Development of a high-precision vacuum standard by means of the expansion method

Toshiyuki Kataoka

Key words
Vacuum measurement, vacuum standard, expansion method, JCSS, national standards

In order to ensure the reliability and traceability of the Sapphire Diaphragm Gauge (model SPG), manufactured and sold by Azbil, we introduced a vacuum generation system that is based on the expansion method as our vacuum standard. We investigated the factors that cause measurement uncertainty and reduced them. Then, the accuracy of our vacuum generation system was verified by comparing our measurement values with those of the National Institute of Advanced Industrial Science and Technology (AIST). The results confirmed that the system had sufficient capabilities. On that basis, we were registered as a calibration service provider of JCSS (Japan Calibration Service System) in the vacuum gauge category, securing the traceability, etc., of the SPG Sapphire Diaphragm Gauge.

1. Introduction

In recent years, the importance of vacuum measurement is increasing, especially in semiconductor manufacturing processes. Azbil has been selling model SPG sapphire diaphragm vacuum gauges (fig. 1), mainly for semiconductor production applications, since 2011. This product uses sapphire as the material for the sensing element because it provides excellent corrosion resistance, heat resistance, and mechanical properties [1] [2] [3].

Fig. 1. External appearance of model SPG sapphire diaphragm vacuum gauges

A vacuum is a state in which space is filled with gas at a lower pressure than normal atmospheric pressure. Azbil's diaphragm vacuum gauges are used to measure pressure in the vacuum range from near atmospheric pressure to several ten thousandths of atmospheric pressure.

To maintain traceability at the precision level required for our sapphire diaphragm vacuum gauges, a higher precision vacuum standard is necessary. However, since there was no calibration service provider that could supply such a standard, Azbil needed to establish a vacuum standard itself.

This article discusses the vacuum standard established by the Technology Standardization Department's Measurement Standards Section, whose purpose is to evaluate the characteristics of Azbil's diaphragm vacuum gauges and to calibrate them with adequate reliability for their specified degree of precision. The article also discusses measures to reduce measurement uncertainty.

2. Overview of the expansion method

Regarding the pressure range covered by diaphragm vacuum gauges, a piston pressure balance, which generates a highly stable and precise pressure, is generally used as a pressure standard for pressures from around 5000 Pa up to around atmospheric pressure, and the Measurement Standards Section owns one. The structure of a piston pressure balance is shown in figure 2. The pressure medium is pressurized to cause the piston to float. A weight is put on the piston, and when the forces are balanced, a stable pressure is generated. Since pressure is defined as force per unit area, the pressure generated by the piston pressure balance can be obtained by dividing the downward force due to the mass of the weight and piston by the effective cross-sectional area of the piston cylinder.

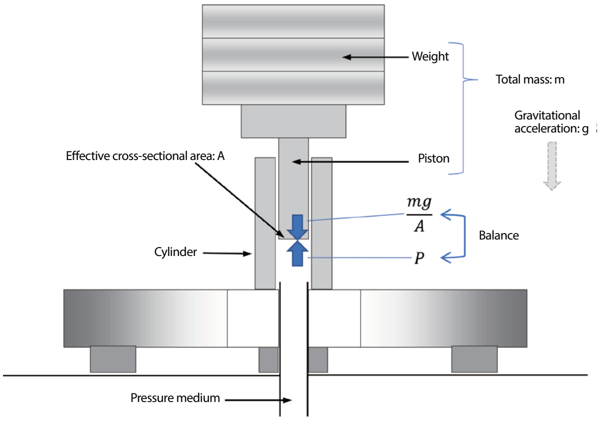

Fig. 2. Measurement principle of a piston pressure balance

However, the sapphire diaphragm vacuum gauges mentioned above have particular measurement ranges, such as the 133.32 Pa range or the 1333.2 Pa range, all of which cannot be covered only with the piston pressure balance. Accordingly, we decided to make a vacuum generation apparatus based on the expansion method (fig. 3) to serve as a precision standard that would accommodate such vacuum ranges.

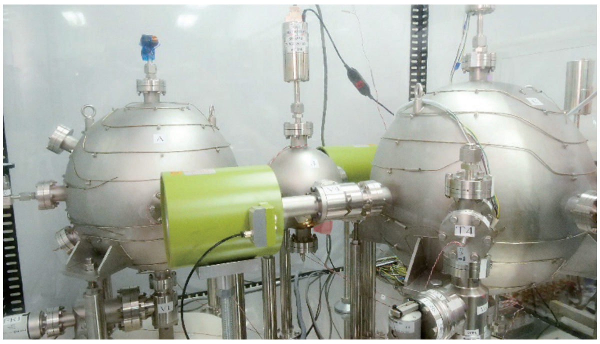
Fig. 3. External appearance of the expansion method system

The expansion method is a way of generating a specific vacuum pressure. It is shown in figures 4-1 to 5.

(1) Two chambers, A and B (with a capacity of V_A and V_B, respectively), are connected via a valve, and each chamber has a pipe for evacuation. The vacuum gauge being calibrated or an absolute pressure gauge for initial pressure measurement is mounted on one of the chambers. The absolute pressure gauge is calibrated beforehand with a piston pressure balance.

(2) First, when the valve between the chambers is open, gas with a pressure of several tens of kPa is injected and the initial pressure inside the chamber P_0 is measured (fig. 4-1).

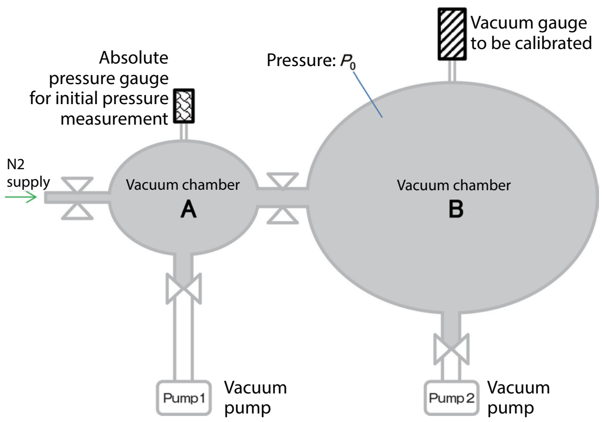

Fig. 4-1. Principle of the expansion method: description (2) in the text (simplified schematic diagram)

(3) The valve between the chambers is closed (fig. 4-2).

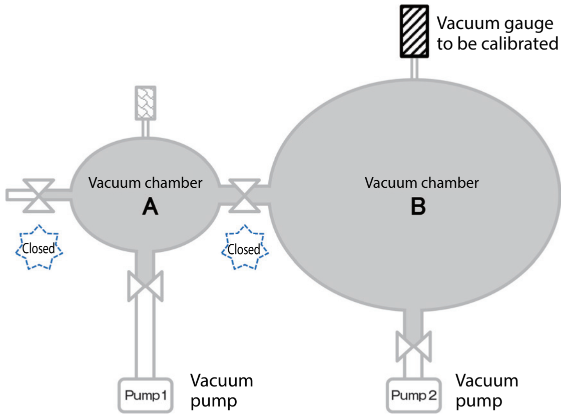

Fig. 4-2. Principle of the expansion method: description (3) in the text

(4) Only one of the chambers (chamber B, in this case) is evacuated (fig. 4-3).

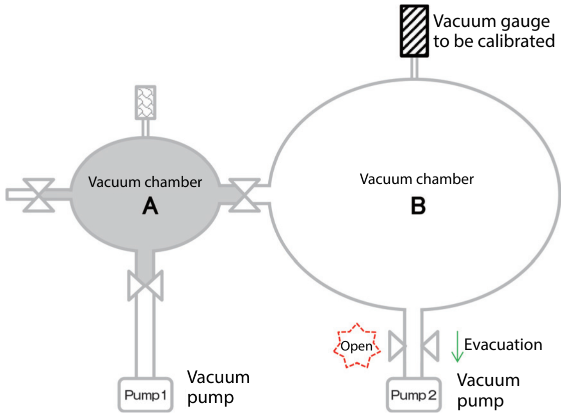

Fig. 4-3. Principle of the expansion method: description (4) in the text

(5) When evacuation is complete, the chamber is sealed (fig. 4-4).

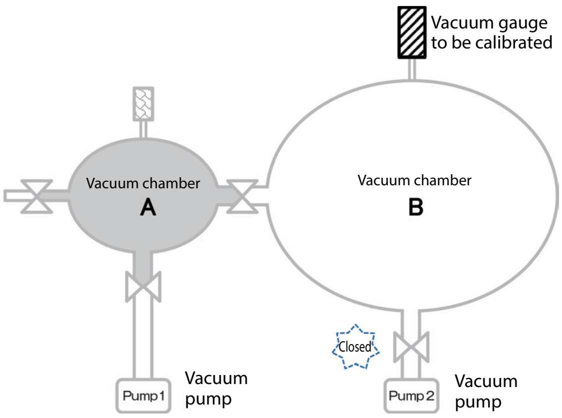

Fig. 4-4. Principle of the expansion method: description (5) in the text

(6) When the valve between the chambers is opened again, the gas that was only inside chamber A fills the evacuated chamber (B) so that both chambers have the same pressure (fig. 4-5). According to Boyle's law, the pressure P_1 is:

$$P_1 = P_0 \frac{V_A}{V_A + V_B} \quad \text{Equation (1)}$$

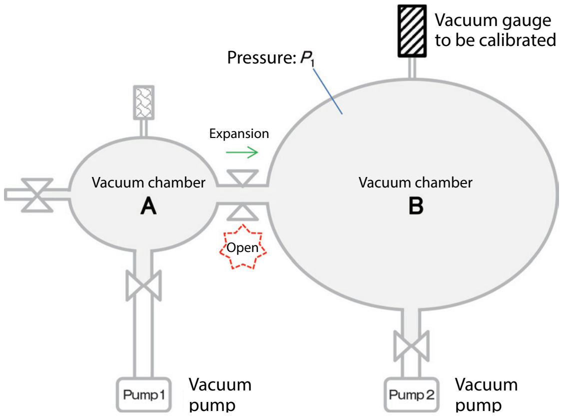

Fig. 4-5. Principle of the expansion method: description (6) in the text

(7) When in this state steps (3) to (6) above are repeated, the final pressure P_2 is:

$$P_2 = P_1 \frac{V_A}{V_A + V_B} = P_0 \left(\frac{V_A}{V_A + V_B} \right)^2 \quad \text{Equation (2)}$$

Similarly, when the steps are repeated n times, the final pressure P_n is:

$$P_n = P_0 \left(\frac{V_A}{V_A + V_B} \right)^n \quad \text{Equation (3)}$$

Here, α in the following is called the expansion ratio.

$$\alpha = \frac{V_A}{V_A + V_B} \quad \text{Equation (4)}$$

(8) The above-mentioned repetition continues until the internal pressure of the chamber reaches the target value.
For instance, if expansion ratio α is 0.1, the desired pressure inside the chamber is 30 Pa, the initial pressure P_0 is 30 kPa, and the number of expansions n is 3, the following can be derived from formulas (3) and (4):

$$P_n = P_0 \alpha^n = 30000 \times 0.1^3 = 30$$

At this point the output value of a vacuum gauge to be calibrated can be measured and then compared with P_n.

In steps (1) to (8), an accurate value for expansion ratio α is necessary. Formula (4) above for the expansion ratio α uses the capacity of the chambers, V_A and V_B. It is difficult, however, to obtain sufficiently accurate values for them. Consequently, the expansion ratio α is derived from equation (5) before calibrating the vacuum gauge. In other words, an expansion is performed in the same way as in steps (1) to (8), and based on the pressures before and after the expansion (P_0 and P_1), the expansion ratio α is obtained as follows:

$$\alpha = \frac{V_A}{V_A + V_B} = \frac{P_1}{P_0} \quad \text{Equation (5)}$$

However, it must be ensured that both P_0 and P_1 are within the range of calibration for the absolute pressure gauge used for initial pressure measurement.

Note that although the apparatus that was actually installed (fig. 3) has three chambers, partly for the purpose of achieving multiple expansion rates, the principle of the system is the same as that described here.

3. Error factors in the expansion method and their reduction

Although the expansion method operates on the principle described above, there are various factors that could lead to error in both the initial pressure and the expansion ratio, so it is necessary to sufficiently reduce the impact of these factors. Conceivable factors mainly include the following:

- Performance of the absolute pressure gauge used for initial pressure measurement
- Deviation between the (real) gas inside the chamber and the (ideal) gas in the equation of state
- The degree of vacuum reached inside the chamber by evacuation
- Leakage and outgassing
- Changes over time and the spatial distribution of temperature in the chamber

When we launched the construction of the vacuum standard, there was no institution in Japan other than AIST that had a calibration apparatus based on the expansion method, so we conducted evaluation and improvement through trial and error. The following provides an overview of the improvements made.

3.1 Performance of the absolute pressure gauge for initial pressure measurement

The performance of the absolute pressure gauge for initial pressure measurement has an impact not only on the initial pressure, but also on deriving the expansion rate. This is because if the linearity of the absolute pressure gauge is not satisfactory, the expansion ratio will vary depending on the pressure that is measured. This is also true with respect to aging over time. Therefore, an absolute pressure gauge with excellent linearity and little long-term change that can measure at high precision over a wide range is needed.

Accordingly, the Measurement Standards Section decided to use Azbil's sapphire diaphragm vacuum gauge in the 100 kPa range. By continuing to experiment with expansions of varying initial pressures, we verified that the pressure dependency of the expansion ratio obtained with this gauge and the change of the expansion ratio over time was sufficiently small.

3.2 Deviation between the real gas and the ideal gas

Section 2 explained that the pressure inside the chamber was obtained by Boyle's law, but this assumes that the gas inside the chamber is an ideal gas. Although the behavior of a real gas closely follows the state equation of an ideal gas in a range where the pressure is sufficiently low, its behavior deviates from that of an ideal gas as the pressure increases. The difference at a pressure of 10 kPa and more, for instance, is no longer negligible. Accordingly, instead of using the measurement of the absolute pressure gauge for initial pressure measurement as when obtaining the expansion rate or the pressure after expansion, the impact due to the deviation between the ideal and real gases is reduced by compensating in the following way.

First, the molar volume V/n is obtained from the pressure in the chamber measured by the absolute pressure gauge, using the van der Waals equation of state, which expresses a real gas.

$$\left(P_r + \frac{a}{\left(\frac{V}{n} \right)^2} \right) \left(\frac{V}{n} - b \right) = RT \quad \text{Equation (6)}$$

P_r : Measured pressure V : Volume
n : Amount of substance R : Gas constant
T : Temperature a and b : Van der Waals' constants

Next, by applying the molar volume V/n to the state equation of an ideal gas, the pressure in the chamber P on the assumption of an ideal gas is used (i.e., Boyle's law is applicable).

$$PV = nRT \quad \text{Equation (7)}$$

3.3 Degree of vacuum reached by evacuation

Although evacuation is done by the process explained in section 2, the absolute pressure inside the chamber will not reach 0 Pa (which is impossible). Since gas on the order of approximately 10^{-6} Pa will remain, that portion must be considered in the calculation.

3.4 Impact of leakage and outgassing

If there is leakage from the chamber, outside air will enter when the chamber is sealed under vacuum, which would be a measurement error factor. Similarly, if gas molecules adhering to the inner wall of the chamber detach (outgassing) when the chamber is sealed under vacuum, the pressure in the chamber will rise. This is another measurement error factor. Therefore, when the apparatus was made the inner wall of the chamber was given a mirror finish. In addition, although attention is paid to the pipe joint (gasket seal) in order to minimize leakage, the impact of a slight leak cannot be ignored if the pressure that needs to be achieved is very small. If this is the case, the pressure after expansion is continuously measured for a certain amount of time to obtain the speed of pressure increase inside the chamber, and the resulting value is deducted.

3.5 The impact of temperature

Change of the temperature inside the chamber over time has an impact on the measurement results. If there is a change in the temperature at each stage of the expansion process described in section 2, it is necessary to consider Charles' law in addition to Boyle's law to obtain the pressure. Namely, if the temperature before the expansion is T_0 and the temperature afterward is T_1, the following equation holds:

$$P_1 = P_0 \frac{V_A}{V_A + V_B} \cdot \frac{T_1}{T_0} \qquad \text{Equation (8)}$$

With regard to the impact of the spatial distribution of the temperature inside the chamber, the chamber itself (other than the vacuum pump section) is covered to protect it from drafts, and an air circulator is used inside the cover to keep the temperature even. (Also, the room where the expansion method apparatus is installed is air conditioned to a certain temperature and humidity.) As a result, the temperature difference between the chambers is no more than 0.05 °C. Additionally, compensation is made for the difference in temperature.

4. Improvement of the calibration system

While the expansion method enables pressure to be generated and measured with very small uncertainty, it also has disadvantages. For example, measurement takes a relatively long time. We therefore made a comparative calibration apparatus (fig. 5) using a sapphire diaphragm vacuum gauge calibrated by the expansion method apparatus as the standard instrument and mounting it and the vacuum gauge to be calibrated on the same chamber. We calibrate with this apparatus by applying the target pressure and comparing the values of both gauges. The standard instrument used for the comparative calibration apparatus is periodically calibrated by the expansion method apparatus. Although the uncertainty with comparative calibration is greater than that of the expansion method, less time is needed for measurement. By choosing between these two methods according to the targeted uncertainty, the Measurement Standards Section has built a system for efficient calibration.

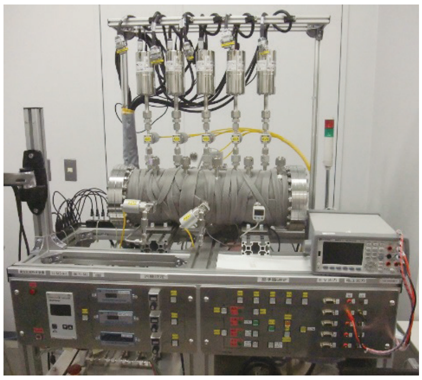

Fig. 5. External appearance of the comparative calibration system

5. Ensuring traceability to a national standard

As described above, we constructed an in-house vacuum standard apparatus for vacuum gauge calibration using an absolute pressure gauge calibrated with a piston pressure balance for initial pressure measurement and then obtaining the expansion ratio α of the expansion method. We periodically ask AIST to calibrate the piston balance, since it is a designated secondary standard instrument. Its minimum supply pressure value is about 10 kPa. Starting with this standard instrument, expansion is repeated with the expansion method apparatus to calibrate pressures such as 100 Pa and 10 Pa. Therefore, the correctness of its measurement performance must be evaluated objectively.

Accordingly, we decided to evaluate the performance of our vacuum standard by measuring the same vacuum gauge (a 133.32 Pa range diaphragm vacuum gauge) with our vacuum standard and with AIST's national standard and then comparing the results (a proficiency test).

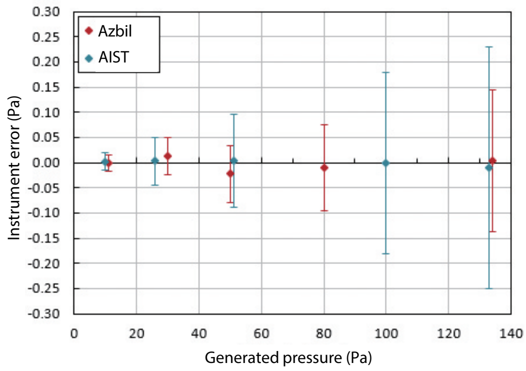

Fig. 6. Instrument error between Measurement Standards Section and AIST measurements of the same diaphragm vacuum gauge

Table 1. The E_n number in the above comparison.

Calibration value [Pa]	E_n number
11	-0.16
30	0.15
50	-0.25
133	0.05

Figure 6 shows the results. The error bars in the graph indicate the expanded uncertainty of the individual results of the Measurement Standards Section and AIST. The degree of overlap of the error bars can be used to determine the conformity of both parties' measurements. From this graph, it can be seen that they have excellent conformity with each other. Table 1 shows the calculated E_n numbers. As the table shows, the difference between the two sets of results is sufficiently small. A similar comparison with AIST was done for our comparative calibration apparatus using the same vacuum gauge as above. The result was that the absolute values of the E_n numbers were 1.0 or less.

Based on the above results, we applied and were registered as a JCSS calibration service provider for pressures from 10 Pa to 133.32 kPa. Table 2 shows our registered calibration and measurement capability (CMC) as of 2021.

Table 2. CMC of the Measurement Standards Section for vacuum gauges

Calibration range	Expanded uncertainty (Confidence level: approx. 95 %) P: Measured pressure [Pa]
10 Pa to 40 Pa	0.20 $P\times10^{-2}$
Over 40 Pa to 133.32 Pa	0.12 $P\times10^{-2}+0.010$ Pa
Over 133.32 Pa to 1333.2 Pa	0.085 $P\times10^{-2}+0.15$ Pa
Over 1333.2 Pa to 13,332 Pa	0.060 $P\times10^{-2}+1.5$ Pa
Over 13332 Pa to 133,320 Pa	0.010 $P\times10^{-2}+10$ Pa

Note that the only types of measurement instruments currently accepted as secondary standard instruments are the piston balance and the spinning rotor gauge.* Figure 7 shows the pressure ranges covered by the standards. As shown in figure 7, there are no secondary standard instruments that cover most of the pressure ranges mostly used by the sapphire diaphragm vacuum gauge, and therefore we cannot directly receive a calibrated standard from AIST.

Consequently, we decided to compare Measurement Standards Section and AIST measurements of the same vacuum gauge (using the spinning rotor vacuum gauge, which is also a means of receiving standards from AIST) at 0.01 to 1 Pa, which is an even lower pressure range than that registered with JCSS.

Figure 8 and Table 3 show the results. They should be interpreted in the same manner as figure 6 and table 1. As can be seen, we found that the difference with the national standard was sufficiently small, particularly at 0.1 Pa and 1 Pa.

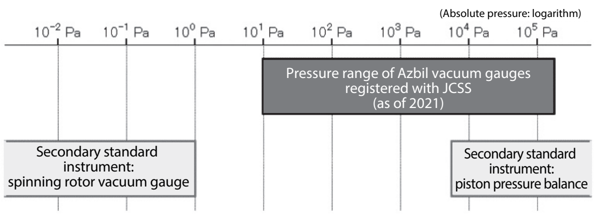

Fig. 7. Pressure range of our Measurement Standards Section's vacuum gauges registered with JCSS (top) and the pressure ranges of secondary standard instruments calibrated by AIST (bottom)

* Also called a viscosity gauge. It is a measurement instrument in which a rotating metal sphere is suspended in midair to obtain the pressure in that space based on the amount of reduction in rotation speed.

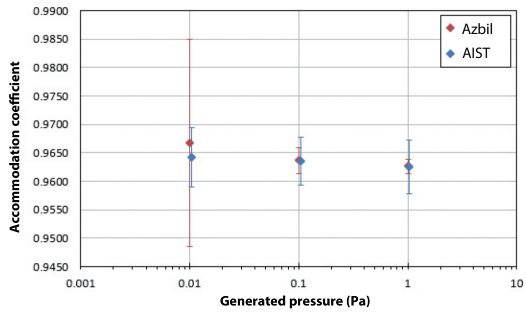

Fig. 8. The accommodation coefficient (ratio of the vacuum gauge output value to the generated pressure value) observed when the same spinning rotor vacuum gauge was measured by the Measurement Standards Section and AIST

Table 3. E_n numbers for the above comparison test.

Calibration value [Pa]	E_n number
0.01	0.16
0.1	0.25
1	0.15

As described above, the results of our calibration were sufficiently consistent with the calibration results of AIST, both on the order of 10 kPa for piston pressure balance calibration, and for spinning rotor calibration results at 0.1 Pa and 1 Pa, for which expansion was repeated for calibration using the expansion method apparatus. If our calculations of the expansion ratio, compensation, etc., were even slightly incorrect, the measurement values in the vacuum range (of the spinning rotor vacuum gauge) reached by repeated expansion would never have matched those of AIST. This consistency further verifies the correctness of our measurements in the ranges for which our vacuum gauges are registered with JCSS, including ranges where we do not directly receive standards calibration from AIST.

We periodically ask AIST to calibrate the spinning rotor vacuum gauge, since it is a secondary standard instrument. Using it along with our piston pressure balance, which of course is also a secondary standard instrument, to ensure traceability, we have constructed a system by which we can continuously evaluate the correctness of the two instruments' performance.

Figure 9 is a diagram of this system.

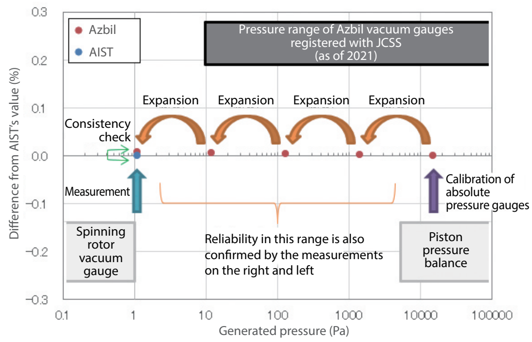

Fig. 9. Verification of measurement reliability in the range of the Measurement Standards Section's vacuum gauges registered with JCSS

Accordingly, we are able to ensure the traceability of our sapphire diaphragm vacuum gauge products in the pressure range from 10 Pa to 133.32 kPa.

6. Conclusion

In order to evaluate the properties and ensure the reliability and traceability of SPG sapphire diaphragm vacuum gauges manufactured and sold by Azbil, we have constructed a vacuum generation apparatus that uses the expansion method as our vacuum standard. We investigated the factors that lead to uncertainty in measurement and reduced their impact. Then, we compared the same vacuum gauges against AIST's values and confirmed the adequacy of our measurement performance. We also improved our calibration system by introducing a mechanism for comparing calibrations, and we became registered with JCSS in the vacuum gauge (measuring instruments) category, which has enabled us to ensure the traceability and reliability of SPG sapphire diaphragm vacuum gauges.

Currently, our JCSS registration in the vacuum gauge (measuring instruments) category is for pressures from 10 Pa to 133.32 kPa, as shown in Table 2. This range covers most of the pressures at which our sapphire diaphragm vacuum gauges will probably be used, namely from the 133.32 Pa range to the 133.32 kPa range. However, increased demand for high vacuum is predicted to continue, mainly in the semiconductor field. Along with market demand, we plan to increase our ranges that are registered with JCSS in order to cover higher vacuum ranges.

References

[1] Sekine, Masashi et al. "Development of the sensor chip and package for a high-temperature sapphire capacitance diaphragm gauge." *azbil Technical Review*, 2011.

[2] Yoshikawa, Yasuhide et al. "Development of a high-temperature sapphire capacitance diaphragm gauge." *azbil Technical Review*, 2011.

[3] Ishihara, Takuya et al. "Extending the measurement range of a sapphire diaphragm vacuum gauge to 13 Pa." *azbil Technical Review*, 2018.

Author affiliation

Toshiyuki Kataoka Measurement Standards Section
Technology Standardization Department
Azbil Corporation