Measurement 45 (2012) 2449–2451

Contents lists available at SciVerse ScienceDirect

Measurement

journal homepage: www.elsevier.com/locate/measurement

Accurate conductance measurements of a pinhole orifice using a constant-pressure flowmeter

James A. Fedchak *, Dana R. Defibaugh

National Institute of Standards and Technology, 100 Bureau Drive, Gaithersburg, MD 20899-8364, USA

ARTICLE INFO

Article history:
Available online 22 November 2011

Keywords:
Rarefied gas flow
Circular orifice
Low gas flow
Flowmeter
Gas flow standard

ABSTRACT

A pinhole orifice with a known conductance can be used as a secondary flow standard. Commercially available laser-drilled pinhole orifices with diameters ranging from 1.0 μm to 50 μm can have molecular-flow conductances ranging from about 0.1 μL/s to 200 μL/s for N_2 at 23 °C. Gas flows of 10^{-11}–10^{-6} mol/s can easily be produced by applying an upstream pressure in the range of 1–10^5 Pa. Accurate measurements of the orifice conductance as a function of pressure are required to use the pinhole orifice as a basis of a flowmeter. We use a constant-pressure flowmeter to make accurate measurements of the conductance of a 20 μm orifice as a function of pressure for gas flows of Ar and N_2 into vacuum. We present results of these conductance measurements for an orifice with a nominal diameter of 20 μm. The N_2 conductance of this orifice ranged from 30 μL/s to 60 μL/s over the range of pressures investigated, and was measured with an uncertainty of better than 0.2% ($k = 2$) for upstream pressures greater than 10 Pa.

Published by Elsevier Ltd.

1. Introduction

A flow constriction with a known conductance, C, can be used as a gas flow standard. The pressure difference across the constriction is proportional to the gas throughput q_{PV} and the molar flow rate $\dot{n}_C$:

$$(P - P_d)C = q_{PV} = \dot{n}_C RT. \tag{1}$$

Here R is the universal gas constant, T is the gas temperature, P is the pressure upstream of the constriction and P_d is the downstream pressure. For flows into vacuum chambers, $P \gg P_d$ and the downstream pressure can be considered to be zero.

We are interested in using laser-drilled pinhole orifices to produce flows of N_2 gas in the range of 10^{-11}–10^{-6} mol/s because a flowmeter based on an appropriate set of orifices would be easy to automate and use for vacuum gauge calibrations. In principle, any type of constriction could be used; however, commercially available laser-drilled orifices with diameters from 1 μm to 50 μm are well suited because they will produce N_2 gas flows into vacuum in the range of 10^{-11}–10^{-6} mol/s when an upstream pressure of 10 Pa to 100 kPa is applied. This is a convenient pressure range since capacitance diaphragm gauges (CDGs) and resonant silicon gauges (RSGs) can be used to determine the upstream pressure to within about 0.3% ($k = 1$) over a pressure range of 10 Pa to 130 kPa. Temperate uncertainties of better than 0.1% are easily achieved with calibrated platinum resistance thermometers. The real challenge lies in determining the conductance of the orifice constriction to high accuracy. A constant-pressure flowmeter can be used to meet that challenge, and in this paper we present measurements of the conductance of a 20 μm orifice for N_2 and Ar gas flows over the pressure range of 1 Pa to 100 kPa. For $P > 10$ Pa the resulting uncertainty was less than 0.2% ($k = 2$).

2. Measurement technique

2.1. Description of the orifice

The measurements were made on an orifice with a nominal diameter of 20 ± 2 μm (manufacturer's tolerance).

* Corresponding author.
E-mail address: james.fedchak@nist.gov (J.A. Fedchak).

0263-2241/$ - see front matter Published by Elsevier Ltd.
doi:10.1016/j.measurement.2011.10.046

The orifice was laser-drilled into a nickel foil of nominal thickness $L = 12.7\ \mu m$ that was supported by a thicker nickel-coated CuBe substrate. The substrate was sealed between the end faces of two stainless steel tubes by compressing the orifice substrate between two indium foil gaskets.

Although the indium foil created an adequate seal for the present measurements, our implementation of this sealing technique was unsatisfactory for a flow standard. The compression slightly distorted the orifice substrate, and it also caused small particles of indium to shear off and stick to the orifice substrate and tube walls. The possibility that a particle could obstruct the orifice, or that relaxation of mechanical stress induced by the distortion could slowly change the conductance over time is not acceptable in a flow standard. We are investigating better mounting and sealing techniques, such as vacuum brazing to a stainless steel flange.

2.2. Measurement apparatus and equation

For many years, the NIST constant-pressure bellows flowmeter (BFM) has been used as a flow standard to calibrate vacuum gauges and He leaks [1]. It is also possible to use the BFM to directly measure the conductance of a constriction, as was done by Jousten et al. [2] and discussed by Jitschin [3]. We will now describe our measurement technique with the aid of Fig. 1; additional details of the BFM can be found in McCullough et al. [1].

For a typical vacuum gauge calibration, the BFM produces a known flow of gas by maintaining a constant pressure P in a variable volume V that is upstream of an adjustable flow constriction. The variable volume is defined by a flexible bellows surrounded by a reservoir of incompressible oil. Gas lost through the constriction is compensated by a reduction of the variable volume at a known rate $\dot{V}$. A differential CDG is used to monitor the pressure difference between the variable volume and a fixed reference volume, and electronic feedback holds P constant by driving a piston into the oil to maintain zero pressure on the differential CDG. Thus the pressure maintained in the variable volume is the same as the reference volume, and the gas flow rate is proportional to $P\dot{V}$. Accurate measurements of the pressure within the reference volume are made using a combination of CDGs and a RSG.

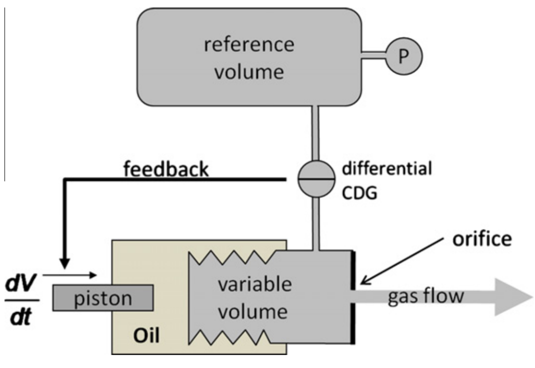

Fig. 1. Diagram of the bellows flowmeter (BFM).

For the present conductance measurements, the variable constriction was replaced by the pinhole orifice. The molar rate-of-change within the variable volume is given by a combination of the molar outgassing rate $\dot{n}_{OG}$ and the flow rate through the constriction:

$$\dot{n} = \dot{n}_{OG} - \dot{n}_C = \frac{1}{RT}\frac{\mathrm{d}(PV)}{\mathrm{d}t}. \tag{2}$$

At low pressures, $\dot{P} \neq 0$ and a small pressure rise $\dot{P}_{OG}$ due to outgassing in the reference volume is observed. By combining the above definitions with Eq. (1), we can derive the measurement equation for the conductance of the pinhole orifice:

$$C = -\dot{V} - \frac{1}{P}(\dot{P}_{OG}V - \dot{n}_{OG}RT). \tag{3}$$

The first term represents the rate-of-change of the variable volume, and the last two terms in parenthesis represent contributions from outgassing within the reference and variable volume. The outgassing terms are about a 1% correction to the total conductance at $P = 1$ Pa and about 0.1% at $P = 10$ Pa. For $P \gg 10$ Pa, the measurement equation simplifies to $C = -\dot{V}$.

2.3. Measurement uncertainty

The total relative uncertainty for the conductance measurement can be written as:

$$u_c = \sqrt{u_{\dot{V}}^2 + u_{OG}^2 + u_P^2 + u_T^2}. \tag{4}$$

The first term is the uncertainty of the volume rate-of-change and it is the major component of the uncertainty for $P > 10$ Pa. Its value is $u_{\dot{V}} = 0.1\%$ ($k = 2$) over the entire range of pressures investigated [1]. The second term, u_{OG} is the uncertainty due to outgassing in both the variable and reference volumes, and takes into account all uncertainties associated with the last two terms in Eq. (3). Both $\dot{P}_{OG}$ and $\dot{n}_{OG}$ are known to only about 20%, but are mitigated by a factor of $1/P$. At the lowest pressure where data was taken (1.5 Pa), $u_{OG} > 0.4\%$ ($k = 2$) and for $P > 10$ Pa, u_{OG} is less than 0.1%. Aside from the small outgassing terms, the measurement Eq. (3) does not depend on pressure; however, as is clear from Fig. 2, the measured conductance is a function of pressure. We estimated u_P by multiplying the pressure measurement uncertainty, $\delta P/P$, by the slope of $C(P)$ in Fig. 2 and obtained $u_p \leqslant 0.02\%$ over the entire range of $C(P)$ investigated; therefore u_p does not make a relevant contribution to the conductance uncertainty. The measured conductance is also a function of temperature. In molecular-flow, the conductance is proportional to the average gas velocity, $\bar{v}$ which is a function of $T^{1/2}$. For the purpose of estimating the temperature uncertainty, we assume that a $T^{1/2}$ dependence holds over entire range of pressures investigated, and obtain $u_T = \frac{1}{2}\left(\frac{\delta T}{T}\right) < 0.1\%$ ($k = 1$). Type A uncertainties do not make a significant contribution to the total combined uncertainty; measurements of C repeated to within 0.02% ($k = 2$). The total uncertainty is summarized in Table 1.

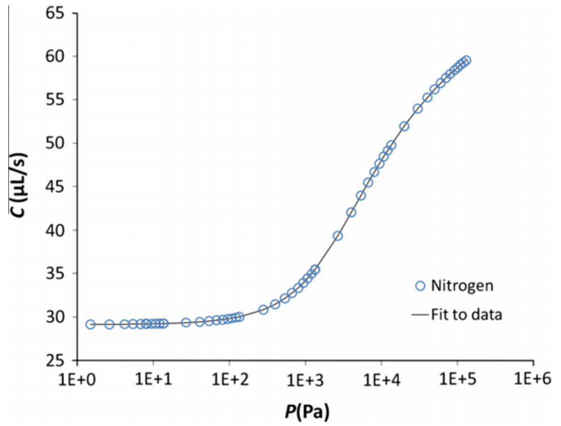

Fig. 2. Measured N_2 conductance as a function of pressure.

Table 1
The total relative uncertainty of the conductance measurements as a function of upstream pressure.

Pressure (Pa)	u_c ($k = 2$); percent uncertainty
1.5	0.46
10	0.14
⩾100	0.12

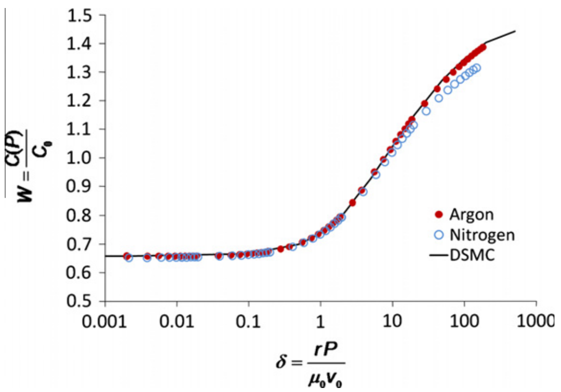

Fig. 3. Comparison of the measured conductance for Ar and N_2 with the numerical results of Ref. [4].

3. Measurements and discussion

Fig. 2 shows the N_2 conductance measurements for the 20 µm orifice and a fit to the data. To be useful as a flow standard, it is necessary to have a method of interpolating the conductance data that does not significantly increase uncertainty. We found that a ratio of polynomials in ln(P) provided an excellent fit to our data. To demonstrate the fit uncertainty, the function was fit to only half of the data. The resulting values from the fit agreed to the entire data set to within ±0.04% ($k = 2$).

Conductance measurements were also made using Ar gas. It is interesting to compare the measurements for both gases with the numerical results of Varoutis et al. [4] who used a direct simulation Monte Carlo (DSMC) method to calculate the flow of rarefied monatomic gas through a short tube into vacuum. To compare our measurements to the calculation, it is necessary to know the orifice radius, r, and the length, L. We used the nominal substrate thickness $L = 12.7$ µm and chose $r = 10.974$ µm to match our Ar measurements to the DSMC results at the lowest pressure measured. The results given in Fig. 3 are shown as a function of the gas rarefaction parameter $\delta = rP/\mu_0 v_0$; here μ_0 is the gas viscosity and v_0 is the most probable gas velocity. The measured conductance was converted into a reduced flow rate, W, by normalizing to C_0, defined as the conductance of an infinitely thin orifice in the molecular-flow regime and given by $C_0 = \pi r^2 \bar{v}/4$. Plotting the DSMC results required an interpolation between values of L/r in Table 1 of Ref. [4], which added an unknown and perhaps significant error. Even so, the DSMC results for $L/r = 1.157$ agree with our Ar measurements to within 1% over the entire measured range of gas rarefaction parameters.

The N_2 results do not fare as well: they agree with the Ar results and the calculated values to within 1% for $\delta < 5$, but they are more than 4% smaller than the calculated value at $\delta \sim 160$. A similar difference between Ar and N_2 conductance measurements was observed by Jitschin et al. [5] in their measurements of a 1.2 mm orifice with $L/r = 0.016$.

4. Summary and conclusions

We demonstrated the ability to measure the conductance of small flow constrictions with low uncertainty. Such measurements are useful for producing secondary flow standards. Our results show a difference between the conductance of Ar and N_2 gas for $\delta > 10$ that is similar to that observed by Jitschin et al. [5]. We plan to make additional measurements on pinhole orifices of different sizes with an improved mounting arrangement. We also plan to make accurate diameter measurements of the pinhole orifices to facilitate better comparison with other work.

References

[1] K.E. McCullough, C.R. Tilford, C.D. Ehrlich, F.G. Long, Low-range flow meters for use with vacuum and leak standards, J. Vac. Sci. Technol. A 5 (3) (1987) 376–381.
[2] K. Jousten, H. Menzer, R. Niepraschk, A new fully automated gas flowmeter at the PTB for flow rates between 10^{-13} mol/s and 10^{-6} mol/s, Metrologia 39 (2002) 519–529.
[3] W. Jitschin, Gas flow measurement by the thin orifice and the classical venturi tube, Vacuum 76 (2004) 89–100.
[4] S. Varoutis, D. Valougeorgis, O. Sazhin, F. Sharipov, Rarefied gas flow through short tubes into vacuum, J. Vac. Sci. Technol. A 26 (2) (2008) 228–238.
[5] W. Jitschin, M. Ronzheimer, S. Khodabakhshi, Gas flow measurement by means of orifices and venturi tubes, Vacuum 53 (1999) 181–185.