Nano-holes for vacuum applications

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Abstract. In order to calibrate leak-detection instruments, devices based on permeation leaks and physical leaks are used. Unfortunately, permeation leaks are very sensitive to small temperature fluctuations and can be used only with those gases for which permeating materials are available. While, physical-leaks that overcome these restrictions are susceptible to clog, in particular when the gas flow through them is in viscous regime. Furthermore, for many type of leak devices, the conductance is not predictable based on dimensional, gas species and temperature data, for this reason no estimates or only rough estimates of leak rates are possible for conditions under use where they differ from those of the calibration. Here, we present the fabrication and characterization of a new kind of physical-leak devices based on nano-holes, which overcome these problems. Nano-holes, with diameters less than or equal to 200 nm, work in molecular-flow regime up to atmospheric pressure and for this reason they do not clog. The nano-holes are manufactured by milling a silicon nitride membrane by means of Focused Ion Beam (FIB), and their shapes are characterized by both Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM). Because of the capability of the AFM to acquire surface three-dimensional images with very high resolution, it is a very useful tool to perform a topographic characterization of the nano-holes.

1. Introduction
Leak detection techniques are employed widely in systems in which must be guaranteed the leak-proof, such as vacuum equipment, packaging, or cryogenic storage applications. The most used devices to obtain quantitative measurements in leak detection are the Magnetic-sector or quadrupole-mass spectrometers. Generally, these spectrometers are calibrated using permeation or physical leaks. The permeation leaks are glass element immersed in a helium reservoir, which generate a gas flow that is used as reference. The leak rate obtained with these leaks is ranging from $10^{-11}$ to $10^{-5}$ Pa m$^3$ s$^{-1}$. However, the permeation leaks are very sensitive to the temperature fluctuations, have only one value of leak rate and work only for those gases for which permeating materials are available. Whereas, in the case of the physical leaks, in particular orifices, the tracer gas flows through a restriction or an aperture, generating a leak rate that is greater than or equal to $10^{-8}$ Pa m$^3$ s$^{-1}$. They can be used for any gas, including mixtures, and it is possible to change the flow rate tuning the inlet pressure [1]. Unfortunately, the orifices have great tendency to clog. Moreover, physical leaks work in an unknown flow regime. This means that there is need of a calibration curve in order to use it [2]. It has been a great improvement to fabricate orifices with dimension at nanometer size since these devices work in a

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molecular-flow regime up to atmospheric pressure, and overcome all drawbacks characteristic of the physical and permeation leaks [3]. Today the nanotechnology development provides several techniques and instrumentations able to fabricate nanometer holes with precise metrology [4-6]. In this paper we show the fabrication by means of Focused Ion Beam (FIB) and the characterization by means of atomic force microscopy (AFM) and scanning electron microscopy (SEM) of nano-holes, which work as physical leaks. Moreover, we show the performance of the nano-holes in helium flow generation and a rough estimation of their conductance obtained from geometric parameters.

2. Fabrication
The nano-holes are drilled in a Silicon Nitride ($\text{Si}_3\text{N}_4$) membranes low stress, made by SPI [7]. The dimensions of the membranes are custom made with chip size of 5 mm $\times$ 5 mm, window size 100 $\mu$m x 100 $\mu$m, thickness 200 nm and it is able to resist at 1 atm of pressure differential, in Figure 1 it is possible to see a representation of the chip cross-section with a nano-hole, which is milled from the upper side of the membrane.

To drill the nano-holes we utilized 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 1-20000 pA, and a minimum spot size of 7nm. Nano-holes with a diameter around 200 nm have been drilled with a single spot of 30 keV, using a current of 100 pA and a dwell time of 2s.

The membranes, after the geometric characterization, were mounted on perforated copper disks CF16 compatible in order to test their performance in helium flow generation. The copper disks have been shaped in order to allow an effective assembling of the membrane with the disk by means of the Varian Torr-Seal glue, as depicted in Figure 2.
3. Geometric characterization

The geometric characterization of the nano-holes allows us to evaluate the conductance of the nano-holes using theoretical estimation. First of all we collected SEM images of these nano-holes just after that they were drilled. From the SEM images it is possible to measure the open area of the nano-holes with high precision.

Subsequently the nano-holes were analyzed by means of AFM, the nano-holes images were collected in tapping mode using tips Olympus OMCL AC160TS with an apical radius of 10 nm. The AFM analysis of the nano-holes has displayed some limits due to the geometry of the interaction between the AFM-tip and the nano-holes. Usually the AFM images are reconstructed based on the assumption that the AFM-tip/sample interaction happens between the apex of the tip and the sample surface, unfortunately during the AFM-imaging of the nano-holes this assumption is not always true. In others words the tip/sample interactions have different geometric configurations, which induce different effects into the image processing. In Figure 3 are displayed three different situations in function of the geometry of the interaction between the AFM-tip and the sample surface: a) the tip interacts with the flat upper portion of the sample, the interaction happens between the apex of the tip and the surface; b) the tip approaches the edge of the nano-hole the interaction start to change, but in these condition the AFM-image of the nano-hole is still reliable; c) the tip is moved past the edge of the nano-hole and the interaction happens between the lateral surface of the tip and the walls of the nano-hole, the interaction tip/sample become much more complex when at a certain point the tip fills completely the nano-hole. Taking into account of all the configurations of the interactions tip/sample, it is clear that it exists a threshold, parallel to the membrane surface, below which the AFM data are no more reliable, i.e. below this threshold the interaction does not happen more between the apex of the tip and the sample surface, but it becomes much more complex and involves the whole tip and the walls of the nano-hole. Therefore the AFM analysis gives us information on the morphology of the inlet part of the nano-holes. The AFM analysis of the inlet part of the nano-holes shows a funnel-shaped region with a Gaussian profile due to the FIB beam profile, as it is possible to see in the figure 3d.

![AFM-tip/sample interactions](image)

**Figure 3.** AFM-tip/sample interactions: a) the interactions tip/sample happen between the apex of the tip and the surface; b) the interaction begins to change compared with previously case, but the measure is still reliable; c) the interaction became very complex and the measure is no more reliable; d) an example of AFM-imaging of a nano-hole (2 μm x 2 μm image size). Below it is possible to see the funnel-shaped region of the nano-holes.

In order to obtain more information on the actual shape of the inner part of the nano-holes we have cut and analyzed the cross-sections of several nano-holes using the FIB and the SEM. To minimize the damages due to the FIB milling we have deposited platinum on the nano-holes and after the milling
the nano-holes cross-sections were analyzed by means of the SEM. In Figure 4 it possible to see an example of the shape analysis of a cross-section of a nano-hole with a radius of 80 nm.

![Figure 4. SEM image of the FIB cross-section of a nano-hole with a radius of 80 nm. The bevelled edge of the upper part of the nano-hole is due to the Gaussian FIB beam profile.](image)

Since the analysis of the cross-section involves the destruction of the nano-holes, we have extrapolated the shape for the inner part of the nano-holes tested as leaks. By merging all together the information obtained from SEM, FIB and AFM analysis we were able to obtain a geometric characterization of the nano-holes, i.e. we can model the nano-hole like a tube with circular section and the upstream side funnel-shaped with a Gaussian profile.

4. Helium flow testing

The apparatus used for the calibration of the nano-holes is shown in Figure 5. It is composed of a vacuum chamber evacuated by a Navigator TV1001 Varian turbomolecular pump and equipped with a Varian ionization gauge UHV-24 and a custom-made Prisma Balzers quadrupole-mass spectrometer. In order to perform the helium flow test we have followed the same procedure described by Firpo et al. in [3].

![Figure 5. Experimental setup of the vacuum system for testing the nano-holes.](image)
5. Results and discussion

In order to obtain a reliable measurement of the nano-holes by means of the SEM, we have calibrated the SEM using a calibration grating, which has been supplied by the Italian Institute of Metrology (INRIM). The pitch of the calibration grating used in order to calibrate the SEM is \( p = 462.92 \) nm with standard uncertainty of 0.025 nm (calibration certificate n. 12-0381-02 by INRIM). After the SEM calibration the error in the SEM-images is less than 2%.

![Figure 6](image)

**Figure 6.** Inlet side images of the nano-holes investigated: above SEM-images and below AFM-images. The thickness of the membrane is the same for all nano-holes and is 200 nm, while the radii are: \( R_a = 93 \) nm, \( R_b = 85 \) nm, and \( R_c = 110 \) nm, for all values the error is around 2%. The tail visible in the images of the nano-hole \( a \) is due to a temporary instability of the FIB beam.

In Figure 6 are shown the SEM and AFM-images of the nano-holes investigated. The shape and the dimensions of the open area depend on several factors, for instance the temporary instability of the ion beam or the slight fluctuation of the FIB current, because of no conductivity of the membrane, can produce the tail visible in figure 6c, ion beams slight astigmatic can give elliptic shapes rather than circular, instead slight differences in the focus of the ion beam can give different size of the open area.

The radii of the nano-holes have been calculated measuring the open area of the nano-holes from the SEM-images, so we have extrapolated the radius for each nano-hole assuming that the nano-holes are perfectly circulars. The nano-holes radii are: \( R_a = 93 \) nm for the nano-hole \( a \); \( R_b = 85 \) nm for the nano-hole \( b \); and \( R_c = 110 \) nm for the nano-hole \( c \); for all these value the error is around 2%.

From the AFM-images is possible to obtain the profiles of the nano-holes, which are fitted with a Gaussian function. The points where finishes the funnel-shaped part and begins the straight tube are individuated by the comparison between the AFM-profile and the Gaussian function (see Figure 7A). Therefore in the case of the nano-hole \( a \), we obtained that funnel-shaped part finishes about of 50 nm below the membrane surface, so the length of the straight tube part of the nano-hole is around of 150 nm. In the others two cases the inlet region has almost the same dimensions, this is reasonable since all nano-holes have been drilled using the same FIB parameters.
The results of the helium flow measurements for the nano-holes a, b, and c are reported in Figure 8. The graphs show the linear dependence between the throughput and the upstream pressure up to 10^5 Pa. A fit with a power law has been added to the graphs and the exponents result to be very close to 1, confirming the linearity of the throughput in function of the upstream pressure, i.e. nano-holes with diameters around 200 nm work in molecular-flow regime.

Since the nano-holes investigated work in molecular-flow regime it possible to estimate the theoretical value of the conductance from the geometric parameters using the following equations:

\[ C = \frac{\bar{v}}{4} A P \quad \text{and} \quad P = \frac{14 + 4\frac{T}{D}}{14 + 18\frac{T}{D} + 3\left(\frac{T}{D}\right)^2}, \]

where \( C \) is the conductance, \( \bar{v} \) is the mean thermal speed, \( A \) is the open area, \( P \) is the transmission probability for a circular tube of length \( T \) and diameter \( D \) \[8\]. Therefore in order to calculate the conductances we have considered two different geometries. For the first one, we have approximated the nano-holes with straight tubes, so we have three tubes with the same length, 200 nm, but with three different radii. For the second one, we have neglected the funnel-shaped region of the nano-holes, considering only their straight tube part in the calculus of the conductances. The experimental and
theoretical results are reported in table 1. As it is possible to see in the table 1, the higher discrepancy between the experimental and theoretical data is obtained for the nano-hole c, which has the most irregular shape, while in the others two cases we have a very good agreement between the data experimental and theoretical ones.

Table 1. Nano-holes conductances. The helium flow tests have been performed at the temperature of 23°C.

| Nano-hole | Experimental Cond. (l/s) | Theoretical Cond. (l/s) | Comparison (%) |
|-----------|--------------------------|------------------------|----------------|
| a         | 5.14·10^{-9}            | 3.89·10^{-9}           | 24.3           |
| b         | 4.37·10^{-9}            | 3.86·10^{-9}           | 22.6           |
| c         | 4.59·10^{-9}            | 6.39·10^{-9}           | -39.2          |

6. Conclusions
Thanks to the high resolution capability of the AFM which makes it possible to characterize the inlet part of the nano-holes. The AFM imaging of the nano-holes can give us a 3D reconstruction of the inlet part of the nano-holes, without destroying them. The inlet region of the nano-holes has a funnel-shaped morphology, which is generated by the FIB beam. In fact the power of the FIB beam has a Gaussian distribution. From the analysis of several nano-holes cross-sections, it is possible to see that the funnel-shaped region is connected to a duct with straight walls, this duct can be considered like a tube. Moreover, making a fit of the AFM profile of the inlet region with a Gaussian function, it is possible to find where the straight tube region of the nano-hole begins. So it is possible to approximate the nano-hole with a tube with circular section and the same open area of the nano-hole. The comparison between the experimental values and the theoretical estimations of the conductances, of the nano-holes tested, has shown that the best agreement is obtained neglecting the inlet funnel-shaped part of the nano-hole. In addition it is also evident that more regular is the actual shape of the nano-holes and more precise is the theoretical estimation of the conductances. The precise geometric characterization of the nano-holes by means of the AFM and the SEM gives the possibility to perform numerical simulation studies of these devices using their actual shape without any approximations.

Acknowledgements
This work is funded by EMRP (European Metrology Research Programme). The EMRP is jointly funded by the EMRP participating countries within EURAMET and European Union.
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