Behaviors of capacitive and Pirani vacuum gauges

Case study on time effect

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1 Introduction

Today, extensive research is carried out in various industrial fields such as nanotechnology, microelectronics, aerospace, medicine, pharmacy, etc. In all these industries, complex processes are required in the vacuum environment in order to achieve a high-quality product, so accurate measurement of the pressure is really important [1–3]. For example, the pressure of the operating environment in deposition systems is one of the most effective quantities in the quality of the properties of the layers and the final products.

The physical, electrical, mechanical, and optical properties of the layers, including adhesion, density, permeability, surface strength, hardness, applied stress, transmission coefficient and refractive index change during deposition by pressure variations [3, 4–7].

At the moment, vacuum systems are at the forefront of research activities in innovation and high-tech products. Therefore, with the increasing use of these systems, especially in the area of the up and middle vacuum in the new sciences and industries, it is necessary to develop appropriate measurement methods and to use gauges that are reliable in accuracy and the validity. In other words, they are needed to be calibrated because the calibration of the sensors is important due to the repeatability of the process, achieving optimum quality, tracking production problems, preventing damage to the vacuum generator and user safety [8–11].

Vacuum gauges of McLeod, thermocouple, Pirani and capacitive are among the most widely used equipment in vacuum systems. Due to the wide range of pressure in these systems, a combination of two vacuum gauges of Pirani and capacitive is used in the chamber to measure the pressure in the lower and medium range (range 1013 to 10⁻³ mbar) and the high vacuum range (range 10⁻³ to 10⁻⁷ mbar), respectively [12, 13].

In this study, the calibrations of two Pirani and capacitive vacuum gauges are investigated. The Pirani vacuum gauge is a thermal conductivity vacuum gauge in which platinum, nickel or tungsten filaments are used so that it is designed and constructed based on the resistance dependence of a heated wire to its temperature. Since the performance of these vacuum gauges types is influenced by the dimensions and the thermodynamic properties of gas molecules with filaments, the pressure reading is completely dependent on the gas type existed.

SUMMARY

Knowing the exact pressure of the process has a great impact on the validity of the results, product quality, energy efficiency, and in some processes, on the security of work with the system. Hence, the calibration of the barometers and the accuracy of the readings should be taken seriously. Choosing the appropriate time interval for re-calibration is done according to the extent and conditions of use, uncertainty, and the inaccuracy allowed in measurement, constructive suggestion, and some other things. Failure to pay attention to the importance of periodic calibration in vacuum gauges leads to some irreparable losses in the research project and the vacuum generator system. In this study, using McLeod’s barometer, the deviation of capacitive and Pirani vacuum gauges is investigated at different time intervals in the middle vacuum range, and it is determined that the vacuum gauge faces a serious deviation from the actual calibrated amount for upper and lower ranges of middle vacuum in the same working pressure range over time.
in the compartment environment. A dry and neutral gas is usually used to calibrate this type of vacuum gauges, and the correction factors are applied based on the gas type. The capacitive vacuum gauge consists of a very thin flexible diaphragm and two fixed conductive plates, the so-called electrode. In fact, these two conductive plates are two panels of a capacitor with varying capacities, which under the pressure difference between the enclosure and the adjacent space, the diaphragm goes closer or farther to the fixed electrode, and thus the capacity of the capacitor varies by changing the enclosure pressure. The electronic circuit of this vacuum gauge converts the variable capacity to the equivalent pressure and displays it in the monitor.

The main advantage of this vacuum gauge is that the reading pressure is not dependent on the gas type in the compartment environment. The only parameter influencing the vacuum gauge performance is the pressure difference between the two environments, which is independent of dimensions, thermodynamic and electrical properties or other features of calibration gas [13]. As these gauges, like other measurement devices, lose their accuracy over time and require calibration, a comparison between the pressure reading and a standard pressure has been used for calibration of these vacuum gauges, and then the deviation amount in measurement is calculated and correction coefficients will be applied [8, 9, 14].

There are two primary and secondary standards for calibration of the vacuum gauges. Primary standards have the highest measurement quality which these standards are based on physical laws and accurate measurements of related physical quantities. In the secondary standard, a comparison is used between the vacuum gauge that is under calibration and one which is calibrated. A vacuum gauge which is calibrated in accordance with the primary standards and other vacuum gauges are calibrated in comparison with it, is considered as the reference vacuum gauge. The most common primary standards are mercury vacuum gauge, volumetric expansion and aperture current [15, 16]; these three primary standards and secondary standard are used for pressure calibration in many international standardizations and calibration centers [9, 10, 15, 17-19]. Depending on the vacuum gauge type and how it works, a proper timeline should be selected for their re-calibration.

There are many factors affecting the timeline selection including the constructive advice, the uncertainty needed to measure, the maximum permissible inaccuracy, the risk of process execution outside the desired range, the presence of corrosive gases in the process, environmental conditions (temperature, humidity, vibration, radiation, etc.), previous calibration data, maintenance information, user training, operating conditions and amounts [20]. In this paper with the technical report, based on the factors mentioned above, the timeline effect on the calibration of the two vacuum gauges was investigated, then, according to the functional pressure range of the two Pirani and capacitive vacuum gauges, they are calibrated using the primary standard method accompany with the McLeod mercury vacuum gauge and the results are presented in this report.

2 Experimental Details

In this research, a McLeod mercury vacuum gauge is used which can show pressure to $10^{-3}$ mbar with a sufficient accuracy. McLeod vacuum gauges are generally composed of a mercury reservoir, a double-glassed tube, a scaled plate and a closed tube. The image of this vacuum gauge is shown in Fig. 1.

A NW10 flange, connected to a quartz glass tube, is connected to the vacuum gauge output. The sealing of this tube and the above flange is achieved by three silicon O-rings between its inner wall and the outer wall of the flange, so that the total leakage at the connection point decreases.
to its minimum during the calibration period.

The volume of mercury needed for this gauge is between 210 and 240 cm³ for each type. Mercury should be purged into the gauge with the safety criteria and under a ventilator cavity. In normal conditions (before experiment), the mercury reservoir should be in a horizontal position and the NW10 openings should be closed with a suitable cap. To carry out the calibration by this system, the opening is connected to a vacuum system with NW10 connector (Fig. 2). A three-way vacuum connection is used to install non-calibrated vacuum gauge alongside McLeod.

Vacuum connections include several pieces. For each of the vacuum gauges connected to the vacuum generator system, there must be a manual valve located between them and the vacuum compartment. The vacuum gauges should be completely symmetrical relative to the symmetry plate of the compartment both in terms of geometry and connections and parts connected to them. The chamber may have two symmetry plates, which is the symmetry plate that passes through the pump and divides the strait between the discharge pump and the compartment into two equal parts. Now, after complete installation of the vacuum gauge for calibration and the reference vacuum gauge, the compartment will be discharged when the entire calibration system is ready. During the calibration process, the following points are required:

- The temperature range of the compartment should be 23 ± 3 °C and should not vary more than 1 degree during the calibration period.
- For calibration gas, 99.9% or higher purity of nitrogen is recommended. Other gases with the same purity or even mixture of gases with suitable ratios may be used in calibration.
- The base pressure in the calibration chamber should be less than 0.1 times the lowest determined pressure value by the reference gauge for calibration. Basic pressure should be lower than difference between the vacuum gauge under calibration and the reference vacuum gauge.
- Before starting the calibration, the base pressure and all zero reads of the vacuum gauges should be recorded.
- Care should be taken to maintain the effective discharge rate during the calibration process. Any return of oil vapor to the vacuum system should be prevented.
- Gas inlet can be in one of these two ways: the gas enters the pipe between the vacuum chamber and the pump system or separately is placed on the vacuum chamber symmetry axis.
- The base pressure p₀ can be measured in the same conditions as the vacuum system during calibration. First calibration pressure, in both, static models or stable equilibrium, is applied according to the following paragraphs.

After completing the measurements at the final target pressure, the device should be drained during calibration for controlling the leakage, significant absorption, wall contamination, or defect in the pump and so on. It is necessary for the machine to reach the base pressure or if possible, 0,001 times the final pressure for a maximum of 10 minutes. Otherwise, the calibration system should be reorganized (e.g. leak test, pump test, cleaning, cooking) and the calibration process should be repeated.

In this study, capacitive vacuum gauge (made by Edwards, the Baro-cel 600 model) and the Pirani vacuum gauge (manufactured by Edwards, the PR10K model), after 14 to 72 months passed from its calibration, are individually and symmetrically connected to vacuum device (produced by ACECR, Iran) with a McLeod vacuum gauge. The reason to set vacuum gauges symmetrically is to measure the pressure of the same by them. An interface vacuum valve is inserted between the vacuum compartment and the vacuum gauges which this valve is initially closed. Before discharging and unlocking this valve, the PR10K sensitivity monitor became warm and stable in on mode for 20 minutes. Then, the compartment is discharged by the rotary pump, and the pressures displayed by the two barometers are recorded and their curves are plotted against time.

3 Results
3.1 The results of Pirani vacuum gauge calibration

As stated in the introduction, the Pirani barometers are known as heat transfer vacuum barometers which its main part is a thin filament with a high thermal resistance coefficient which is exposed to the pressure of the vacuum compartment. By applying a voltage to both filament sides, it is heated, and then, by decreasing the chamber pressure, the number of molecules decreases. Consequently, the collision with the filament is also declined which means that less heat is taken from the filament. Since the resistance is temperature dependent, the filament resistance is increased and fewer current passes through the filament for a constant voltage. Change is the current indicates the change in the gas pressure. In another model of the Pirani barometric, with the attachment of a variable resistance to the filament, the filament resistance is maintained constant independent from the temperature decrease. The second type measures the wider range of pressure with a greater accuracy.

Whereas the performance of this barometer is affected by the thermodynamic properties and dimensions of gas molecules, the pressure reading is completely dependent on the type of gas in the compartment. In the calibration of these barometers, a dry and neutral gas is usually used, and then the correction factors are applied based on the gas type. On the other hand, to remove the environmental changes and effects on the filament, it is placed in Wheatstone bridge circuit, and its variation is compared with a reference filament. The filament is the most vulnerable part of Pirani barometer such that deposition, corrosion, oxidation, deformation and its dimensions can cause an inaccuracy in the pressure reading due to frequent or incorrect use and drive out the pressure gauge from the calibrated state. Therefore, there is a probability of being non-calibrated for this barometer in short periods is possible based on the amount and severity of its use.

In the following, the time passage effect on the calibration of the Pirani vacuum gauge PR10K model, manufactured by Edwards Company, is studied.
over a time period which the results are shown in Fig. 3. It can be seen that whatever the vacuum gauge deviates from the origin of the calibration time, it goes further offset from the calibrated state, so the measurement inaccuracy increases over time because the vacuum gauge sensor does not show a standard inaccuracy in the range of its mission, i.e. range from 0.01 to 1 mbar. This is illustrated in Fig. 4 as the curves are the same as the curves in Fig. 3 with the difference that the scale of the shaft axis is a logarithmic scale. It is clear from Fig. 4 that the shown pressure difference becomes irregular with respect to the higher pressures at a pressure of up to 0.01 mbar which is the application range of these vacuum gauges.

Furthermore, the pressure variation curve (Fig. 3) shows that the deviation threshold of the Pirani vacuum gauge (PR10K model) from the calibration is tangibly within the pressure range of 0.05 mbar, and at the pressures greater than 0.45 mbar, it deviates from the calibrated state of the display.

Because the quantities of this vacuum gauge are of a resistive type, it can be well calibrated by the original standard methods using McLeod calibrator. In Fig. 5, the time variation in the deviation of the Pirani vacuum gauge PR10K model is plotted at pressures of $4.7 \times 10^{-2}$ mbar, $6.3 \times 10^{-2}$ mbar, and $1.8 \times 10^{-1}$ mbar for a period of 6 years. Obviously, in the first year of calibration, the vacuum gauge deviation from the real value is lower than those in subsequent years.

### 3.2 Calibration results of capacitive vacuum gauge

As previously mentioned, the capacitive vacuum gauge is made of a very thin flexible diaphragm and a fixed electrode. In fact, these two conductive plates are two plates of a variable capacitor, so that the diaphragm is located between the two regions with constant pressure and enclosure pressure and it is well sealed from both sides. Under the pressure difference of the enclosure with the adjacent space, the diaphragm becomes far or close to the fixed electrode, and consequently, the capacitance of the capacitor alters by changing the enclosure pressure. The electronic circuit of this barometer converts the variable capacitance of the capacitor to an equivalent pressure and displays it in the monitor.

The main advantage is the independence of the recorded pressure on the type of gas since the parameter affecting the pressure is only the pressure difference between the two environments and is independent of dimensions, thermodynamic and electrical properties, or other calibration gas characteristics. This issue, coupled with the stability, accuracy, and responsiveness, has made it possible to calibrate with an original standard for calibrating other barometers as a secondary standard.

The mechanical components of the capacitive vacuum gauge are in such a way that if they have been used correctly and normally, the time period required for re-calibration will be long. The only part that can bring inaccuracies in the pressure measurement is its diaphragm. The shape change and the diaphragm flexibility due to repeated use will unintentionally change distance between the diaphragm and the electrode, resulting in an inaccuracy at reading pressure. Therefore, it is necessary to calibrate the capacitive barometer after some time so that the change in the capacitance corresponding to the change in the diaphragm shape should be taken into account at the reading pressure.

In order to investigate the above issue, a capacitive vacuum gauge, the Barocel 600 model made by Edwards Company, is connected symmetrically to the inputs of the EDS100 vacuum calibration system after 32 and 70 months from the calibration, and its deviation or non-deviation from the calibration condition have been investigated.

Fig. 6 shows the reading pressure variation curve from capacitive vacuum gauge before calibration compared to the McLeod vacuum gauge with 72 months of operation.

It can be seen from the figure that, over time, the sensors and hardware function of the vacuum gauge have more deviated from the calibration condition and the measurement inaccuracy is increased because the sensor in its function area, range from 0.01 to 0.5 mbar, even shows negative values (Fig. 7). However, the variation curve
after 32 months illustrates deviation from the calibrated state. Its variations are moderately routine and almost in line with changes in a linear sensor. Thus, it is not possible to correct the read value in disturbed areas with any linear approximation or other methods, but it can easily be calibrated in calibrating of a vacuum gauge at the right time.

Over time, the deviation variations of the capacitive vacuum gauge (B1600) have also been investigated at the pressure of 4 mbar during an 8-year period.

4 Conclusions

In this research, the behaviors of capacitive and Pirani vacuum gauges have been investigated over time. The behaviors of the Pirani vacuum gauge PD-PD10 model and the capacitive vacuum gauge Barocel 600 model manufactured by Edwards Company during 6 and 8 years is evaluated so that over time, the measurement of these devices, especially at the edges of the permissible measurement range, experiences deviation.

It also turned out that the measurement deviation value of these devices rises by increasing time from calibration time. The measurement inaccuracy of the capacitive vacuum gauge after 8 years is such that it shows negative numbers in the lower measurement limit range, and it also has 50 % inaccuracy in other areas. This increase in the inaccuracy for the Pirani vacuum gauge is far away more so that the measurement inaccuracy rate increases to more than 100 %, especially on the edges of the permissible measurement range, after only one year of calibration. Over time, the measurement inaccuracy increases exponentially that after 6 years, the inaccuracy rate at the edges of the permissible range of measurement is more than 1000 %.

In the case of measurement devices, it is observed that, in a short time or in the middle range of the permissible measurement range, the measurement value can be compensated for some extent by a coefficient. As regards, over time and at the edge of the range, the measurement deviates from the linear state and these changes become more irregular and cannot be corrected.

According to the results of this study, it is strongly recommended that the vacuum gauge is calibrated after each incorrect use or damage to avoid significant inaccuracies in the measurement. Moreover, in order to achieve an inaccuracy range of less than 10 %, the calibration time should be under 6 months for the Pirani vacuum gauges and less than 2 years for the capacitive ones.

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