Measurement 131 (2019) 269–276

Contents lists available at ScienceDirect

Measurement

journal homepage: www.elsevier.com/locate/measurement

Remarks concerning about the characteristics of the extractor vacuum gauge and the Quadrupole Mass Spectrometer

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ARTICLE INFO

Article history:
Received 9 April 2018
Received in revised form 17 August 2018
Accepted 21 August 2018
Available online 23 August 2018

Keywords:
Extractor Vacuum Gauge
Quadrupole Mass Spectrometer
Correction factor
Stability
Calibration

ABSTRACT

In this study, the stability of an extractor gauge (EXG) was investigated through the determination of correction factors (CFs) over a pressure range 5×10^{-5} Pa to 9×10^{-3} Pa by using a Spinning Rotor Gauge (SRG) traceable to UME Static Expansion System, during 20 months and 15 months for N_2 and He, respectively. Percent and fractional changes in CFs were studied and sensitivities of EXG were also presented for Ar and CO_2. Direct comparison method was used for EXG stability measurements. Additionally, sensitivity measurements of Quadrupole Mass Spectrometer (QMS) were performed in the range of 1.0×10^{-6} Pa to 1.0×10^{-2} Pa at emission currents of 2 mA, 0.4 mA and 0.1 mA for He, N_2 and Ar. Long-term stabilities of QMS for N_2 and He were analyzed for about 2 years. EXG and SRG were used for QMS sensitivity measurements.

The changes in CF of EXG remained within ±5% and ±6%, and fractional changes remained within about ±0.03 and ±0.05 for seven and five different measurement runs for N_2 and He, respectively. The change in CF of EXG based on the mean value of the relevant pressure point is 5.8% and 7.8% at 5×10^{-5} Pa, 5.3% and 5.9% at 9×10^{-3} Pa, and maximum CF changes relative to the first calibrations ranges from −1.7% to 2.4% and from −1.0% to 4.7% within the selected pressure range for N_2 and He, respectively. The changes of QMS sensitivity are strongly dependent to the electron current. Maximum changes of QMS sensitivities over a three month period are 44% and −35% for N_2 and He, respectively. This study was conducted in the frame of EMRP IND12 "Vacuum Metrology for Production Environments" project that was funded by the European Metrology Research Programme (EMRP).

1. Introduction

Quadrupole Mass Spectrometer (QMS) is usually used for characterizing the vacuum environment, determining the partial pressures or diagnosing the residual gas spectrum and various vacuum process based applications in high and ultrahigh vacuum systems. The operation of QMS requires optimization of several electrical parameters including emission current, cathode to anode energy (electron energy), focus voltage etc. The instability of sensitivity of QMS resulting from improper selection of the parameters as well as the usage history of the instrument is well known from previous studies [1–5]. Therefore, it is essential to figure out the stability of QMSs for reliable partial pressure measurements.

The main motivation for this study was to investigate the long-term stability of extactor ionization gauges (EXG). Ionization gauges, such as extractor gauges (EXGs), are widely used for pressure measurement down to about 10^{-10} Pa as transfer standards among metrology laboratories. In this study, one of the most widely commercial XHV gauge, extractor gauge, was used to read pressure measurements. The present work is concerned with measurement results of the QMS and the EXG stabilities over a certain period using dynamic vacuum system.

2. System overview and procedures

A schematic of the measurement set-up is shown in Fig. 1. The system can be roughly divided into two parts, that is, upper chamber and lower measurement chamber. The vacuum chambers are fabricated using stainless steel 304 material in a cylindrical form of diameter 212 mm and the length of 300 mm. Seven DN40CF ports and one DN63CF port are placed symmetrically around the circumference of each chamber. Two ports are used for Omega PT100 1/8 in. temperature sensors. The two chambers are separated by a 10 mm diameter-orifice, which is demountable. The entire chamber is evacuated by a turbomolecular (260 L/s for N_2) and a scroll pump (max.260 L/min) through the orifice. A MKS Spinning Rotor Gauge (SRG), an extractor gauge with iridium yttric

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https://doi.org/10.1016/j.measurement.2018.08.043
0263-2241/

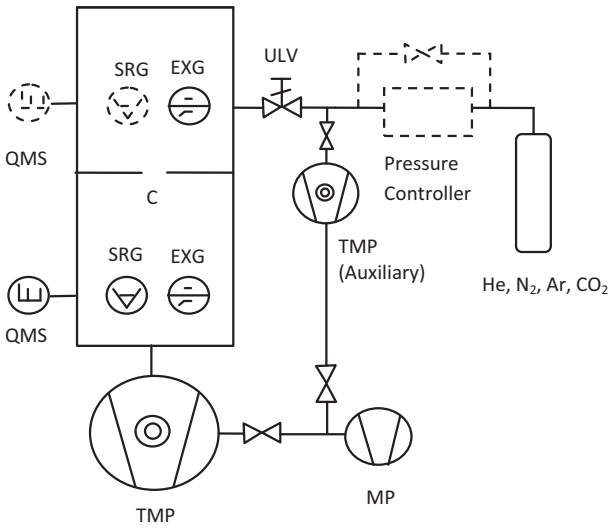

Fig. 1. Schematic diagram of the measurement system is shown. MP: mechanical pump (scroll); TMP: turbomolecular pump; QMS: quadrupole mass spectrometer; SRG: spinning rotor gauge; EXG: Extractor gauge; C: orifice; ULV: ultra-high precision leak valve. Dashed lines represent different configuration used in this study.

oxide coating filament, 1.6 mA emission current and 120 eV nominal electron energy (Leybold IE514 and controller IM540) and a QMS (INFICON, Transpector H200M) not facing each-other are attached at the same horizontal plane of the lower chamber. Compact full range gauges are also mounted on both chambers. All of the gauges were operated by controllers provided by their manufacturers. Ultra-high precision leak valve with the minimum leak rate of 1.3×10^{-11} Pa·m^3/s is provided on the top of the chamber to maintain the desired pressure in the chamber with a specified gas. High purity (99.998%) gas is supplied to the upper chamber through the adjustable leak valve. The ultimate pressures in the chambers are measured by the extractor gauges in the range from 10^{-2} to 10^{-10} Pa. The pumping speed of the auxiliary turbomolecular pump used for evacuation of the gas filling line is 33 L/s for N_2. Correction factors of the extractor gauge are determined by comparison method using the SRG.

2.1. Procedure for EXG stability measurements and evaluation

The calibration factor of the EXG used in QMS sensitivity measurements has to be observed and examined in order to improve the accuracy of the sensitivity measurements. Correction factor (CF) of EXG is defined as the ratio of the true pressure divided by the observed pressure. At first, EXG was mounted on lower chamber of the dynamic vacuum system and the system was evacuated to reach base pressure on the order of 10^{-7} Pa, which is usually takes about one day. No baking procedure was applied to the system including the gauge. Gas purging was applied at about 10^{-4} Pa for about one hour while the filament on. Reference gauge SRG and EXG were turned on and operated at least one day before the measurement. Both gauges were mounted at the same horizontal plane of the measurement chamber for testing. Impurities in vacuum chamber were always checked using the Quadrupole Mass Spectrometer prior to each measurement.

EXG was calibrated by comparing its pressure reading, P_{EXG}, to that the reading of the SRG, P_{SRG}, traceable to UME Static Expansion System.

$$CF = \frac{P_{SRG}}{P_{EXG} - P_{Base}} \quad (1)$$

where P_{Base} is the pressure indication at the background pressure. As seen, pressure readings were obtained by subtracting the background pressure from the indicated pressure.

Subsequent calibrations were carried out over a pressure range 5×10^{-5} Pa to 9×10^{-3} Pa at selected pressure points per decade at intervals of about 3 months during 20 months and 15 months for Nitrogen and Helium, respectively. Pressure readings were taken in increasing sequence.

It is well known that the ionization gauge reading is related to gauge sensitivity by the relationship $P_{EXG} = i_{ion}/(i_e.S)$, where i_{ion} is the ion current on the collector, i_e is the emission current and S is the sensitivity of the gauge [7]. Since pressure readings are taken only from the controller, gauge sensitivity is not determined during the measurements. Because inversion correction factor 1/CF is inversely proportional to sensitivity, gauge sensitivity will affect the measured correction factor.

2.2. Procedure for QMS sensitivity measurements

Sensitivity measurements were taken in a certain pressure range useful to observe QMS linearity. The applied procedure was as follows: No baking procedure was performed in testing. For the calculation, net ion currents of QMSs were obtained by subtracting ion currents at background pressure from those during the measurements. QMS and EXG were warmed up for at least one day before the measurements. The temperature change of the chamber over one day was within 1 K. Emission current value was selected as 2 mA, 0.4 mA ve 0.1 mA. Zero scans were taken for each emission current value with scan range 10–30 amu for Nitrogen and 0–6 amu for Helium, and 19–41 amu for Argon. Dwell time was selected as 1024 ms. In addition to the QMS signal, EXG or SRG readings were taken depending on the pressure range. Only Faraday cup was used as detector, secondary electron multiplier (SEM) was not used in measurements at all. The correction factors determined by SRG were applied on the EXG's pressure readings. As a first step, nominal emission current 2 mA was selected and five different scans were carried out. Thereafter, the next measurements for 0.4 mA and 0.1 mA were performed in the following days. Each measurement for each selected emission current lasted about one working day. Targeted pressures were obtained with variable leak valve and recorded in ascending sequence. Measurements were taken at fifteen different pressure values between 1.0×10^{-6} Pa and 1.0×10^{-2} Pa. Pressure values were selected as follows: for $a \times 10^{-x}$ Pa, where a = 1,3,7; x = 6, 5, 4 with EXG and for $b \times 10^{-y}$ Pa b = 1, 2, 3, 5, 7; y = 3 and for 1.0×10^{-2} Pa with SRG. Nitrogen equivalent mean base pressure value indicated by EXG was 3.9×10^{-7} Pa for selected three emission current measurements. Mean of pressures and QMS signal values of five runs were used to determine the QMS sensitivity. The following formula was used to determine the sensitivity:

Table 1
Technical specifications of the QMS used in current study.

Specifications	
Mass range/amu (atomic mass unit)	1–200
Filament material	Thorium oxide iridium
Type of Ion source	open
Emission Current/mA	2
Nominal Electron Energy/eV	102
Focus Voltage/V	27
RF Operation Frequency /MHz	2.125
Quadrupole rod diameter/mm	6.35
Field axis potential/V (ion energy in mass filter)	10
Sensor length (vacuum side)/mm	264