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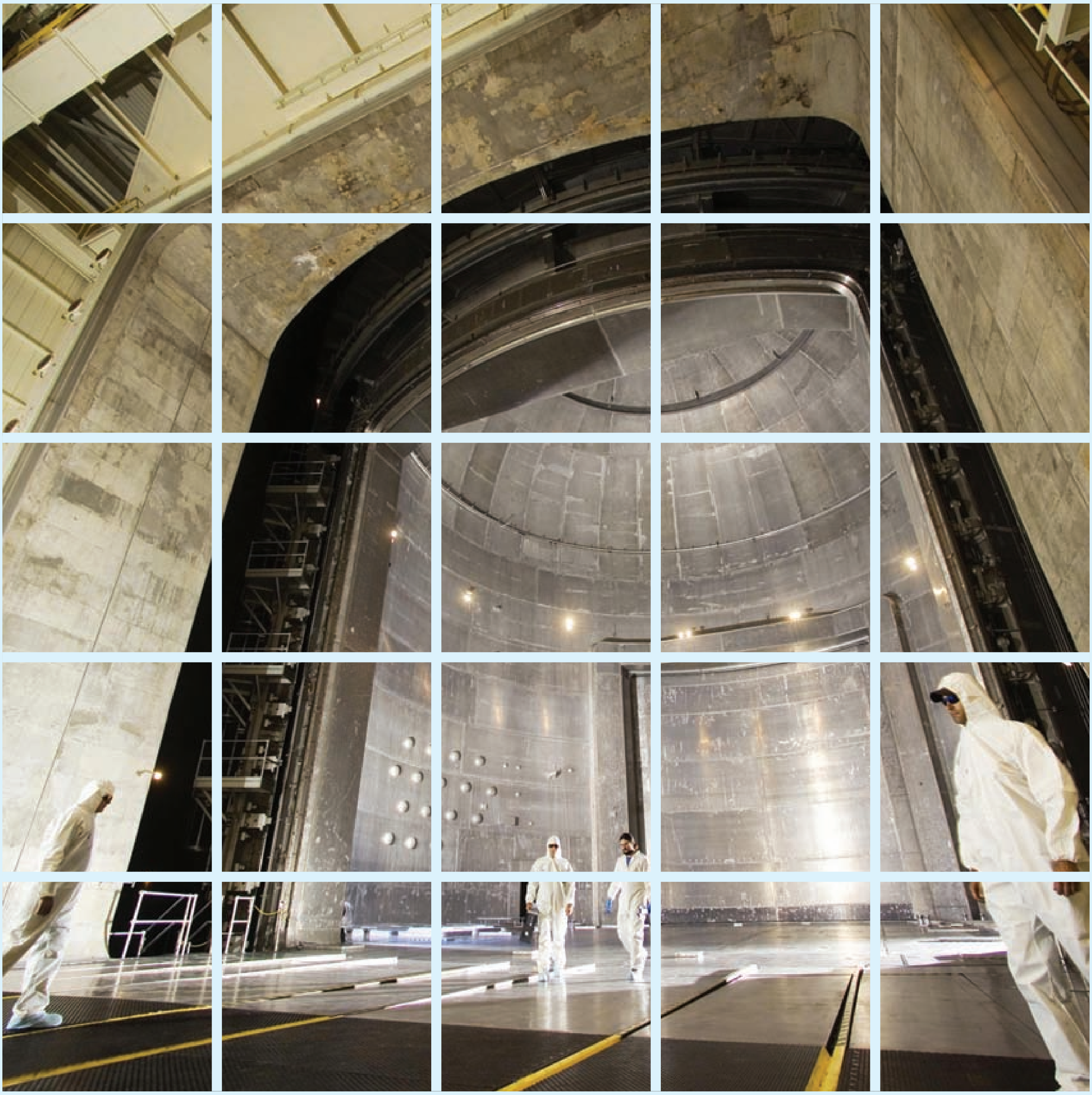
VACUUM CHALLENGES AND SOLUTIONS



NASA

August 2009

Towards portable vacuum standards

Jay H Hendricks and Douglas A Olson examine the challenges of calibrating pressure-measurement guages for vacuum standards.


NIST

Jay H Hendriks next to NIST's ultrasonic interferometer manometer, which provides traceability to the pascal.

Since the middle of the 17th century, when Italian physicist Evangelista Torricelli discovered that a glass tube filled with mercury could be used to measure atmospheric pressure, liquid-column manometers have been used as a primary standard to measure pressure. Even today, they operate on the same principle that pressure can be determined if you know the density and the differential height of a liquid-column manometer, as well as the acceleration due to gravity.

The problem with making reliable pressure measurements in modern research and industrial processes, which can range from ultra-high vacuum (10^{-8} Pa) to atmospheric pressure (10^5 Pa), is that many kinds of gauges are required – from ionization and spinning-rotor gauges to capacitance-diaphragm and resonant-silicon gauges. However, the improper use or incorrect calibration of any of these can result in unreliable measurements that cost time and money.

It is therefore vital that gauges should be traceable to the SI system of units, which have been accurately measured by standards laboratories, such as the National Institute of Standards and Technology (NIST) in the US and other national metrology institutes around the world. Indeed, developing and maintaining suitable high-accuracy standards that allow traceability to the SI units is one of the most significant challenges in vacuum metrology. Standards labs are thus responsible for developing these standards for the unit of pressure – the pascal (Pa).

The NIST Pressure and Vacuum Group operates and maintains mercury and oil liquid-column manometer standards that use pulsed ultrasound interferometry to determine column heights. In this technique a transducer at the bottom of each liquid column generates a pulse of ultrasound (typically ~10 MHz) that propagates up the column, is reflected from the liquid–gas interface and returns to be detected by the transducer. The change in phase of the returned signal is proportional to the length of the column, enabling length changes to be detected with a resolution of 10–20 nm. These primary pressure standards cover 1 mPa – 360 kPa, with uncertainties as low as 5.2 ppm at atmospheric pressure.

The problem is that primary-standard ultrasonic interferometer manometers (UIMs) contain lots of mercury and can only achieve their low uncertainties when operated by skilled personnel in a low-vibration environment with extraordinary temperature control. The UIM at NIST, for example, is in a lab where the temperature is controlled to 0.1 K and measurements are made to within 0.003 K. What metrology institutes, secondary calibration labs, businesses and universities all need are high-stability standards that are easy to transport.

Economic trade agreements require standards labs to compare their primary pressure standards to verify that they are equivalent in achieving the pascal. Additionally, manufacturers and users of pressure gauges want to calibrate their products at their own facilities to save time and money. To address these issues, our group has developed a transportable transfer standard package (TSP) that delivers an SI-traceable calibration with only slightly higher uncertainties than those that could be obtained directly against NIST's UIM primary pressure standard.

A common transfer standard is the capacitance diaphragm gauge (CDG), in which the deflection of a diaphragm is proportional to the applied pressure. CDGs have excellent resolution and short-term calibration stability: long-term calibration drift for recalibrated CDGs is typically only 0.5%.

The calibration stability of the CDG has, however, been a limiting factor in realizing lower calibration uncertainties for low-pressure measurements. For example, NIST's UIM calibrated CDGs provide SI traceability to the pascal for vacuum standards that operate at a much lower pressure (i.e. at higher vacuums). These NIST vacuum standards, which operate by delivering precise flows of gas to ultra-high-vacuum chambers with an orifice of known conductance, are used to calibrate spinning-rotor guages (SRGs) and ionization gauges (IGs).

The SRG operates on the principle that a magnetically levitated and spinning metal ball will decelerate as gas molecules strike it. The IG, meanwhile, works because the gas inside it is ionized and the resulting ion current is proportional to pressure. These gauges are not considered primary standards and therefore they must be calibrated. It is the unbroken-chain of calibration that allows a NIST-calibrated IG or SRG to be traceable to the SI though the NIST UIM. In the example above, the calibration stability of the CDGs is a weak link and NIST began exploring transfer standards with higher calibration stability.

In the mid-1990s the use of microelectromechanical systems (MEMS) enabled pressure-sensor technology to become much more precise and accurate. In particular, resonant silicon gauges (RSGs), which contain two single-crystal silicon resonators encapsulated in a vacuum microcavity, use tiny diaphragms made by micromachining silicon. Changes in pressure on the diaphragm are determined by measuring strain-induced changes in the two resonant frequencies.

Over the past decade, repeated calibration of these gauges has shown that they are very stable, rugged and ideally suited as core technology for a high-stability transfer standard that can be calibrated against primary UIM pressure standards. The RSG sensors (10 kPa and 130 kPa full-scale gauges) have excellent long-term calibration stability of 0.01%, which is over an order of magnitude better than the CDGs. One snag with the RSGs is that their pressure sensitivity is much lower than high-accuracy CDGs below 100 Pa.

Our group at NIST has thus developed and built several high-stability TSPs that are based solely on RSGs or on a hybrid of both CDG and RSG technology. These combine a low full-scale range, excellent resolution and the good short-term stability of the CDGs with the excellent long-term stability of the RSGs. By using the RSG to calibrate the CDG at the time of use, the measurement uncertainty of the CDG is reduced by a factor of 10 or more. The hybrid RSG–CDG technology is housed in a NIST-designed temperature-controlled enclosure that further improves the operational stability of the RSGs and CDGs.

The TSPs consist of a pressure transducer package (PTP) – containing commercially available RSGs and CDGs, manifolds and valves, as well as an ion pump and UHV gauging to maintain and monitor the reference vacuum – as well as support electronics to control, operate and acquire data from the PTP with a laptop computer and custom-designed software. NIST scientists have recently shown that the uncertainty due to calibration stability is in the range of 1–2 ppm at 100 kPa, rising to 0.01% at 100 Pa (*Metrologia* **44** 171).

NIST is currently using these TSPs to provide pressure references for our vacuum-gauge calibration services. Other uses include international comparisons of pressure and a precision atmospheric pressure standard for a secondary-calibration facility in the US. These transfer standards are also expected to find applications in international "round-robin" comparisons of pressure standards that will validate, and potentially lower, uncertainty claims of pressure and vacuum measurements conducted at secondary-calibration laboratories as well as industrial and academic research facilities.

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