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VOLUME DETERMINATION OF A VACUUM VESSEL BY PRESSURE RISE METHOD

Janez Setina[1], *Bojan Erjavec*[2]

[1] Institute of Metals and Technology, Ljubljana, Slovenia (janez.setina@imt.si)
[2] Institute of Metals and Technology, Ljubljana, Slovenia (bojan.erjavec@imt.si)

Abstract ─ A new method for determination of volume ratios and also the absolute value of volumes of vacuum vessels is presented. Method is based on a measurement of rate of pressure rise in a vacuum chamber at constant flow of inert gas. An example of measurement of a volume of a chamber with a nominal volume of 7 L is presented. For pressure measurements we have used a spinning rotor gauge (SRG) and He permeation leak was used as a gas source.

Keywords: volume ratio, pressure rise, spinning rotor gauge

1. INTRODUCTION

Several methods have been employed in vacuum metrology for volume determination. Volume ratios can be determined by different gas expansion methods, which were primarily developed for precise determination of expansion ratios in primary static expansion systems [1], [2], [3] and [4]. Absolute volumes of vacuum vessels can be determined by calculation from dimensional measurements or by gravimetric method (by weighing the vessel filled with a liquid of known density).

Vacuum chambers are often composed of parts with complicated geometries (bellows, vacuum valves etc.) for which the precise dimensional measurements are not possible. It is also not desirable or practical to fill the whole vacuum chamber with a liquid for gravimetric measurements. In such cases volume can be determined by gas expansion method as described in [1].

We have developed another method for volume ratio determination which is based on precise pressure rate of rise measurement using spinning rotor gauge.

If a stable flow of inert gas Q (in units Pa×L/s) is introduced into a vacuum vessel of a volume V_1, a constant pressure rate of rise $\mathrm{d}P_1/\mathrm{d}t$ is obtained:

$$\frac{dP_1}{dt} = \dot{P}_1 = \frac{Q}{V_1} \qquad (1)$$

If the same flow is introduced into a vessel of a different volume V_2, another pressure rate of rise $\dot{P}_2$ is obtained. Volume ratio V_1/V_2 can now be calculated as:

$$\frac{V_2}{V_1} = \frac{\dot{P}_1}{\dot{P}_2} = R \qquad (2)$$

By this method we can determine only volume ratios. To determine absolute value of the volume of the vacuum vessel V_1 we can compose volume V_2 by adding to V_1 another chamber of volume V_0, so that:

$$V_2 = V_1 + V_0 \qquad (3)$$

Chamber V_0 can be of a simple shape (for example a cylindrical extension) so that its volume can be easily determined from dimensional measurement or by gravimetric method. By rearranging equation (3) we obtain expression to calculate V_1 from R and V_0:

$$V_1 = \frac{V_0}{(R-1)} \qquad (4)$$

2. EXPERIMENTAL SETUP

Our experimental setup is schematically shown in Fig. 1. Vacuum chamber is ultrahigh vacuum compatible and is of all metal construction (with Conflat flanges).

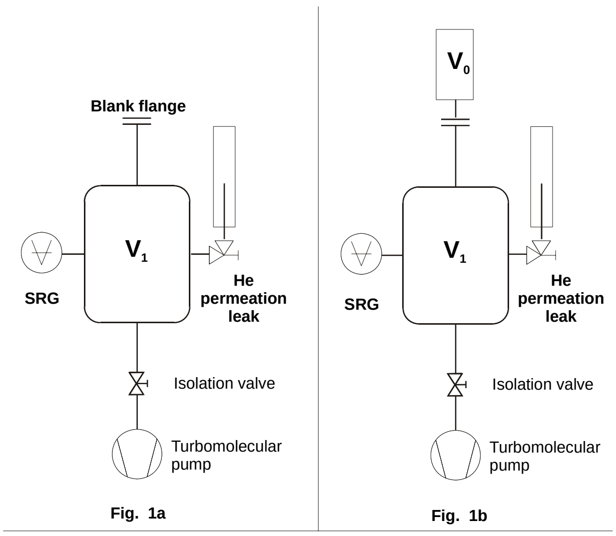

Fig. 1. Experimental setup for volume determination.

All metal vacuum isolation valve is installed between the chamber and a turbomolecular pump. A spinning rotor vacuum gauge (SRG) is attached to the chamber for precise pressure measurements.

As a gas source we have used a helium permeation leak. Short term variations of He flow from such sources are caused only by temperature variations of the permeating element. Therefore it is only necessary to assure a constant temperature of the leak to get the constant gas flow, or to know the temperature coefficient of the gas flow to correct for any deviations of temperature during measurements of pressure rate of rise $\dot{P}_1$ and $\dot{P}_2$. The nominal He flow of the leak at 23°C was 2×10^{-5} Pa×L/s.

2.1. Pressure rate of rise measurements using SRG

SRG has been selected for pressure measurements because it is truly inert vacuum gauge, it exhibits very good linearity and excellent short term stability. It has also very good resolution of the order of 10^{-6} Pa (or better than 0.01% at pressures above 1×10^{-2} Pa). To suppress the pressure rise of outgassing from chamber walls (mainly hydrogen) a nonevaporable getter (NEG) cartridge has been installed in the chamber. NEG pumps active gasses, but not He from the leak.

Fig. 2 shows pressure rise in volume V_1 over a time period of 4800 s. All pressure readings are given as He equivalent. Isolation valve to the turbomolecular pump has been closed at time t=0 s. It should be noted that for rate of rise measurements the offset correction of SRG readings is not necessary (at t<0 the true pressure in the chamber was below 10^{-6} Pa, but the displayed SRG reading without offset correction was 8.238×10^{-4} Pa). It should be also noted that it is not necessary to know the rotor accommodation coefficient for He gas, because the same rotor is used for measurements of P_1 and P_2 and we have to measure only ratio R of rate of pressure rise, (2) and (4). Therefore nominal accommodation coefficient σ =1 can be entered into SRG controller.

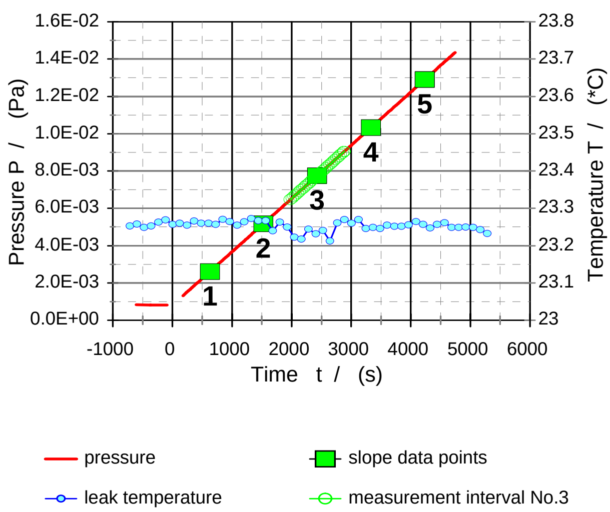

Fig. 2. Pressure rise due to He gas flow into volume V_1 and He leak temperature.

Rate of pressure rise $\dot{P}_1$ is equal to the slope of $P_1(t)$ line and has been calculated by linear regression analysis over a time period of 900 s. Therefore 5 independent values of $\dot{P}_1$ can be obtained from the measurements in Fig. 2. The statistical analysis of results is given in Table 1. Relative standard error of the calculated slope s($\dot{P}_1$)/($\dot{P}_1$) was between 0.06 and 0.1 %

Table 1. Statistical analysis of results from Fig. 2.

No.	mean P_1	mean T	$\dot{P}_1$	s($\dot{P}_1$)	
	Pa	°C	Pa/s	Pa/s	relative
1	2.6E-03	23.26	2.8565E-06	2.10E-09	0.074%
2	5.2E-03	23.26	2.8544E-06	1.74E-09	0.061%
3	7.8E-03	23.24	2.8556E-06	2.68E-09	0.094%
4	1.0E-02	23.26	2.8533E-06	1.58E-09	0.055%
5	1.3E-02	23.26	2.8550E-06	2.44E-09	0.086%

2.2.Temperature dependence of He gas flow

Measurements of $\dot{P}_1$ have been repeated at different temperatures in the interval from 22.3°C to 23.5°C. Results are presented in Fig. 3. From this measurement we can determine following linear approximation to calculate $\dot{P}_1$ in the temperature interval 22.3°C<T<23.5°C:

$$\dot{P}_1 = \left(1.0886\times10^{-7}\times\frac{T}{{}^\circ C} + 3.2384\times10^{-7}\right)\ \text{Pa/s} \qquad (5)$$

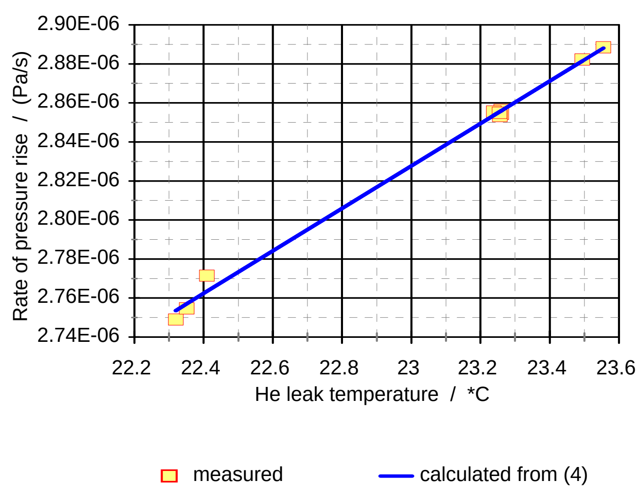

Fig. 3. Temperature dependence of $\dot{P}_1$.

3. VOLUME DETERMINATION

Vacuum chamber has several connection ports and one of them is normally closed by a blank flange (Fig. 1a) and this configuration represents volume V_1. To get volume V_2 the blank flange has been removed and replaced by a simple cylindrical extension tube. (Fig.1b). Added volume of this extension tube V_0 has been determined by gravimetric method and is equal to 0.8873 L.

Rate of pressure rise measurements $\dot{P}_2$ in volume V_2 were repeated 5 times. Results are presented in Table 2. Corresponding values of $\dot{P}_1$ at given temperature T were calculated using (5) and volume V_1 using (4).

Table 2. Results of rate of pressure rate of rise measurements $\dot{P}_2$ in volume V_2 and calculated volume V_1.

$\dot{P}_2$	T	$\dot{P}_1$ (5)	$R = \frac{\dot{P}_1}{\dot{P}_2}$	V_1 (4)
Pa/s	**°C**	**Pa/s**		**L**
2.5071E-06	22.98	2.8255E-06	1.12698	6.988
2.5068E-06	22.99	2.8266E-06	1.12758	6.955
2.5083E-06	22.99	2.8266E-06	1.12689	6.992
2.5102E-06	23.00	2.8277E-06	1.12649	7.015
2.5113E-06	23.02	2.8299E-06	1.12683	6.996

Mean value of five measurements of V_1 is 6.989 L and the standard deviation about mean is $s(V_1)$=0.019 L. This means that type A relative standard uncertainty of presented volume measurements equals to 0.28 %. This is nearly ten times larger than the type A uncertainty of R. The reason for this increase of uncertainty is that R is close to 1, so (R-1) in (4) is relatively small and the error becomes significantly larger.

4. CONCLUSIONS

A new method for determination of volume ratios and also the absolute value of volumes of vacuum vessels is presented. Method is based on a measurement of rate of pressure rise in a vacuum chamber at constant flow of inert gas. An example of measurement of a volume of a chamber with a nominal volume of V_1=7 L and added volume V_0=0.89 L is presented (volume ratio $R=(V_1+V_0)/V_1$=1.127). For pressure measurements we have used a spinning rotor gauge (SRG) and He permeation leak was used as a gas source.

In the first part of experiment we determined the temperature dependence of He gas flow, which was approximated by a linear function in a narrow temperature range. Subsequently this linear approximation was used to correct for flow variations due to the temperature variations of He permeating element.

Uncertainty of volume determination depends also on the ratio of volumes R and increases considerably in a case, where R is close to 1. Even in relatively unfavourable case where R=1.127, the type A relative uncertainty of measured volume was less than 0.3%.

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