Tuesday Afternoon, November 1, 2005

Vacuum Technology
Room 200 - Session VT-TuA

Calibration: Pressure and Flow Metrology

Moderator: P.J. Abbott, NIST

2:00pm **VT-TuA1 A New Determination of the Volume Ratio of the NPLI Static Expansion System*, P. Mohan,*** National Physical Laboratory, India

Recently we have acquired high accuracy resonant silicon gauges of two different ranges, one 130 kPa full scale and the other 1 kPa full scale, for measurement of the initial and final pressures. These gauges have been utilized in a cumulative expansion process for the measurement of the volume ratio of the NPLI Static Expansion System which is nominally 2820. With these new gauges, and with the use of calibrated Platinum Resistance Thermometers mounted inside the vacuum chambers, it has been possible to measure the volume ratio with a relative expanded uncertainty (k=2) of 0.0015. The standard thus characterized has been used to calibrate SRGs at a number of pressures in the range 0.1 Pa to 1 Pa in steps of 0.1 Pa. The Gauge Constant at each of these pressures, defined as equal to the ratio of the indicated pressure to the true pressure is then plotted against the true pressure. The resulting straight line plot has a negative slope and its intercept equals the gauge coefficient. The two SRGs, NPL-0 and NPL-2 thus calibrated are used (i) as a device for measuring the pressure rise in the flowmeter of the NPLI orifice flow system and (ii) as a secondary standard for the calibration of the user gauges.

2:20pm **VT-TuA2 Precise Volume Measurement of Bellows with its Length for a Constant Pressure Flowmeter*, K. Arai,*** *H. Akimichi, M. Hirata,* AIST, Japan

A constant pressure flowmeter has been used to generate very low gas flow from 10@super -5@ to 10@super -10@ Pa m@super 3@ s@super -1@ as a reference standard to calibrate standard leaks. The rate of the flow is obtained by a volume change in a time while the pressure is kept constant. There are several methods to change the volume of the flowmeter. Bellows directly elongated and shortened by a pulse-driven linear actuator was used for precise measurements of the displacement and low outgassing due to the all-metal structure. In this study, the volume change in the bellows was measured as a function of its length by Boyle's law. The flowmeter was isolated from pumps and gas sources. The ratio of the volume change to the initial volume was obtained from the ratio of the pressures before and after shortening the bellows. The absolute value of the volume change was obtained from the difference of the pressure ratio by introducing a well-known volume (ball bearings). Pressure measurements were performed by capacitance diaphragm gauges (CDGs). The repeatability of the pressure ratio measurements was 3x10@super -5@, which was comparable to the chamber temperature fluctuation of 30 mK. The deformation of the bellows was caused by a pressure difference between inside and outside of the bellows. The pressure difference was kept under 2500 Pa by controlling the outside pressure. The remaining deformation was compensated by the value of the pressure difference. The volume change in the bellows by shorting it by 25.00 mm was 32.24 ml. The uncertainty of the measurement was 0.07% after consideration of the temperature fluctuation, the difference from the ideal gas and the thermal transpiration effect of the gauge. The volume changed with its length quadratically, as estimated from the structure of bellows.

2:40pm **VT-TuA3 High-accuracy Vacuum Calibration System*, S. Tison,*** *S. Lu, S. Sukumaran,* Mykrolis Corporation

Many industrial vacuum processes require high-accuracy vacuum measurements to enable process capabilities critical to the device attributes or performance. Semiconductor device manufacture, flat panel displays, optical coatings, and vacuum metalurgy are a few examples where vacuum measurements are critical to product performance. While these measurements are critical, there is a lack of high accuracy industrial calibration systems that can support these needs; particularly at pressures below 100 Pa. A new vacuum calibration system has been developed for calibrating vacuum gages in the range of 1 Pa to 100 kPa with uncertainties ranging from +/-0.02% to +/-0.2% of reading. The paper describes this system which utilizes high stabilty transfer standards in conjunction with pressure expansion techniques to achieve reliable high-accuracy vacuum gage calibration and characterization. The data indicates that automated high accuracy vacuum measurements can be made with a single vacuum system utilizing transfer standards in conjunction with a "primary type" pressure expansion technique which relies on the known ratio of two calibrated volumes to achieve accuracies previously unachievable solely by utilizing vacuum transfer standards.

3:00pm **VT-TuA4 Development of a New High-Stability Transfer Standard Based on Resonant Silicon Gauges for the Range 0.1 kPa - 130 kPa*, J.H. Hendricks,*** *A.P. Miiller,* NIST

The National Institute of Standards and Technology (NIST) has developed a new transfer standard capable of absolute-mode and differential-mode operation in the range 0.1 kPa to 130 kPa. The transfer standard is based on Resonant Silicon Gauges (RSGs) of the same type used to provide superior calibration stability in recent CCM Key Comparisons of absolute and differential pressure standards,@footnote1,2@ which covered pressures up to 1 kPa. The new NIST transfer standard consists of two 10-kPa RSGs, two 130-kPa RSGs, and an ion pump to provide a reference vacuum when making absolute pressure measurements. The pairs of RSGs provide redundancy but, more importantly, they enable Youden analyses to be applied to the data to identify systematic differences between pressure standards. The RSG transfer standard package has demonstrated good short term zero stability and pressure resolution over the 0.1 kPa to 130 kPa range, and has demonstrated long term instability of a few ppm at 130 kPa, increasing to 0.01% at 0.1 kPa. In terms of long term stability, the new NIST transfer standard package is nominally commensurate with piston gauges at pressures down to 10 kPa, and extends measurement capability down to 0.1 kPa. Although its intended use is for intra-laboratory comparisons of primary standards at NIST, this transfer standard may also find applications in international comparisons of pressure standards. @FootnoteText@ @footnote 1@ Miiller, A. P., Bergoglio, M., Bignell, N., Fen, K. M. K., Hong, S. S., Jousten, K., Mohan, P., Redgrave, F. J., and Sardi, M., Final report on key comparison CCM.P-K4 of absolute pressure standards from 1 Pa to 1000 Pa, Metrologia, 2002, 39, Tech. Suppl., 07001.@footnote 2@Miiller, A. P., Cignolo, G., Fitzgerald, M. P., and Perkin, M. P., Final report on key comparison CCM.P-K5 of differential pressure standards from 1 Pa to 1000 Pa, Metrologia, 2002, 39, Tech. Suppl., 07002.

3:20pm **VT-TuA5 New Calibration Apparatus for 1, 10, 100 Torr Capacitance Diaphragm Gauges*, S.Y. Woo,*** *I.M. Choi,* Korea Research Institute of Standards and Science, Korea

Capacitance diaphragm gauges are electromechanical pressure sensors in which the displacement of a stretched thin metal diaphragm is detected by a capacitance measurement. They have gained widespread international popularity not only as high accuracy transfer standards but also as reliable replacements for many other vacuum gauges used in process industries. In order to calibrate such accurate vacuum gauges, laser or ultrasonic mercury manometers have been used. However, complexity, health concerns of mercury vapor, and cost of these manometers made it difficult to use in most calibration laboratories. As a substitute, a gas-operated pressure balance can be used with some modifications. In this article, we introduce a new apparatus for the calibration of the precise vacuum gauges. This device has a unique mass handling structure that allows masses to be changed in-situ without breaking the vacuum. It covers three pressure ranges of 1 Torr, 10 Torr, and 100 Torr. Using this apparatus, the characteristics of three commercial vacuum gauges (MKS, Model 626) were studied.

3:40pm **VT-TuA6 Real Time Calibration of Leak Detection Sensitivity*, X. Chen,*** *T.B. Huang, L.B. Xiao, L.Z. Cha,* Tsinghua University, China

The most important characteristic for leak detection is the smallest leak rate that an instrument, method, or system is capable of detecting under specified conditions. It is used to call the smallest detectable leak rate the leak detection sensitivity. One permeation type reference leak with a fixed leak rate is commonly used to calibrate the leak detector and the method has been standardized. It is no doubt that is basic and important to guarantee the instrument in optimized condition. However, the real level of the leak detection sensitivity depends on not only the test instrument, but also the system and methods. The detection limit will be higher or lower if the partial flow pump or accumulation method is used. Sensitivity calibration is necessary to distinguish between leak detector and leak detection. The demand for quantitative leak tests has increased sharply in recent times because the information is more important for the quality control. Real time calibration of the leak detection sensitivity can be achieved by comparing the tested leak rate with a fixed reference leak rate, a series of such reference leak rates or a variable reference leak rate. The last one is optimal for calibration since it provides the reference leak rate in the similar range of the tested leakage rate under the real situation. An adjustable reference leak rate system is put forward with wide dynamic range from 10 @super -7@ to 10 @super -14@Pa.m @super 3@ /s by changing the inlet helium pressure and concentration of a platinum wire-glass reference leak with molecular flow characteristics. The real time calibration for the leak detection has been realized quantitatively not only for the instrument, including the ultra sensitive MSLD system, but also for the methods and system, even for the ultra sensitive leak detection. The detected leak rate under the real situation can be quantitatively calibrated which attracts more and more attention but has not been standardized until now.

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