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Pressure measurement from 1 atm to 0.01 Pa using a quartz oscillator

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The quartz friction vacuum gauge is based on change in the resonance impedance of the quartz oscillator for a wrist watch with ambient gas pressure. Thermal change and fluctuation of the resonance impedance limit the lowest measurable pressure to 1 Pa for the practical gauges and to 0.1 Pa under well-controlled experimental conditions. In this study, nitrogen gas pressures down to 0.01 Pa were measured with an error of about 10% by adopting the quartz oscillator designed to detect the temperature as a function of the resonance frequency as the sensor.

1. Introduction

After the present authors reported pressure dependence of resonance electric impedance of the quartz oscillator for a wrist watch and theoretically explained the dependence on a simple model[1], vacuum gauges using the oscillator as pressure sensors have been designed and tested[2–4]. Performance of the first gauge was proved to be suitable for pressure monitoring of the range from 1 atm to 1 Pa[2]. The oscillator adopted was in the shape of a tuning fork of about 5 mm in length and its intrinsic impedance Z_o, defined as the resonance impedance Z measured at pressures less than 1×10^{-3} Pa, was about 10 kΩ. In the case of nitrogen gas, the increment of the impedance, denoted as ΔZ $(=Z-Z_o)$, was around 100 kΩ at 10^5 Pa and was proportional to gas pressure at the rate of about 100 Ω/Pa for pressures below 50 Pa. For this gauge, the error in the measurement of ΔZ was around 10 Ω which corresponds to 0.1 Pa. The main origin of the error was revealed to be thermal variation of Z_o. And the measured thermal variation of Z_o was about 100 Ω in the temperature range from 5 to 50°C. For reducing this effect and for measuring pressures of the order of 0.1 Pa, a temperature stabilized gauge head was successfully used for the following gauges[3,4].

In this work, the error in pressure measurement was lowered to about 1×10^{-3} Pa by introducing the tuning fork oscillator designed as a thermometer.

2. Experimental

The tuning fork shaped oscillator utilized in this study is designed to have linear thermal dependence of the resonance frequency. The oscillator, to be called T-oscillator in this report, is schematically shown in Figure 1. The configuration is almost the same as that of the watch oscillator (W-oscillator), which is designed to have a constant resonance frequency of 32,768 Hz for a wide temperature range. Only the relations of crystallographic orientation of the quartz crystal and the T-oscillator configurations differ from that of the W-oscillator. The angle, θ, defined as the angle between the longitudinal direction of the oscillator and the z-axis of the crystal, as sketched in Figure 1, is around $+5°$ for W-oscillators, whereas it is selected in the range of -15 and $-30°$ for the T-oscillator.

The circuit diagram used for measuring characteristics of the oscillator and the performance of the friction vacuum gauge is shown in Figure 4. Measurement and control of temperature of the oscillator installed on a copper rod in the gauge head was performed by using an 8-bit microprocessor to which a thermocouple and a Peltie device are connected.

The pressure of the vacuum chamber used for studying the gauge performance was measured by the controller and two

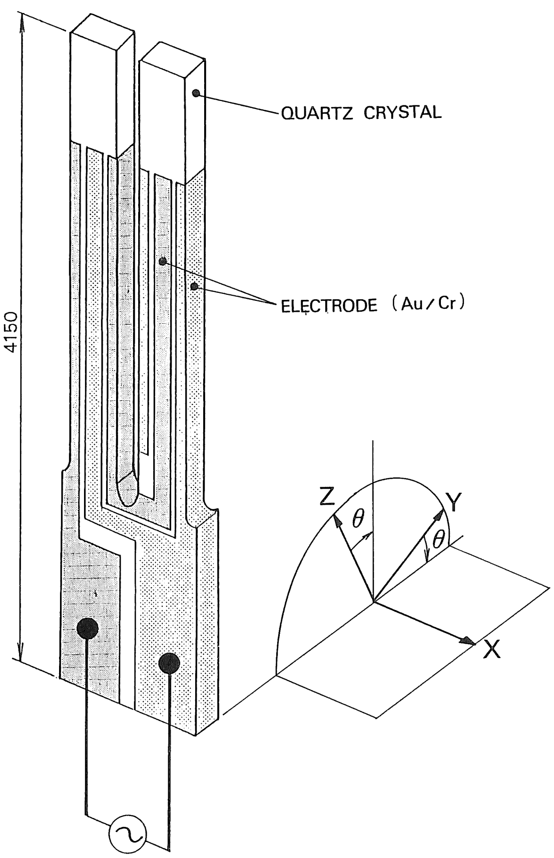

Figure 1. Thermometer oscillator. The crystal axis (x, y, z) of the quartz crystal relative to the oscillator configuration are shown as the inset on the right.

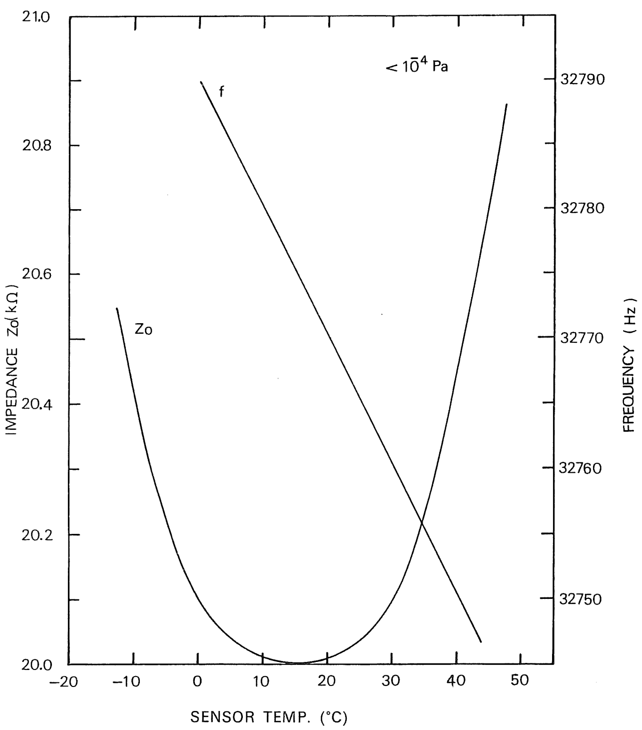

Figure 2. Intrinsic impedance Z_o and resonance frequency vs temperature Z_0 and the frequency f of the oscillator were measured at a pressure less than 1×10^{-4} Pa.

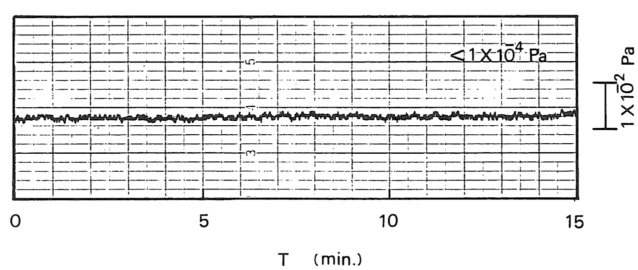

Figure 3. Fluctuation of Z_0 at constant temperature. The measurement was performed under a pressure less than 1×10^{-5} Pa at 20 ± 0.1°C.

heads of the diaphragm manometer (MKS, Baratoron 390HA) with an error less than a few per cent above 1×10^{-2} Pa, and more than that in the 10^{-3} Pa region.

3. Experimental results

The relation of the resonance frequency f of the T-oscillator and temperature T was measured to be in linear relation with the slope of 1 Hz/°C as shown in Figure 2. The differences in the slopes among 20 oscillators are less than 1%. In the range from 5 to 50°C, it is confirmed that the temperature of the oscillator can be estimated with an error less than 0.1°C, by counting the square wave output of the oscillator for 1 s.

The intrinsic impedance Z_o was calculated and recorded by the microprocessor from the current and voltage of the oscillator under pressure less than 1×10^{-4} Pa. The recorded values are plotted in Figure 2 vs the temperature estimated by the counted frequency and the f–T relation described above.

Variation of Z_o of the oscillator at 20 ± 0.1°C under 1×10^{-4} Pa was recorded as shown in Figure 3. The span of the vertical axis is 8 Ω and that of the horizontal axis is 15 min. The measured fluctuation was estimated to be less than 2×10^{-5} Pa from the ratio of ΔZ to pressure, namely 200 Ω/Pa for the data in Figure 5.

Then the resonance impedance Z of the T-oscillator at 20°C was measured for nitrogen gas pressure from 10^{-3} to 10^5 Pa. The values of ΔZ plotted in Figure 5 were calculated from the measured Z and Z_o, which was estimated from the relation between Z_o and temperature T deduced from the resonance frequency. The figure clearly shows that measurement of pressure from 10^{-2} to 10^5 Pa is possible for nitrogen gas. The dashed line in the figure is given by least mean square fitting of coeffcients of theoretical formula[5] with experimental values of ΔZ above 0.1 Pa.

For the examination of the absolute accuracy of the pressure measurement by the T-oscillator for nitrogen gas, the microprocessor with the calibration table corresponding to the data, as shown in Figure 5, stored in its memory was used to calculate the pressure from measured value of ΔZ. And the estimated pressure was plotted against the pressure measured by the diaphragm gauge in Figures 6 and 7. The pressure of the horizontal

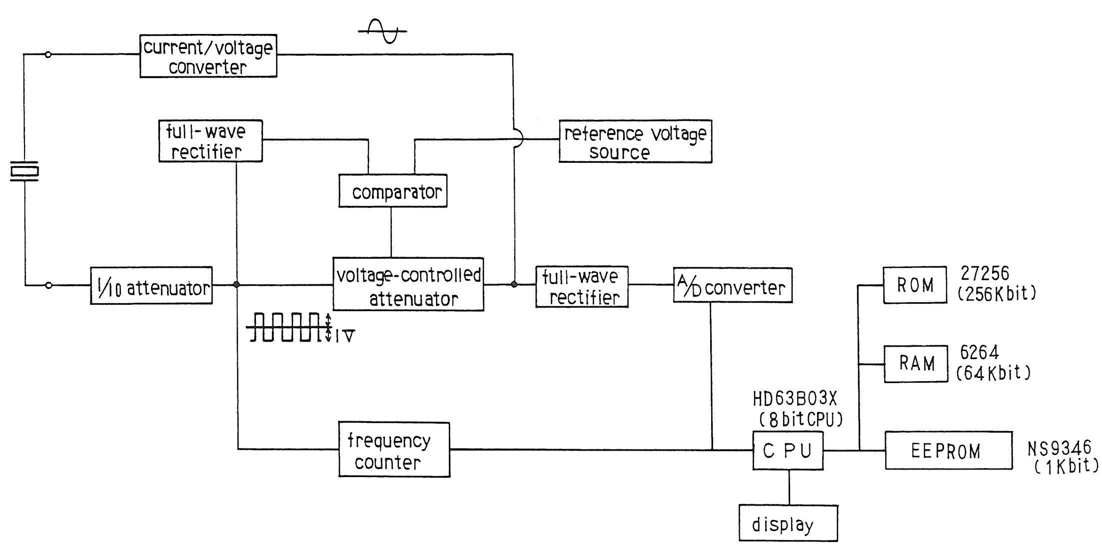

Figure 4. Circuit diagram of the quartz friction gauge.

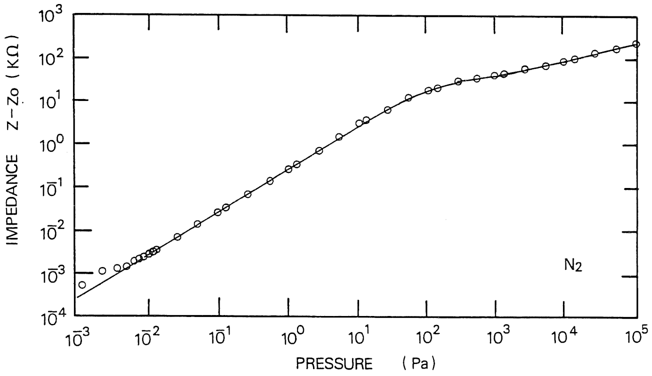

Figure 5. Pressure dependence of the impedance increment $\Delta Z(Z-Z_o)$. The dashed curve is theoretically fitted to experimental data.

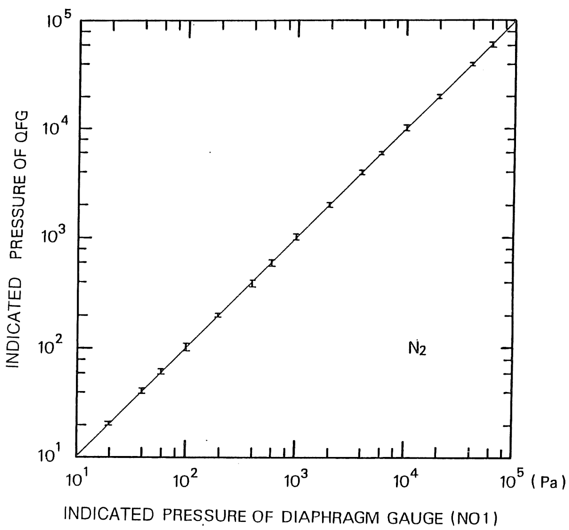

Figure 6. Comparison of the QF gauge and a diaphragm gauge (10–10^5 Pa). The pressure of the horizontal axis was measured by the diaphragm gauge with the 1000 Torr head.

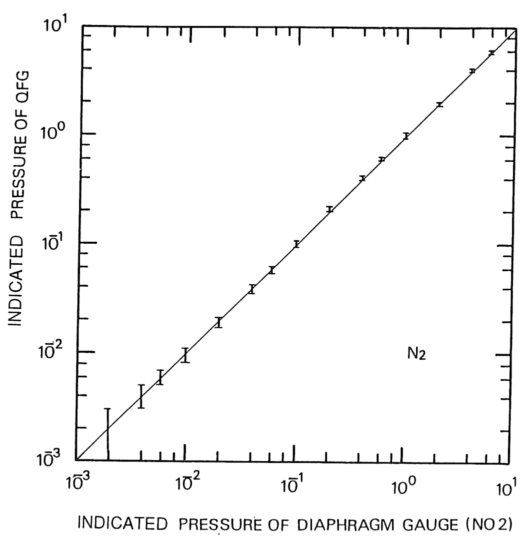

Figure 7. Comparison of the QF gauge and a diaphragm gauge (10^{-3}–10 Pa). The pressure of the horizontal axis was measured by the diaphragm gauge with the 1 Torr head.

axis of the former figure was measured by the 1000 Torr gauge head, and that of the latter figure was given by the 1 Torr gauge head. Each of the error bars corresponds to the data spread for 10 measurements.

4. Discussion

The T-oscillator introduced in this work made it possible to measure the temperature of the oscillator with enough accuracy to estimate thermal variation of Z_o as shown in Figure 3. The possible causes of the variation corresponding to energy loss from the oscillator are mechanical friction in the oscillator, internal friction in the thin film electrode on the oscillator, and/or energy dissipation due to mechanical transmission of the vibration through the supporting structure of the oscillator. The shape of the curve of the variation suggests correlation with the thermal variation of the resonant frequency which shows good agreement with theoretical analysis; however, there is no theoretical attempt to reveal this variation at present. Instead, the experimental data are reproduced more accurately than in previous study[3]. By using digitally recorded data corresponding to the curve in Figure 2, we can estimate Z_o with an error less than 1Ω at temperatures from 5 to 25°C, less than 0.5 Ω for 8–23°C, and 0.2 Ω for 13–20°C. In the last temperature range, the deviation of the values of Z_o estimated from the pressure dependence of ΔZ shown in Figure 5 is equivalent to about 1×10^{-3} Pa. And the typical fluctuation of Z at the temperature of 20 ± 0.1°C is also equivalent to 1×10^{-3} Pa as shown in Figure 4.

Therefore, the relation of ΔZ ($=Z-Z_o$) to nitrogen pressure P as shown in Figure 5 is supposed to give a reliable basis for calibrating the quartz friction gauge for pressures above 1×10^{-2} Pa. The dashed curve represents the theoretical formula given by the authors[5] after the least mean fitting to the experimental data above 1×10^{2} Pa. The agreement between the theoretical curve and the experimental data enables us to estimate the pressure from the measured values of Z and f of the T-oscillator with an error of less than 10% above 1×10^{-2} Pa. This is confirmed by comparison with the measurement by diaphragm gauges as shown in Figures 6 and 7.

The deviation of the plots in the range of 10^{-3} Pa in Figures 5 and 7 is left for future study, since it might be related to the error in pressure measurement by the diaphragm gauge.

5. Concluding remarks

In this study for lowering the minimum detectable pressure of the quartz friction gauge, we adopted a thermometer type quartz oscillator of tuning fork shape and digital circuits to calculate the pressure by measuring the frequency and resonance impedance of the oscillator.

By measuring the resonance frequency of the oscillator, its temperature T was determined with an error less than 0.1°C. Therefore, it was possible to obtain the accurate relation between Z_o and T. Based on this relation, Z_o was estimated with an error of about 0.2 Ω which is equivalent to 1×10^{-3} Pa for nitrogen gas. The observed fluctuation of Z_o was less than 0.2 Ω. Therefore, pressures down to 0.01 Pa were determined with an error of about 10% for nitrogen.

The results obtained in this work give the technical basis of a practical quartz friction vacuum gauge with a miniature gauge head, very low power consumption, and accuracy better than 10% in the pressures range from 1 atm to 0.1 Pa.

Acknowledgement

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Gas concentration analysis with a quartz friction vacuum gauge

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The quartz friction vacuum gauge is based on pressure dependence of the resonance electric impedance of a miniature quartz oscillator of tuning fork shape. The results of the theoretical and the experimental studies clarified the properties of the gauge for various pure gases in the pressure range 10^{-3} Pa to 1 atm.

An algorithm, introduced in this study for calculating the pressure as a function of the impedance, improved the accuracy of pressure readings of the gauge and made it possible to analyze a binary mixture of gases. Pressure dependence of the resonance frequency of the oscillator was adopted as an additional quantity, depending on kinds of gases, and utilized to measure the pressure during purging of a vacuum chamber with hydrogen. An analysis with the gauge of a mixture of argon and helium was tested successfully for practical monitoring of concentration in the pressure range 1 atm to 1 Pa. Copyright © 1996 Elsevier Science Ltd.

Introduction

The quartz oscillator for a wrist-watch has been used as a sensor for the measurement of gas pressure down to 1 Pa[1]. Then a similar oscillator designed specially for temperature measurement was adopted for digital compensation of thermal variation of the intrinsic electric impedance of the oscillator. The oscillator made it possible to measure pressures from 1 atm to 1×10^{-2} Pa with an error around 10%.[2] And the pressure dependence of the impedance measured for several kinds of pure gases showed agreement with the theoretical formula given in the previous study.[3]

As the next approach to an ideal vacuum gauge, we studied the possibility of the quartz friction vacuum gauge (QFG) to discriminate kinds of pure gases and to analyze concentration of a binary gas mixture. For this purpose, an algorithm was devised to give an approximation of the pressure as a function of the impedance. The algorithm described in the following section reduced the error in the pressure reading considerably.

Algorithm for calculating the pressure from the impedance

The theoretical analysis based on the string-of-beads model showed that the square of the increment of the resonance electric impedance ΔZ of a bending oscillator is proportional to p^2M/T in the molecular flow region and to $pM\eta$ in the low vacuum region, where p is the pressure of the gas, M is the molecular weight, η is the coefficient of viscosity, and T is the temperature.[4] The measured pressure dependence of the impedance showed agreement with the unified formula of the following expression which was derived from the slip theory and Millikan's empirical expression for the intermediate pressure region:[3]

$$\Delta Z/C = 6\pi\eta' R + 3\pi R^2(2\eta'\rho\omega)^{1/2} \quad (1)$$

where C is a constant, R is the radius of the oscillating ball, ω is the resonance angular frequency of oscillation, and η' is the apparent coefficient of viscosity. And η' is given as follows:

$$ER\eta/\eta' = ER + (\eta/p)\cdot(\pi R_o T/2M)^{1/2} \quad (2)$$

where R_c is a gas constant, E is a numerical constant depending on the shape of the oscillator. The second term on the right side represents the coefficient of slip.

Because of the difficulty in rewriting eqn (1) into a formula suitable for calculating the pressure p from the given value of ΔZ in the whole pressure region, we introduced two formulae.

In the viscous flow region, where $\eta' = \eta$, eqn (1) is written as the following:

$$\Delta Z/C = 6\pi\eta R + 3\pi R^2(2\eta\rho\omega)^{1/2} \quad (3)$$

Then eqn (3) is rewritten into the form $p = F(\Delta Z)$:

$$p = ((\Delta Z - K_1)/K)^2, \quad (4)$$

where

$$K_1 = 6C\pi\eta R \quad (5)$$

and

$$K_2 = 3\pi R^2 C(2\eta\rho\omega)^{1/2} \quad (6)$$

Since,

$$\rho = M/(R_o T). \quad (7)$$

In the pressure regions of the molecular flow and the intermediate flow, the second term on the right side of eqn (1) is much smaller than the first one. The equation is reduced as follows:

$$\Delta Z = 6C\pi R\eta/(1+(\eta/ERP)\cdot(\pi R_o T/2M)^{1/2}) \quad (8)$$

This equation is transformed to the following equation:

$$p = K_3\Delta Z/(1-K_4\Delta Z), \quad (9)$$

where,

$$K_3 = (1/6C\pi R_2 E)\cdot(\pi/2\rho)^{1/2} \quad (10)$$

$$K_4 = 1/6C\pi R\eta. \quad (11)$$

The values of K_1 and K_2 were estimated by fitting to the experimental relation between the pressure and the impedance for H_2, N_2 and Ar in the viscous flow region. The K_1 and K_2 values obtained were proportional to η and $(\eta M)^{1/2}$ respectively, as indicated by eqns (5) and (6). The values of K_3 and K_4 derived from the experimental results for the three gases also showed good agreement with the proportional relations given by eqns (10) and (11), respectively. After experimental estimation for the constants of C, R and E, it is possible to calculate K_1, K_2, K_3 and K_4 for any kind of gas from their viscosity coefficients and molecular weights. However, these values did not bring a smooth connection between eqns (4) and (9).

Then we adjusted the value of K_4, which plays a dominating role in the intermediate flow region,

$$K_3\Delta Z/(1-K_4\Delta Z) = ((\Delta Z-K_1)/K)^2 \quad (12)$$

Following Newton's method, eqn (12) was solved for ΔZ as a cubic polynomial. K_4 was determined to give a multiple root. Then, ΔZ at the connecting point of the two formulae of eqn (4) and eqn (9), was determined as ΔZ_c by Newton's method.

The calculation of K_1, K_2 and K_3 followed by those of K_4 and ΔZ_c were carried out by an 8 bit microprocessor within 1 s for any kind of gas. Then the calculation of the pressure from the impedance was done within 10 ms.

For simplicity, we call the approximation procedure of this work TFA (Two Formulae Approximation), and the adopting of the unified formula of Ref. 3 as SFA (Single Formula Approximation).

The values of pressures calculated by TFA were compared with those estimated by SFA from experimental values of the impedance for nitrogen gas. The experimental apparatus, procedure and conditions were almost the same as those given in the Ref. 2. As shown in Figure 1, the differences between the pressure calculated by TFA and the pressure reading of the diaphram gauge were smaller than those by SFA. In the pressure range 10^{-2}–10^5 Pa, the difference of the pressures by TFA were less than half of those given by SFA. The constants and parameters for the figure were as follows. M: 28.013, η: 1.65×10^{-5} Pa s, T: 300 K, R: 7×10^{-5} m, fo: 32 kHz, E: 1.0, C: 1.5 MΩ, K_1: 18.902, K_2: 10.185, K_3: 0.029002, K_4: 0.021685, Z_c: 35.388 Ω.

For several kinds of gas, the results of TFA showed a similar advantage over SFA. Therefore, gas concentration analysis by a QFG was studied on the basis of TFA in the following section.

Concentration analysis depending on the resonance frequency and the impedance

Pressure dependence of the resonance angular frequency. The pressure dependence of the resonance frequency of the quarts oscillator was revealed and reported in Ref. 5. It was confirmed that the change in the angular frequency $\Delta\omega$ is proportional to the density ρ of the gas. Or described in the following equation as an approximation:

$$\Delta\omega/\omega_o = -\rho/(4\rho_o) \quad (13)$$

where ω_o is the resonance angular frequency, and ρ_o the density of crystallized quartz. The ratio of the density of the gas to that of quartz, ρ/ρ_o, is small even at atmospheric pressure. It is possible to determine the type of gas by measuring the frequency together with the impedance in a low vacuum region. Since the reduction in the frequency is proportional to ρ (or molecular weight M), and the impedance is proportional to the square root of the product of ρ and η at given pressure in the viscous flow region. Figure 2 visualizes the frequency reduction as a function of increment of the impedance at the given pressure for five kinds of gases. On $\Delta Z-\Delta f$ ($=\Delta\omega/2\pi$) plane, the minimum value of the vertical axis was determined as the experimental limit of 10^{-3} Hz, and the maximum value of around 10 Hz corresponding to the frequency reduction for a heavy gas at 1 atm. For each kind of gas, the data points from the lowest left end to the highest

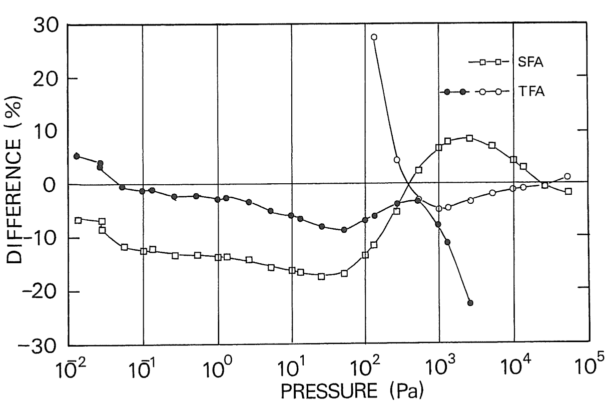

Figure 1. Comparison of pressures estimated by approximation procedure of TFA and SFA. The vertical axis is the difference between the calculated pressure and the actual pressure. The open circles were calculated from the impedance by eqn (4), the filled in circles by eqn (9) and the squares by the unified formula of Ref. 3.

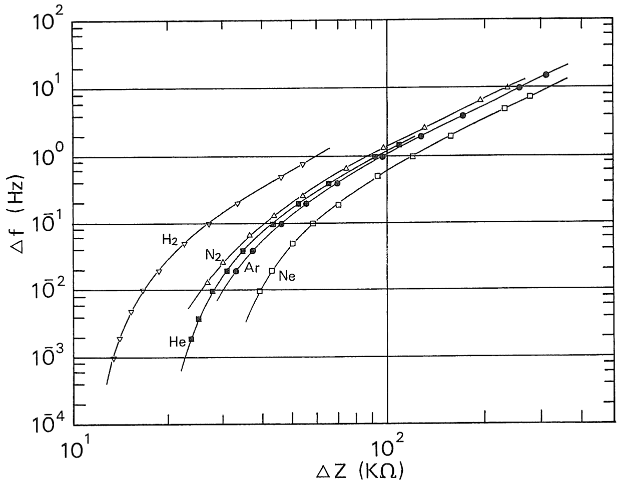

Figure 2. The experimental relationship between the increment of impedance ΔZ and the reduction of the resonance frequency Δf of the oscillator of QFG.

right end corresponded to 133, 267, 667, 1330, 2670, 6670, 13300, 26700, 101300 Pa in order. Each of the curves is monotonic function without any inflection points and does not intersect with other curves. Therefore, the type of a gas and its pressure could be determined by the diagram like Figure 2, if simultaneous measurements of the impedance and the resonance frequency were performed with very high accuracy.

Study on discrimination between nitrogen and hydrogen with a QFG. Figure 2 implied that the discrimination of hydrogen gas from other kinds of gases was possible on the condition that the experimental errors were 0.01 Hz and less than 0.1% of the impedance. So we tested pressure measurement during purging with hydrogen gas.

After a vacuum chamber was evacuated to less than 1×10^{-4} Pa, hydrogen gas was introduced to the chamber. The microprocessor of the controller of a QFG was set to calculate the pressure as pure nitrogen from the impedance. If the frequency was more than a given value, 32 380 Hz in this instance, when ΔZ reaches the given value of 48.5 kΩ (corresponding to about 5×10^4 Pa of H_2 and 10^3 Pa of N_2), the processor was programmed to adopt the parameters for hydrogen. The result was shown as the time chart of Figure 3. The pressure reading of the gauge automatically switched to that for hydrogen at about 1.6 min. Repeated tests carried out for different experimental conditions provided a technical basis for the practical application of this type of switching.

This function of the controller is useful for preventing undesirable over pressure during purging a vacuum chamber with hydrogen gas, with the controller set to air or nitrogen gas.

Concentration analysis with a QFG and a diaphragm gauge

Among the five kinds of gas shown in Figure 2, it seems rather difficult to discriminate between helium and Ar or N_2, since the curves of these gases are close to each other. And it seems more difficult to analyze the concentration of a binary mixture of these gases on the basis of the relation between ΔZ and Δf of a QFG.

For the analysis of the mixture of these gases, we adopted a diaphragm gauge to measure the total pressure and a QFG to gain quantities reflecting the concentration. Binary mixture of Ar and helium was studied in this work.

Experimental procedure. Two vacuum chambers of the same volume of about 0.03 m^3 were used to vary the pressure without changing the concentration. The first chamber equipped with diaphragm gauges and a QFG were connected with a valve to the second chamber which is evacuated by a turbo molecular

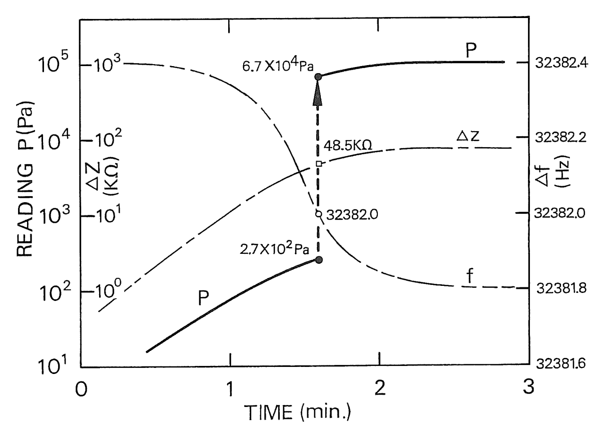

Figure 3. Measurement for purging of a chamber with hydrogen. Variations of the resonance frequency, the impedance of the oscillator and the reading of the gauge were recorded. The reading P was initially shown as nitrogen and automatically changed to the reading as hydrogen at 1.6 min.

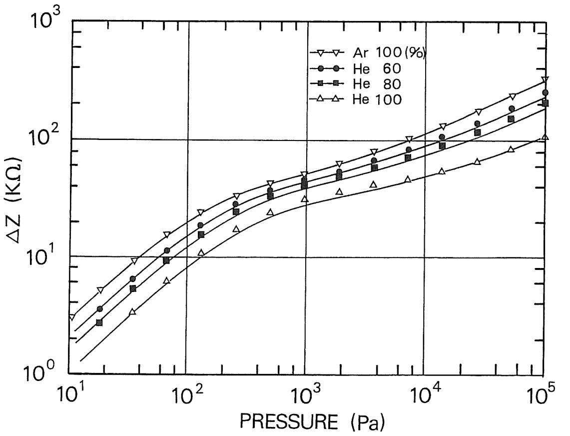

Figure 4. Pressure dependence of impedance measured for Ar, He and their mixture. Measured data were plotted with curves calculated by TFA. Note the good agreement between the measurement and the calculation in the 10 Pa range.

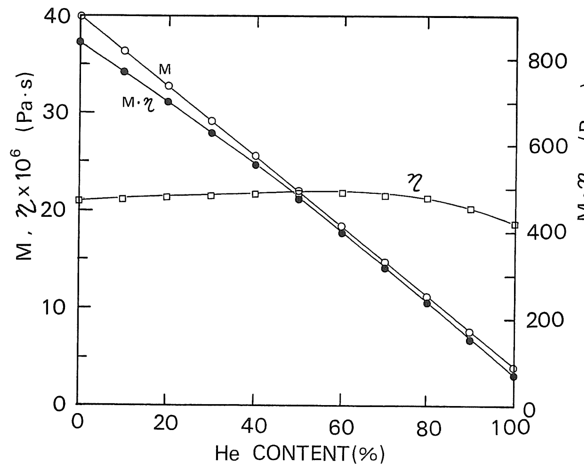

Figure 6. Concentration dependence of the viscosity for the mixture of Ar and He. Viscosity coefficient η, molecular weight and the product of them were shown as a function of the helium content of Ar–He mixture. Note that η is not a linear function of the content.

pump through another valve. After evacuation to 10^{-4} Pa, the later valve was closed and both of the chambers were filled to atmospheric pressure with a gas mixture of argon and helium in a given ratio. Then the pressure of the first chamber was halved by successive handling of the both valves. By repeating this procedure, the pressure was lowered stepwise to 1 Pa, the impedance of the oscillator and the reading of the QFG were recorded with the pressure readings of the diaphragm gauges.

Result and discussion. In Figure 4, the experimental data of the impedance were plotted with curves calculated by following the TFA for gases with 0%, 30%, 60%, 80% and 100% of helium. The differences between the experiment and the calculation were less than 20%.

For a more detailed study, the readings of the QFG set to Ar were plotted in Figure 5 as the ratio to the reading of pure Ar at the corresponding pressure. For comparison, the pressure dependence of the impedance for gas mixtures were calculated by TFA, and converted to the readings of the gauge set for pure Ar, and shown as the curves in Figure 5. The kink on each of the curves corresponded to the connecting point between the two formulae of eqns (4) and (9). The kink was because TFA was not designed to realize analytic continuation at the connection.

These normalized readings and curves showed a monotonic decrease with increase in helium concentration at pressures from 1 to 10^5 Pa. This characteristic corresponded to almost linear reduction of ηM (or $\eta \rho$) with an increase in the helium content, as shown in Figure 6. The values of η were given in Ref. 6.

The standard deviations for the readings of QFG were larger for the lower pressure. They were less than 5% at 10 Pa and about 10% at 1 Pa. It was considered that the reproducibility in the concentration analysis of a binary gas mixture, such as helium and argon, is around 10%. It is necessary to devise an approximation procedure better than TFA for realizing the same order of accuracy in concentration analysis especially in the intermediate flow region.

Judging from the good correspondence of pressure readings with the calculated value in the left end of the figure, it is practical, in the pressure region from 1 atm to 1 Pa, to utilize a QFG with a diaphragm gauge to monitor the concentration of a binary mixture of gases having large difference of ηM values. We are now planning to study on analyses of binary mixtures of gases having the same molecular weight and different viscosity coefficients. For example, the ratios of viscosity coefficients of gas pairs considered are 16.6/9.1 for N_2:C_2H_2, or CO:C_2H_2 and 17.8/8.5 for NO:C_2H_6.

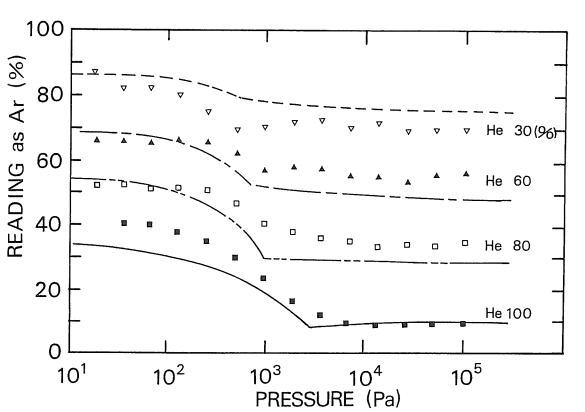

Figure 5. Measured and calculated pressure readings as Ar for He and mixtures with Ar. Data points were obtained as the readings for the gauge controller set for Ar, and the curves were converted to the readings for Ar from the pressure dependence of the impedance calculated by TFA.

Concluding remarks

It was concluded that discrimination between hydrogen and the heavier gases, such as N_2, Ar and CO_2 is possible by measuring the impedance and the frequency of the oscillator of a quartz friction gauge. A QFG used with a total pressure gauge can be applied to monitoring of the concentration of a binary mixture of gases with the reproducibility of about 10% in the pressure range 1 Pa - 1 atm for certain combinations of gases.

It was deduced that there is a room for increasing accuracy in the monitoring by the improvement of the algorithm for calculating pressure from the impedance.

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