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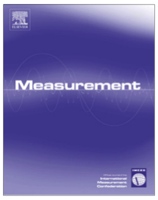

Nano-holes as standard leak elements

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ABSTRACT

Short tubes with diameters of the order of 200 nm were drilled into silicon nitride (Si_3N_4) membranes of 200 nm thickness by focused ion beam technology and examined as leak elements for vacuum technology applications. These nano-holes exhibit molecular flow in the pressure range from high vacuum up to 10 kPa and can therefore be used as predictable leak elements for any non-condensable gas species. The geometrical dimensions were determined by SEM, STEM and AFM techniques. By Direct Simulation Monte Carlo method the conductances of these short tubes were calculated from the measured dimensions. The calculated conductance agreed with the value measured by comparison with a primary gas flowmeter within their respective uncertainties.

1. Introduction

Standard leaks are an important tool to calibrate leak detectors and to provide safety and functionality of tested objects, e.g. pace makers. Most leak rate measurements are carried out with helium as test gas. The container to be tested, however, often contains a different gas or vapour. It is not easy to predict the leak rate for these kinds of gases when the helium leak rate had been measured, since the conversion formula depends on the type of flow, which is often unknown [1]. In addition, when the transitional flow regime between molecular and viscous flow is involved, there are no analytical formulas available [2].

For this reason, it would be advantageous to have standard leak elements which are predictable for any gas species, either from geometry or from a calibration for just one gas species. With such elements it is possible to measure real leaks for any gas and check empirical conversion formulas.

In the molecular flow regime where the mean free path of molecules is greater than a characteristic dimension of an object, conductances and flow rates can be easily predicted from geometrical dimensions. Very small elements like tubes, channels or orifices of the order of 100 nm in lateral dimension will both exhibit molecular flow through them up to relatively high upstream pressures (100 kPa) and generate suitably large leak rates at the same time.

For this reason, such elements were produced by the focused ion beam (FIB) technique and tested for their suitability as standard leak. In the following section we will report on the leak elements fabrication and their geometrical characterization, while in Section 3 we will present the calculation of their conductance by Direct Simulation

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Monte Carlo (DSMC). Section 4 explains the experimental set-up of the conductance measurements by vacuum technique. The results are discussed in Section 5 before we conclude in Section 6.

2. Leak elements fabrication and their geometrical characterization

The nano-holes are drilled in low stress silicon nitride (Si_3N_4) membranes (Fig. 1) by a focused ion beam (FIB) [3,4]. The membranes are custom made on chips with size of 5 mm × 5 mm, window size 100 μm × 100 μm, thickness 200 nm and are able to resist 100 kPa of pressure differential. Considering the geometric characteristics (length and diameter) of the nano-holes used in this work, the FIB machining overcomes the speed limitation of others techniques used to drill nano-holes, and in addition does not need chemically assisted etching by gas injection, such as in the case of others techniques used for this purpose [4–6].

To drill the nano-holes we have used a CrossBeam® workstation 1540XB model by Zeiss, which combines a SEM and a FIB. The Ga^+ ion beam has an energy range from 5 to 30 keV, current of 1 pA–20 nA, and a minimum spot size of 7 nm [7]. For this investigation a nano-hole has been drilled by means of 30 keV Ga^+ beam, using a current of 200 pA and a dwell time of 1 s [8].

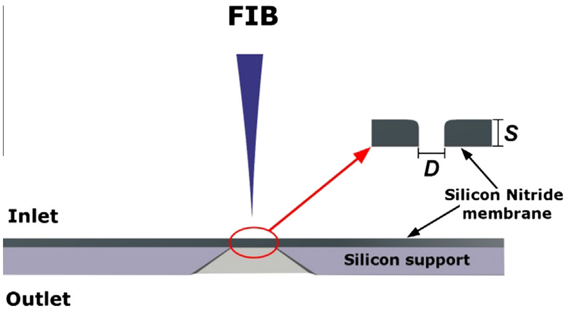

Fig. 1. Schema of the cross-section of the silicon chip with the silicon nitride membrane, where *S* is the thickness of the membrane and *D* is the diameter of the nano-hole.

We have determined the nano-hole geometry by means of Scanning Electron Microscopy (SEM), Scanning Transmission Electron Microscopy (STEM) and Atomic Force Microscopy (AFM). Soon after the nano-hole fabrication we have collected its SEM and STEM images *in situ*. From these images it is possible to measure the open area and determine the equivalent diameter of the nano-hole with a relative uncertainty of 2.5%. This value of uncertainty was achieved by calibrating SEM, STEM and also the AFM with a certified calibration gratings supplied by the Italian National Metrology Institute (INRIM).

Fig. 2 shows the SEM and STEM images of the nano-hole under investigation. The equivalent radius calculated from the open area of nano-hole is 92 nm. In Fig. 2 it is also possible to see the difference between STEM and SEM images of these structures: in the STEM image the edge of the nano-hole shows a stronger contrast than in the SEM image, because the image is obtained detecting the transmitted electrons. This stronger contrast allows a more precise measurement of the open area.

Subsequently, we have characterized the nano-hole by means of AFM collecting images of the nano-hole in tapping mode using tips Olympus OMCL AC160TS with an apical radius of 7 nm. The AFM analysis of the nano-hole shows the surface morphology of the region drilled, i.e. a portion of the surface containing the nano-hole. In spite of some limitations of the AFM analysis, this technique provides a very detailed 3D reconstruction of the inlet region of the nano-hole and of the surrounding area. Fig. 3 shows an example of the nano-hole inlet region morphology.

Due to the nature of the technique, AFM investigates only the inlet area, while the inner and exit part is not accessible (Fig. 4). Since the energy distribution in the ion beam of the FIB has a Gaussian profile, the same shape is partially reproduced in the inlet region. For this reason, the nano-holes inlet area is a funnel with a Gaussian profile. While, to study the inner part of the nano-holes we have used an indirect approach, i.e. we cut and analyzed cross-sections of nano-holes, obtained with the same FIB

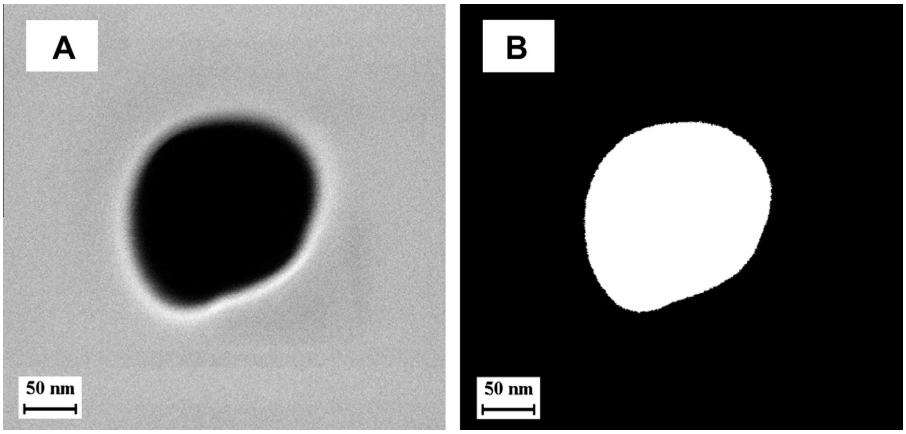

Fig. 2. Leak element investigated: (A) SEM image and (B) STEM image.