needs to be converted to describe that of a real system.

To trace leaks better under practical conditions, we created leak capillaries in the form of short and long tubes with known diameters and lengths (see image above) and then calibrated them using various gases against primary systems developed at national metrology institutes. By comparing measurements of capillary gas flow versus inlet pressure to numerical results, we found that relatively simple analytical equations can predict the flow rate for any gas other than helium. The resulting data can then be used by companies to estimate true leak rates of their products.

Another goal of this work package was to

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Metrology

guide industrial use of commercial helium leak detectors and infrared gas leak analysers for detecting refrigerants. Based on calibrated leaks, we were able to investigate the sensitivity, accuracy, repeatability, linearity and long-term stability of 10 leak detectors from different manufacturers. These findings will enable other firms to improve quality management by quantifying device uncertainties and reliability.

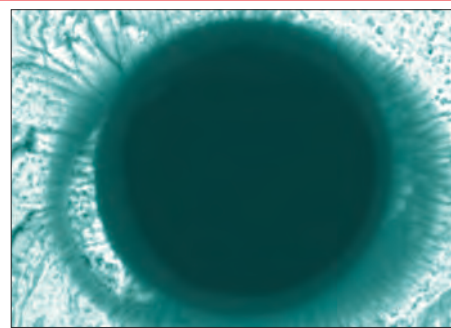
The third work package – led by the Slovenian national standards institute MIRS-IMT – sought to set up a dedicated calibration facility for quadrupole mass spectrometers (QMS). These instruments are not only used for leak detection and residual gas analysis, but also to provide quantitative analysis in processes such as etching or vapour deposition and to quantify outgassing rates. QMS devices are difficult to calibrate, however, because at present there is no traceability to any primary SI standard for partial pressures.

To get round this, the IND12 team therefore fabricated leak elements with a radius of 100 nm using focused ion beams, the flow through which can easily be predicted, and explored the impact of different QMS operational parameters on the sensitivity and linearity of the instrument and the fragmentation patterns of different gases. We concluded that changes to the instrument's electron emission current, electron energy and mass resolution can affect calibrated values of the instrument. Indeed, measurements of these parameters made every three months during a two-year period highlighted the poor stability of QMS instruments and suggests that *in situ* calibration is necessary to achieve accurate measurements.

Another focus of the third work package was to link measurements of outgassing rates, which are very small, to a metrological standard. Two kinds of reference outgassing samples were developed and tested: materials that absorb gases when exposed to high pressure and then release them when they are placed in outgassing measurement apparatus; and devices containing a reservoir for gases and a permeable element to control gas escape from the reservoir. The team investigated the permeation properties of reference materials Viton and PDMS for different gases by constructing a compact device that can be easily placed inside outgassing measurement apparatus, from which we were able to generate suitable outgassing reference samples for many gas mixtures.

Trouble-free vacuum

Key to the success of IND12 has been the close co-operation between industrial partners and national metrology institutes. It is now possible to not only calibrate total pressure vacuum gauges under ideal equi-



M. Bergoglio/IND12

Measured approach The study of leak elements with known geometries, such as this 18 μm -diameter hole drilled by a laser beam and then cleaned using focused ion beams, can help industry to calibrate vacuum-processing equipment.

librium conditions, but also under dynamic conditions on millisecond timescales. The project will also extend the impact of state-of-the-art helium leak detection because it allows the rate of a standard leak to be calculated for many other gas species used in industrial products, although further investigation is needed to determine the extent to which this conversion can be applied to unknown geometries in real situations.

The door has now been opened to more reliable partial-pressure measurements, since researchers can now calibrate partial-pressure analysers via national standards institutes. Although IND12 is a first and important step towards improving vacuum metrology for industry, there is still much to do. One example is to make calibration procedures available both at national standards institutes and *in situ*, which would offer control over the poor long-term stability and metrological characteristics of QMS devices – at least for important applications such as outgassing-rate measurements. To this end, IND12 project members work closely with the International Organization for Standardization to take the necessary steps towards standardization.

New methods for measuring partial pressures by optical methods have also been explored. These technique promise better reliability and faster measurement cycles, also for leak measurements and dynamic pressures, but their effectiveness will probably be limited to higher vacuum pressures above about 0.1 Pa. This has to be explored further in the future. IND12 and subsequent projects currently being planned will take us closer to a time when industry can get the most out of vacuum tools without having to think about them.

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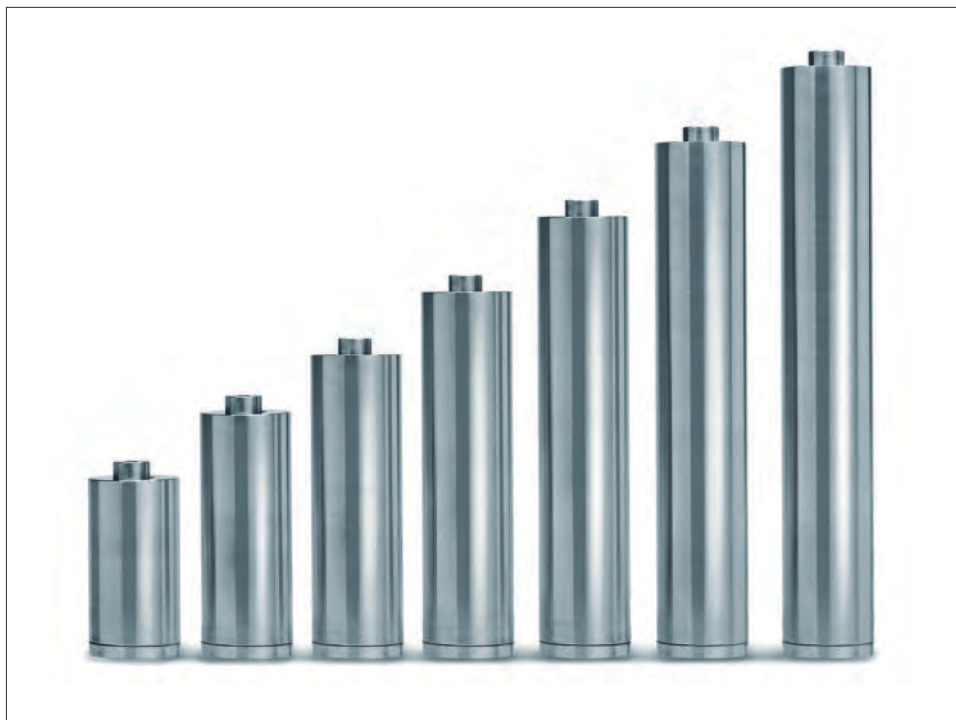
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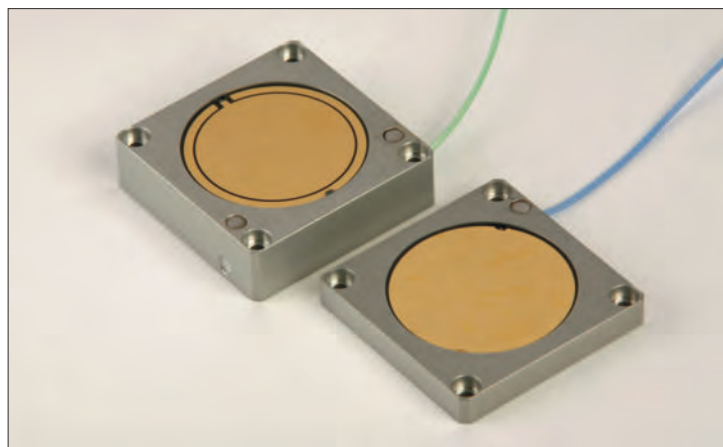
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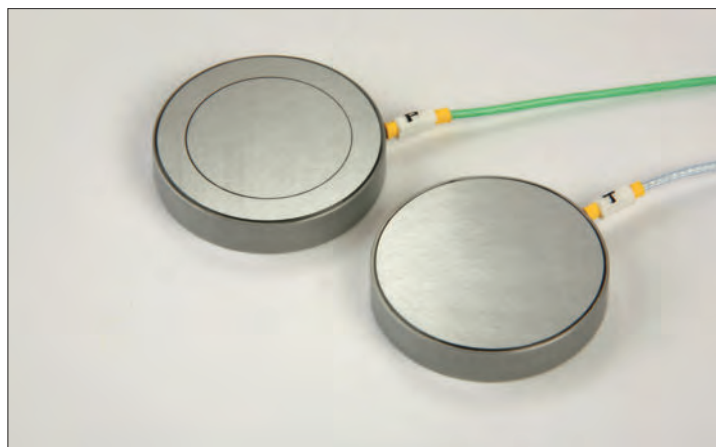
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Events Diary

Key meetings, conferences and exhibitions in the vacuum-technology calendar

2014

30th European Conference on Surface Science

Antalya, Turkey

31 August – 5 September

With 40 confirmed speakers and prizes for the best oral presentation, ECOS30 will cover the gamut surface-science topics including adsorption and desorption, bio-surfaces and interfaces, self-healing structures, surfaces in space and ultrafast phenomena.

www.ecoss2014.org

18th International Conference on Molecular Beam Epitaxy

Flagstaff, Arizona, US

7–12 September

MBE 2014 is an international forum for new developments in fundamental and applied molecular-beam-epitaxy research, including techniques, synthesis of new materials, discovery of new physical properties, formation of novel heterostructures, and the development of innovative devices.

<http://mbe2014.org/>

16th International Conference on Thin Films

Dubrovnik, Croatia

13–16 October

Including four plenary and 18 invited lectures, plus two sessions of poster contributions, the event, which is being co-ordinated by the Croatian, Hungarian and Slovenian vacuum societies, will cover recently established topics in thin-film devices and complementary topics in surface engineering.

<http://ictf16.com/index.php>

Vacuum Expo 2014

Coventry, UK

15–16 October

The UK's premier vacuum technologies exhibition and conference, now in its fifth year, brings together top suppliers and research groups to discuss industry needs, emerging technologies and challenges for vacuum technologies in science and industry. About 300 delegates attended last year's event.

www.vacuum-expo.com



13th European Vacuum Conference

Aveiro, Portugal

8–12 September

The biennial event will cover all aspects of vacuum science and its applications, with a special symposium on ultrathin oxide films and coatings. The 7th European Topical Conference on Hard Coatings and the 9th Iberian Vacuum Meeting will be held jointly.

www.evc13.com

Tec66

Hastings, UK

27 October

In its second year, this conference and exhibition focuses on vacuum and photonics developments relevant to industry and local hi-tech companies.

www.tec66.co.uk

AVS 61st International Symposium and Exhibition

Baltimore, Maryland, US

9–14 November

More than 3000 scientists and engineers, plus over 200 participating companies, are expected to attend the week-long American Vacuum Society forum, which features papers from the society's technical divisions.

<http://avs.org/Meetings-Exhibits/Information>

2014 MRS Fall Meeting and Exhibit

Boston, Massachusetts, US

30 November – 5 December

Organized by the Materials Research Society, the regular meeting will host more than 50 symposia spanning biomaterials, electronics and photonics, energy and sustainability, and nanomaterials.

www.mrs.org/fall2014

PacSurf 2014

Kohala Coast, Hawaii, US

7–11 December

The Pacific Rim Symposium on Surfaces, Coatings and Interfaces will showcase the latest advances in biomaterial interfaces, energy harvesting and storage, nanomaterials and thin films.

www2.avs.org/conferences/PACSURF/index.html

2015

VacuumTechExpo 2015

Moscow, Russia

14–16 April

Russia's principal vacuum event, the 10th international exhibition of vacuum machines, equipment and technologies will host leading manufacturers and aims to demonstrate unique achievements in vacuum machinery, equipment, materials and technologies.

www.vacuumtechexpo.com/en-GB

SVC TechCon2015

Santa Clara, California, US

25–30 April

The 58th annual technical conference of the Society of Vacuum Coaters promotes the learning, application and advance of vacuum coating, surface engineering and related technologies.

www.svc.org

16th International Vacuum Electronics Conference

Beijing, China

27–29 April

The biennial conference will provide a forum for scientists and engineers from around the globe to present the latest developments in vacuum electronics technology at frequencies ranging from the UHF to THz frequency bands.

<http://cie-china.org/ivec2015/>

13th IUVSTA School on Vacuum Gas Dynamics

Thessaloniki, Greece

17–21 May

Aimed at researchers who use rarefied gas dynamics but are not experts in the field, the school aims to fill the gap between specialized literature and practical needs.

<http://iuvsta-school2015.mie.uth.gr>

15th International Conference on Atomic Layer Deposition

Portland, Oregon, US

28 June – 1 July

The annual conference addresses the most recent R&D in atomic layer controlled deposition of thin films.

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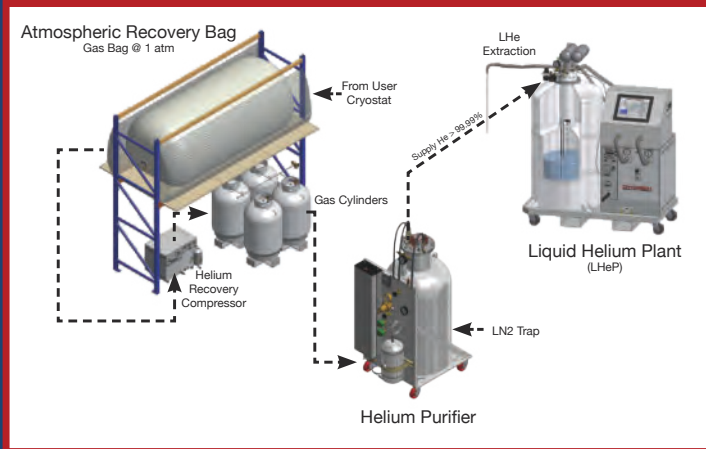
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