1. INTRODUCTION

Development of Pressure Transfer Standard Using a Silicon Resonant Sensor

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Abstract: With the globalization of the economy, production and commerce, reliability of the measurement results for measuring instruments becomes more important in the international trade. In pressure measurement field, many interlaboratory comparisons are carried out among National Metrology Institutes (NMIs) using a pressure transfer standard to verify their degrees of equivalence. Since 1994, YOKOGAWA has been producing a series of digital manometers using a silicon resonant sensor developed independently. YOKOGAWA's silicon resonant sensor has an excellent long-term stability so that it has been adopted as the pressure transfer standard by many NMIs and has been well received. The pressure transfer standard is called Resonant Silicon Gauge (RSG) among NMIs. From December 2016, the National Metrology Institute of Japan (NMIJ), Advanced Industrial Science and Technology (AIST) and the YOKOGAWA started a collaborative research with the aim of establishing evaluation technology and improving the characteristics of the pressure sensors and are developing a portable transfer standard using a new silicon resonant sensor. The various characteristics such as the measurement range, linearity, hysteresis, short-term and long-term stability, etc. depend on YOKOGAWA’s sensor technology. On the other hand, the evaluation of the characteristics and the uncertainty evaluation of the calibration values depend on NMIJ/AIST’s calibration techniques, who maintains the national pressure standards and carry out various calibration works on a daily basis. In this paper, we report the results of the improvement in the characteristics of the pressure transfer standard by YOKOGAWA based on NMIJ/AIST’s

evaluation technique.

1. transfer standard, international comparison, digital manometer, silicon resonant sensor,

resonant silicon gauge

The conclusion of the Mutual Recognition Arrange­ment of the Comité International des Poids et Mesures (CIPM-MRA) has promoted barrier-free trade in the globalized economy. The CIPM-MRA provides for the mutual recognition of the equivalence of national standards, and the equivalence is ensured by inter­national comparison among National Metrology Institutes (NMIs) [1]. The international comparison is commonly carried out with a transfer standard, which is circulated to NMIs in each country. NMIs calibrate the transfer standard in accordance with their own national standards. The calibration results are compared among NMIs participating in the international comparison and the equivalence of national standards is confirmed. In the international comparison of pressure measurement, a pressure balance was mainly used as a transfer standard. However, a pressure balance is large, heavy and very precise. It was not easy to transfer the instrument as the transfer standard from one country to another. NMIs including the National Metrology Institute of Japan (NMIJ), Advanced Industrial Science and Technology (AIST) took notice of a digital manometer that is smaller, lighter, more excellent in portability and easier to handle than the pressure balance. Since the international comparison usually takes a year or longer, the transfer standard is required to have high stability. Although the digital manometer provides less stability than the pressure balance, NMIs have established a method to compensate for the shortage. The reliability of transfer standard was ensured by redundancy with using multiple digital manometers. The long­­­-­term stability of digital manometer was compensated by correcting calibration values using the result of long­­- term stability evaluation [2]. These methods have made it possible to use the digital manometer for the transfer standard and a number of international comparisons using these transfer standards have been conducted.

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YOKOGAWA's digital manometers and pressure sensors have been used for such transfer standards for international comparisons [3-5]. Pressure sensor is a digital manometer without a display and operation keys. YOKOGAWA provides NMIs with pressure sensors, such as 1 kPa differential pressure, 10 kPa differential pressure and 100 kPa absolute pressure models under the model name 265381/Z, and those have earned a good reputation. Also, the 265381/Z is equipped with YOKOGAWA's silicon resonant sensor [6, 7] and this type of manometer is called Resonant Silicon Gauge (RSG) by NMIs. Since NMIs requested that the supply of 265381/Z be continued, we have started developing a successor RSG. The sensor structure of the new RSG is different from that of the conventional 265381/Z, which requires the evaluation of basic performance (linearity, hysteresis and repeatability). For that, YOKOGAWA started a collaborative research with NMIJ/AIST, which possesses national standards and high evaluation technique, to work on the basic performance confir­mation and characteristics improvement of the new RSG.

2. silicon resonant sensor

YOKOGAWA’s digital manometers are equipped with the silicon resonant sensor developed by YOKOGAWA and deemed as RSG. This section gives an overview of this silicon resonant sensor.

The diaphragm, resonator and vacuum chamber covering the resonator that constitute the silicon resonant sensor are all formed of single crystal silicon by semiconductor process technology. Two resonators are placed at positions where tension and compressive forces are generated on the diaphragm. The resonators are excited by a magnetic circuit. As they are in the vacuum chamber, vibration attenuation is　suppressed and a high Q factor can be obtained. When the silicon diaphragm is deformed by application of pressure, the resonators are distorted and their resonant frequencies change. The frequencies of the two resonators are detected and the difference is converted into a pressure value. Also, the RSG output value is corrected using correction factors calculated from the relationship between the applied pressure and the frequencies of the resonators. The silicon resonant sensor has excellent reproducibility and little secular change because its pressure detection part is formed of single crystal silicon, which is an elastic material. This characteristic contributes to the achievement of high stability.

20 ppm

Fig. 1 Calibration results of the 10 kPa RSG #A from 2014 to 2016.

Fig. 1 shows the calibration results of 10 kPa differential pressure RSG which is equipped with YOKOGAWA’s silicon resonant sensor (10 kPa RSG) by comparison to YOKOGAWA's pressure standards. The range of calibration values in 2 years was within 0.06 Pa, and the long-term stability required for a transfer standard showed good results. On the other hand, the inflection points were observed on the calibration curves [8].　YOKOGAWA 10 kPa RSG, which has been used for a transfer standard, has the same tendency. Stability and hysteresis, which are elements of the basic performance, are determined by the structure of sensor, such as the detection part of silicon resonant sensor and the diaphragm of pressure detector section. In contrast, the linearity can be improved with the adjustment method. Therefore, we have newly developed an RSG and worked on the basic performance confirmation and linearity improvement by adjustment.

3. Collaborative research with nmij/aist

We aim to develop an RSG that can be used for a transfer standard for international comparison and that can be continuously supplied. In order to confirm the basic performance of the RSG, development using national standards is necessary. YOKOGAWA started a collaborative research with NMIJ/AIST in 2016. The collaborative research has enabled evaluation by national standards.

An RSG traceable to national standards or an RSG directly calibrated by the national standards was used as the standard device for adjustment. The RSG measured value was corrected with a calibration value and used as a standard value. We repeated simulation with the standard value and the frequency data of resonators and attempted to optimize the pressure points used for the adjustment. The effect of the adjustment was confirmed by directly comparing the adjusted RSG with the national standards. The development was carried out according to the following procedure:

1. YOKOGAWA produces an RSG (referred to as RSG #A).
2. Calibrate the RSG #A (10 kPa by YOKOGAWA, the others by NMIJ/AIST).
3. YOKOGAWA feeds back the result of (2), corrects the output of RSG #A, and use it as the standard device to newly adjust another RSG (referred to as RSG #B).
4. NMIJ/AIST evaluates the RSG #B adjusted in (3) for the confirming the effect of the adjustment.

The following shows the way of adjusting RSGs by YOKOGAWA:

1. Place the RSG to be adjusted in a temperature chamber and the standard device outside the temperature chamber.
2. Apply pressure from a pressure controller to the RSG and the standard device.
3. Obtain the frequencies of the resonators of the RSG and the pressure measurements of the standard device by changing the temperature and pressure.
4. The coefficients of the approximation formula calculated from the acquired data are written to the internal memory of the RSG as a correction factor.

In this research, multiple RSGs were adjusted at the same time. This is the same method as the one used in the manufacturing process of YOKOGAWA digital manometers. Being able to use the existing method to adjust RSGs without requiring a special procedure and without spending a large amount of time and cost is a great benefit for continuous provision of RSGs.



115 mm

Fig. 2 Photograph of the prototype 10 kPa RSG.

Fig. 2 shows the developed prototype of 10 kPa RSG. It has input ports on the front. However, it has no display and operation keys. Acquisitions of pressure values and various settings are performed through USB communication with a PC. A USB port is provided on the rear surface. The external appearance is the same regardless of the pressure range.

In this collaborative research, we developed RSGs of 10 kPa differential pressure, 200 kPa gauge pressure, 1000 kPa gauge pressure, 3500 kPa gauge pressure and 130 kPa absolute pressure. NMIJ/AIST evaluated characteristics of the RSGs such as linearity and stability. The following sections introduce the characteristics of the RSGs obtained by the evaluations.

4. Characteristics of rSGs

4.1 Calibration results and linearity of 10 kPa RSG

The 10 kPa RSG #B was adjusted using the 10 kPa RSG #A shown in Fig. 1 of Section 2 as the standard device.　For the evaluation, calibration was performed by direct comparison to the generated differential pressure using double pressure balances. Ten points of measure­ment pressure were selected in the range of 1 Pa to 10 kPa and the line pressure was 100 kPa. The 'ABABA’ method [9] was used for the sequence of differential pressure generation. Measurement was performed during pressure ascending processes. The pressure was ascended five times and measurement was carried out once for each. The average of the measured values was used as the calibration pressure value.

5 ppm

Fig. 3 Calibration results of 10 kPa RSG #B1 and #B2. The error bars refer to expanded (*k* = 2) uncertainties.

Fig. 3 shows the calibration results of two units of RSG #B. Calibration data in the figures hereafter except in Fig.8 were provided by NMIJ/AIST. These two units were calibrated at the same time. The maximum deviation to the NMIJ/AIST pressure standards was 0.07 Pa. The linearity of the RSG #B was greatly improved compared with the RSG #A. The maximum difference between the two units was 0.03 Pa. We confirmed that this　difference could be obtained by simultaneously adjusting multiple RSGs in combination with the pressure controller and standard device. It is possible to supply RSGs having small variations in characteristics when multiple RSGs are required to improve the reliability of transfer standard.

2 ppm

Fig. 4 Linearity deviations from a regression line of 10 kPa RSG #B and #C. The RSG #C was adjusted using the RSG #B as standard.

The RSG #B was adjusted using the RSG #A calibrated by YOKOGAWA as the standard device. Although the RSG #A was traceable to national standards, it was deep in the hierarchy from the national standards. Therefore, the RSG #C was adjusted using the RSG #B calibrated by the national standards as the standard device. The calibration results of RSG #B and #C were compared with the regression lines and the linearity comparisons were made. Fig. 4 illustrates the results of linearity comparison. The RSG #C shows further improved linearity compared with the RSG #B.

4.2 Short-term and long-term stability of 10kPa RSG

2 ppm

Fig. 5 Short-term stability of the 10 kPa RSG #B.

Fig. 5 shows the results of the measurement during pressure ascending processes. The pressure was ascended five times and measurement was carried out once for each. This confirmed the short-term stability. Even in the measurement results at 10 kPa, which had the largest variation, the standard deviation was 0.028 Pa, which was 3 ppm or less with respect to the full scale.

2 ppm

Fig. 6 Long-term stability of the 10 kPa RSG #B.

Fig. 6 shows the results of two calibrations performed 10 months apart to confirm the long-term stability. The calibration results coincide within 0.04 Pa. It was confirmed that the stability was excellent both in the short term and in the long term with the evaluation by NMIJ/AIST, where the procedure was highly controlled.

4.3 Influence of line pressure and temperature of 10 kPa RSG

While the line pressure of a differential pressure gauge at the time of calibration is usually 100 kPa, it may be used with the line pressure set to vacuum in international comparison of absolute pressure ranges [4]. In this case, as the line pressure differs from that at the time of calibration by 100 kPa, it is necessary to check the influence of changes in line pressure in advance [8]. The RSG #B was calibrated with line pressures of 75 kPa, 100 kPa and 125 kPa, and the influence per line pressure of 100 kPa was estimated from the calibration results.

10 ppm

Fig. 7 Influence of the line pressure on calibration results of 10 kPa RSG #B. Change in calibration results per 100 kPa of line pressure.

Fig. 7 shows the change in calibration results when the line pressure changed by 100 kPa. A change in the span was observed, and the amount of change in the calibration results was 0.6 Pa at 10 kPa. The RSG #B was adjusted so that the zero point was not affected by the line pressure, but the span was not adjusted. There is a possibility that the line pressure dependence of span can be corrected by changing the line pressure and acquiring span data, then including it in adjustment parameters. Since the characteristics are linear, it may also be possible to make corrections using regression lines. These should be examined in future research.

There is a transfer standard using a temperature­controlled enclosure in order to reduce the influence of ambient temperature [4]. If the temperature characteristics of an RSG that is incorporated in the transfer standard are improved, the influence of temperature can be minimized with the use of enclosure, or there is a possibility that the enclosure can be omitted. The correction factor of RSG #B was calculated using the adjustment parameters of RSG #B that were obtained in the range of -10 °C to 50 °C.

20 ppm

Fig. 8 Influence of temperature variation on output of the 10 kPa RSG #B.

Fig. 8 shows the results of the measurement of input-output characteristics at some temperatures by YOKOGAWA, with deviations from 23 °C. In the range of -10 °C to 50 °C, the maximum temperature coefficient was 0.03 Pa/°C. Although the adjustment range was set to -10 °C to 50 °C in this research, there is no such temperature change in the environment of the international comparison [4]. It is considered possible to reduce the influence of temperature by optimizing the adjustment temperature range.

4.4 Characteristics of other pressure range RSGs

The international comparison of pressure measure­ment is performed over a wide pressure range [10]. This section reports the characteristics of the 130 kPa absolute pressure RSG (130 kPa abs RSG) and the 3500 kPa gauge pressure RSG (3500 kPa RSG), which were developed in consideration of the expansion of RSGs into other ranges than 10 kPa. An adjustment was made using a pressure controller and a standard device following the procedure used for the 10 kPa RSG. The RSG #A calibrated by NMIJ/AIST was used as the standard device. For the evaluation of the adjusted RSG #B, calibration was performed by direct comparison with the NMIJ/AIST pressure balance. The measure­ments were carried out in each of three calibration cycles with pressure ascending and descending. The average of the measured values was used as the calibration pressure value.

(a)

38.5 ppm

(b)

38.5 ppm



Fig. 9 Calibration results of 130 kPa abs RSG (a) #A and (b) #B. The error bars refer to expanded (*k* = 2) uncertainties.

RSG

#B

14.3 ppm

RSG

#A

Ascend.

Descend.

Calib. curve

Fig. 10 Calibration results of 3500 kPa RSG #A and #B. The error bars refer to expanded (*k* = 2) uncertainties.

Figs. 9 and 10 show the calibration results of the standard device RSG #A and the adjusted device RSG #B. The deviation from the national standards could be kept within 10 ppm of full scale. Particularly, a large effect was seen in improving the linearity of 130 kPa abs RSG. This shows that the method using a standard device calibrated by the national standards for adjustment is effective for RSGs of various pressure ranges.

(a)

3.8 ppm

Ascending

Descending

(b)

2.9 ppm

Ascending

Descending

1 cy.

2 cy.

3 cy.

Fig. 11 Short-term stability of (a) the 130 kPa abs RSG #B and (b) the 3500 kPa RSG #B.

Fig. 11 shows the input-output characteristics in three cycles for the 130 kPa abs RSG #B and the 3500 kPa RSG #B. The short-term stability was confirmed. The standard deviations of the three measurements were 0.5 Pa and 4.0 Pa, respectively, and the relative value of standard deviation with respect to the full scale was equivalent to that of the 10 kPa RSG. We confirmed that the reproducibility of repeated measurement was excellent.

5. conclusion

Through the collaborative research with NMIJ/AIST, we attempted to improve the characteristics of RSGs used for the transfer standards for international comparison. The developed RSGs were evaluated by NMIJ/AIST by direct comparison with the national standards. The method in which the standard device was corrected by the calibration value and used for adjustment was used to improve the linearity.

For the 10 kPa RSG, as shown in subsection 4.1, the linearity with respect to the national standards has been greatly improved and adjustment with small difference between the two units is possible. In subsection 4.2, its excellent stability was confirmed in both the short term and long term. The performance of the transfer standard is expected to be improved by replacing the conventional RSG with this new RSG.　Also, the line pressure and the temperature dependence described in subsection 4.3 are expected to improve by optimizing the adjustment conditions. This should be examined and evaluated in future research.

In subsection 4.4, the adjustment method proved to be effective by the evaluation of the 10 kPa RSG was applied to the RSGs of other ranges. The deviation within 10 ppm of full scale with respect to the national standards has been achieved by making the adjustment using the standard device directly calibrated by the national standards. We believe it is possible to use RSGs even in international comparisons over a wide pressure range.

The transfer standard using a digital manometer has improved the efficiency of international comparison of pressure measurement. The use of digital manometers in international comparison is expected to increase in the future. For that, digital manometers that are compact, lightweight and having excellent characteristics are indispensable, and YOKOGAWA has the accumulated RSG technology that can achieve such digital manometers. We will continue to contribute to the advancement of pressure measurement in Japan and overseas through development including RSG characteristics improve­ment and range expansion.

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