Vacuum 131 (2016) 111–114

Contents lists available at ScienceDirect

Vacuum

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

Short communication

# New leak element using anodic aluminum oxide

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A R T I C L E I N F O

*Article history:*
Received 28 April 2016
Received in revised form
7 June 2016
Accepted 8 June 2016
Available online 10 June 2016

*Keywords:*
Leak element
AAO
Shadow coating technique
Molecular flow
Conductance

A B S T R A C T

A leak element has been developed based on anodic aluminum oxide (AAO) by means of the shadow coating technique. The gas flow through the leak element can exhibit the molecular flow in the pressure range from high vacuum to atmospheric pressure for helium and other heavy gases, since the AAO is made of multi tubes with the diameter of about 70 nm. The shadow coating technique could achieve a specific number of tubes on AAO to satisfy the conductance required for vacuum applications. Some key characteristics of molecular flow are demonstrated according to the conductance measurement using difference method. The flow rate of any other test gases introduced in to the vacuum chamber can be predictable with the known flow rate of helium through the fabricated leaks.



Leak element is an essential tool to obtain quantitative measurements in the calibration of the leak detectors and ionization gauges. At present, crimped capillary leaks and orifices are often used to restrict the tracer gas flow and generate a leak rate [1–3]. However, it is difficult to further decrease the gas flow because the minimum feature dimension of the leaks is above one micron. As a result, the transitional flow between molecular and viscous flow often occurs in the restricted path. Because the conversion formula is related to the flow type and often unavailable, it is rather difficult to predict the flow rate for gas species when the helium leak rate has been measured. Therefore the conductance of standard leaks for various gas species is needed for calibration.

It will be obviously advantageous to fabricate leak elements with featured dimension at nanometer size since these devices can work in a molecular-flow region up to atmospheric pressure. With such elements the flow rate of any gas species is predictable from a calibration for just one gas species using empirical conversion formulas. For this reason, there is an increasing demand for standard leaks which can maintain the molecular flow up to relatively high upstream pressures. A new leak element using a sintered stainless steel filter with a pore size of less than 1 μm has been developed for in-situ calibrations of ionization gauges and quadrupole mass spectrometers (QMSs) [4]. Nano-holes with 200 nm diameters were fabricated into silicon nitride film using focused ion beam technology [5,6]. Both leaks exhibit molecular flow in the pressure range from high vacuum up to $10^4$ Pa and can therefore be used as predictable leak elements for any non-condensable gas species.

In this paper, we report a new leak element based on AAO, which could be employed in the range where the gas flow through it realizes molecular flow at a pressure of less than $10^5$ Pa. AAO is hexagonally arranged honeycomb structures with a uniform pore size of less than 100 nm [7]. Each pore could be considered as a long tube with nanometer size, which is an ideal structure and can be used as a leak element working in a molecular-flow regime up to atmospheric pressure. The leak element fabrication and its geometrical characterization are introduced. The flow rate is also measured using difference method, which shows the linear dependence on the inlet pressure and the inverse of the square root of molecular weight of gas species.

High-purity (99.999%) aluminum foil with the thickness of 50 μm was used to fabricate porous alumina membrane by a typical two-step electrochemical procedure [8,9]. The pore size and the

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http://dx.doi.org/10.1016/j.vacuum.2016.06.007
0042-207X/

spacing between adjacent holes of porous alumina membrane can be controlled by adjusting the anodization condition. The diameter of the tube is easy to keep under 100 nm, which can exhibit molecular flow even at the pressure of $10^5$ Pa. However, the number of tubes in 1 $mm^2$ of AAO is nearly $10^8$, resulting in the total conductance too high. In order to obtain specific conductance of standard leak compatible to vacuum applications, there is a challenge to get an AAO with a specific number of tubes.

In our work, a shadow mask coating technique [10,11], as shown in Fig. 1, was proposed to prepare a AAO with a specific effective area. An object was directly put onto the AAO substrate as a shadow mask. Then the sample was coated with a thermal deposition aluminum layer. The thickness of the metal layer should be above 400 nm to seal the tubes so that the gas is unable to flow through them. After the mask removed, an aperture with a specific number of pores was exposed as large as the mask on the AAO. Because of the shadow effect, the size of the aperture for the gas flow can be varied from tens micron to millimeter according to the desired conductance. The thickness of the metal coating was measured by Tenco Alpha step-500 stylus profilometer. The size of aperture was characterized by optical microscopy. In addition, the profiles of porous alumina membrane were examined by scanning electron microscope.

After the fabrication and the characterization of the pore size and the open area of the leak, the membrane is mounted between a silicon slice and a perforated copper disk, which is compatible and could replace the CF35 sealing, by the means of the Torr-Seal (trade name by Agilent Company) glue [6]. A hole with the diameter of 1 mm was drilled by laser ablation in the silicon slice. AAO is so brittle that must be clamped between the silicon slice and the perforated copper disk to resist 100 kPa of pressure differential. Vac-Seal (trade name by Kurt J. Lesker Company) was spin-coated on the silicon to guarantee leak proof between the silicon and AAO. Then the membrane is clamped between two CF35 flanges in order to test the performance in gas flow generation. Fig. 2a shows the assembling scheme of the leak element and Fig. 2b shows the assembling scheme of the leak element between two CF35 flanges.

The experimental apparatus of our measurement system for the conductance of the leak assembly is self-made based on the difference method, which has been reported elsewhere [12]. Difference method is a kind of dual-tank accumulation technique, in which a reference vacuum chamber is connected to the measurement chamber [12,13]. The mass flows could be achieved according to the pressure difference between the measurement and reference chambers by the differential pressure transducer (model Duwei DW200).

The gas flow rate $q$ (Pa $m^3$ $s^{-1}$) from the leak element can be expressed by the following formula:

$$q = V_m \frac{\Delta p_m}{\Delta t} \quad (1)$$

where $V_m$ ($m^3$) is the volume of the measurement chamber, which was previously determined by a different experiment, $\Delta P_m$(Pa) the pressure increase in the measurement chamber, $\Delta t(s)$ the measurement time.

The conductance of the leak element is given by

$$C_m = \frac{q}{P_u - P_d} \quad (2)$$

where $C_m$($m^3$/s) is the measured conductance of the leak element, $P_u$(Pa) the upstream pressure of the leak element, $P_d$(Pa) the downstream pressure of the leak element, which is far less than the upstream pressure $P_u$. By inserting Eqs. (1) and (2), the conductance of the leak element can be expressed by the following formula:

$$C_m = \frac{V_m \cdot \Delta P_m}{\Delta t (P_u - P_d)} \approx \frac{V_m \cdot \Delta P_m}{\Delta t \cdot \Delta P_u} \quad (3)$$

In the molecular flow regime, the conductance of the leak element is proportional to the inverse of the square root of the molecular mass, so the conductance of the leak element for a test gas is described as

$$C_m(\mathrm{M_n}) = C_m(\mathrm{He}) \sqrt{\frac{\mathrm{M_{He}}}{\mathrm{M_n}}} \quad (4)$$

where $C_m(\mathrm{M_n})$ ($m^3$/s) is the conductance of the leak element for a test gas,$C_m(\mathrm{He})$ ($m^3$/s) the experimentally determined conductance of the leak element for He, $\mathrm{M_n}$ the molecular mass of a test gas, $\mathrm{M_{He}}$ the molecular mass of He.

To establish the upstream pressure of the leak, the volume of the pressure stabilizing chamber must be large enough. In order to be sure that gas adsorbed at the walls of the circuits is removed and the only gas remaining inside the setup is the operating gas for flow rates measurement, the setup must be pumped down to less than

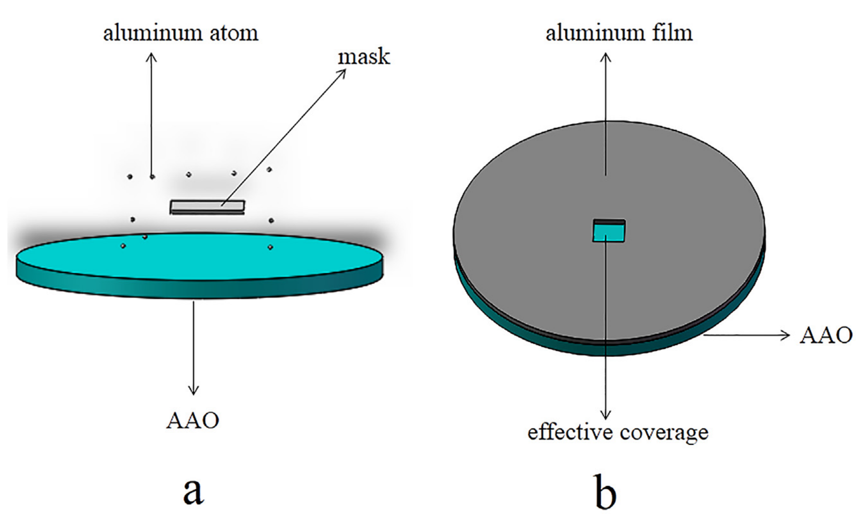


**Fig. 1.** The diagram of shadow coating technique.