

FEDSM-ICNMM2010-30488

GAS WALL INTERACTIONS OF RAREFIED GASES IN MEMS: A NEW EXPERIMENTAL DEVICE WITH INTEGRATED SENSORS

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ABSTRACT

A newly designed device for the experimental characterization of rarefied pressure driven gas flows is presented. The device is intended for both the thermal and the hydrodynamic analysis of gas flowing inside a single micro channel.

The innovative feature of the present design is the integration of temperature and pressure sensors inside the micro channel itself. The sensors are fabricated as an array on longitudinal thin film membranes, installed on a polymeric supporting frame.

A peculiar multi layer configuration of the device allows, on the one hand, the mounting of the sensor layer as a separate channel upper wall and, on the other hand, the interchangeability of the test sections.

The sensors directly face the gas stream, registering the temperature and the pressure profiles along the channel. This gives additional information about the gas behavior compared to the simplified assumption of a linear profile developing between the measured inlet and outlet values.

Examples of integrated sensors in micro channels have been already realized; however the majority of them are in silicon channels, as only with silicon fabrication technologies the sensor sizes can be reduced down to the micron range. This of course limits the field of application of the obtained results. In the present case, although silicon technology is still employed for the sensor manufacturing, this refers to the sensor wall only, while the remaining three-wall-channel is machined into an exchangeable foil. The foil, in principle, can be made of any material and can be easily replaced.

Several channel dimensions and materials, as well as different surface roughness levels can be tested with the same

device, making it very flexible and suitable for a broad characterization of gas wall interactions.

A future experimental campaign will investigate the influence of roughness and material on the flow and the heat transfer characteristics. This is a very critical point in rarefied gas applications in MEMS, as the similarities with conventional flows as well as new features need to be identified and analyzed.

With the described configuration it is possible to have insight into the flow parameters and to understand the actual behavior of gases under specific flow conditions. This is important in order to obtain a proper modeling and to validate the results of simulations and calculations on rarefied gas flows in micro channels.

KEY WORDS

Rarefied gases, gas-wall interactions, integrated sensors, roughness effects.

NOMENCLATURE

A	Channel cross-section area
D	Channel hydraulic diameter [$D = 2A/(l+h)$]
f	Friction factor [defined by eqn. (2)]
h	Channel height
Kn	Knudsen number [$Kn = \lambda/D$]
l	Channel length
\dot{m}	Mass flow rate
Nu	Nusselt number [$Nu = \alpha L/\kappa$]
p	Pressure

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q''	Heat flux
R	Specific gas constant
T	Gas temperature

Greek symbols

ΔL	Distance between two consecutive internal pressure measurements
σ	Streamwise momentum accommodation factor [$\sigma = (2 - \sigma_m) / \sigma_m$]
σ_m	Tangential momentum accommodation coefficient
λ	Gas mean free path
κ	Gas thermal conductivity
Π	Inlet/outlet pressure ratio
μ	Gas dynamic viscosity

Subscripts

i	Inner part of the channel
o	Outlet section of the channel
w	Channel wall
x	x -th internal measuring point along the channel

BACKGROUND

In the last couple of decades, the number of applications of micro-electro mechanical systems (MEMS) has constantly increased. The basic elements of MEMS are micro channels, the characteristic dimensions of which fall below the millimeter range.

Gas flows in microfluidic systems draw particular interest for the highly efficient performances that they can offer both in heat transfer and in fluid dynamic processes.

A large number of publications and studies on this field is present, as investigations are necessary for the determination of the transport and the heat transfer properties of the flow.

One of the crucial points for researchers is the assessment of the validity of conventional theory at the microscale.

In an early work Wu and Little [1] reported friction factors for silicon trapezoidal channels (with hydraulic diameters between 55.81 and 83.08 μm) which were about 10-30% higher than the theoretical values. They investigated a wide range of Reynolds numbers (up to 15000) and reduced their data with conventional friction factor correlations for compressible flows. An early dependence on roughness of the friction factor has been found starting from the laminar regime and this behavior has been ascribed by the authors as a peculiar characteristic of micro flows. Also Nusselt numbers higher than expected have been found [2].

Trapezoidal geometry has been tested by Harley et al. as well (with hydraulic diameters ranging from 1.01 to 35.91 μm), who reported a reduction of the friction factor with respect to

the classical theory for Reynolds up to 10^3 [3]. In particular the authors proposed a locally fully developed first order slip-flow model for the data fitting, taking into account the rarefaction and compressibility effects on the flow.

On the other hand, Ding et al. [4] tested triangular and rectangular stainless-steel channels (ranging between 400-600 μm of hydraulic diameter) and measured friction factors noticeably higher than those predicted for conventionally-sized channels. The authors also pointed out a strong dependence on the wall-roughness.

Despite the large amount of available references in literature, the different results are often in disagreement with each other. To this regard Morini highlighted in his review work [5] how certain aspects of fluid flows in micro channels still need to be investigated and fully understood. It has been as well pointed out that often similar experimental works (i.e. studies carried out under similar testing conditions) present disaccord conclusions. On the other hand a progressive chronological agreement among the experimental results can be found, thanks to the increased accuracy of experimental systems. In [6] it has been also verified that some of the documented disagreements with conventional theory can be reduced if the experimental uncertainty is properly taken into account.

Recently, particular attention has been paid to the identification and the understanding of the so-called scaling effects, which appear when the characteristic dimensions of the systems are reduced. Compressibility, rarefaction and gas-wall interactions are usually neglected in the modeling and the in the analysis of conventionally sized channels. However, as their effects on the flow are size-depending, the improper evaluation of their contribution for micro channels can lead to incorrect results. An overview of the works concerning these features is given in [7].

Compressibility effects arise because the high pressure drops characterizing gaseous flows in micro channels result in a non negligible variation of the density along the flow direction with respect to the classical incompressible case. As a result, also the velocity profile is modified.

It is conventionally stated that compressibility effects cannot be neglected when the Mach number is higher than 0.3 [8] and the ratio between absolute pressure drop and inlet pressure is higher than 0.05 [9].

Another relevant aspect to be carefully considered is the rarefaction of the gas flowing inside the channel. When the dimensions are reduced, the system characteristic length becomes comparable to the mean free path of the gas molecules, and the continuum assumption for the flow modeling fails. In general when rarefaction is not predominant (slip flow) the boundary conditions of the continuum equations can be modified to extend their validity. A theoretical overview of rarefied gaseous flows in micro channels can be found in [10].

Arkilic et al. [11] suggested a 2D modified set of compressible Navier-Stokes equations with first order slip-flow

boundary conditions. The theoretical model has been validated with experiments for the measurement of the mass flow in a silicon micro channel 52 μm wide and 1.33 high. The tested flows presented Reynolds number ranging between $1.4 \cdot 10^{-3}$ and $12 \cdot 10^{-3}$ and an outlet Knudsen number of 0.155. In a later work Arkilic et al. [12], starting from mass flow measurements, proposed a method for the computation of the tangential momentum accommodation coefficient (TMAC) used in the definition of the slip velocity.

Finally, numerous numerical solutions for the Graetz heat transfer problem integrated with slip boundary conditions have been proposed. An example of numerical analysis can be found in [13], where the heat transfer problem is solved for slip flow in rectangular geometry.

The key point to understand the behavior of rarefied gases is the gas-wall interaction along the flow. This is mainly because the deviations from the continuous assumption first occur in the region called Knudsen-layer, which extends a few mean free paths from the wall.

It is clear that the characteristics of the channel material, as well as the wall surface quality play a crucial role for the determination of the flow transport properties.

At microscale the relative influence of the surface quality on the channel sizes drastically increases. Due to the typical irregular distribution of the dimensions and the geometries of the roughness profiles, the integration of surface quality in analytical and numerical models is rather difficult. On the other hand, an accurate experimental characterization of roughness effects requires the accessibility of local information inside the micro channels, causing significant technical challenges. As a consequence, no general conclusions are available in literature for roughness effects on rarefied gas flows in MEMS. Indeed only few works can be found treating systematically the influence of surface quality in micro channels. Tang et al. [14] for example performed an experimental study for different kind of micro channels and micro tubes. The authors reported a deviation of the friction factor depending on the roughness level. In Croce et al. [15] and Croce and D'Agaro [16] a numerical approach for the modeling of roughness is presented. Although this kind of study can give some interesting suggestions for further investigations, it makes also evident the strong approximation that a regularly shaped roughness model brings about.

It is possible to sum up the following conclusions from the background literature review:

- Despite the numerous attempts to achieve a unique and reliable modeling for gas flows in MEMS, several aspects still need to be clarified and further investigated. To achieve exhaustive results, experiments should be undertaken taking into account all the flow aspects, analyzing their effects on the flow, to understand whether and when they can be neglected.
- Gas-wall interactions and in general scaling effects (such as compressibility, rarefaction and roughness) should be

taken into account in the modeling of gases flowing in micro channels.

- The actual role of the channel material, the manufacturing processes and the channel wall roughness on gas flows in micro channels is not well determined. This is mainly due to the difficulties of local characterization which is required for the study of these features.

EXPERIMENTAL APPROACH

The difficulty of performing direct measurements is probably the reason for the lack of knowledge in the field of roughness influence characterization for MEMS. The majority of experimental investigations in micro channels are performed with measurements of the inlet and outlet properties (e.g. temperature and pressure), from which the internal flow behavior is extrapolated. However, on a local scale the characteristics of the flow might differ from those in conventional larger channels, introducing completely new features or influencing the flow properties. In particular, the behavior of rarefied gases deviates more and more from that of a continuous fluid as the Knudsen number (Kn), the ratio between the gas mean free path and the characteristic length of the flow, increases. In these conditions, slip velocity and temperature jump appear at the wall, and a local experimental approach becomes fundamental for correct identification of the transport and the thermal properties of the flow.

A number of attempts for the integration of sensors in micro channels are reported in literature.

Jiang et al. [17] developed polysilicon thermistors for local temperature measurements in micro channels, while a micro machined temperature sensor array for a glass micro channel has been developed by Xue and Qiu [18].

Park et al. [19] integrated on a silicon micro channel resistance temperature detectors manufactured by deposition of platinum layers.

Chen et al. [20] manufactured heated silicon micro channels with integrated temperature and pressure sensors by depositing doped polysilicon. The authors also pointed out the problems related to the axial heat conduction on the side walls, which leads to significant heat losses. A technique for the fabrication of complex micro channel arrays integrating internal sensors and insulating side walls is presented in [20-23]. From these works it is evident the technical complication deriving from such manufacturing processes, and the problems that need to be solved to reach measurements with good resolutions [22].

The reported examples demonstrate the possibility of accessing local information in micro channels, but a wide implementation of these tools for the study of MEMS has not yet been achieved. One of the disadvantages of the above mentioned solutions is the limited field of applicability. Indeed, if the fabrication of integrated sensors in silicon is quite feasible, on the other hand it is rather difficult to create micro sensors on different substrates (i.e. metal, ceramic, polymers, etc.).

The present work describes a new experimental device designed for the characterization of gas flowing inside micro channels made from different materials. It allows a direct insight into the gas behavior and into the gas-wall interactions throughout the flow. The apparatus has been designed for both hydrodynamic and thermal characterization of the flow, which are realized with the integration of micro sensors inside the channel. Moreover, the testing conditions can be modified to achieve different rarefaction levels by decreasing the working pressure in the channel.

The main objective of the experiments is the identification of the influence of the surface quality on the characteristics of rarefied gas flows. For this purpose, tests with different materials and configurations can be performed, as the device allows an easy exchange of the test sections without modification of the whole measuring system. As a result, the experimental procedure is standardized, and it is easy to make comparisons between the different materials or surface properties.

EXPERIMENTAL SETUP

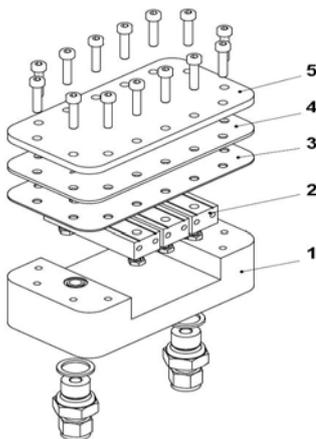


Figure 1: Scheme of the experimental apparatus with a multi layer configuration. (1) base; (2) heating blocks; (3) channel foil/test section; (4) sensor foil/fourth channel wall, (5) cover.

The device has a multi layer configuration, where each layer covers a particular function and can be handled almost independently. A scheme of the design is shown in Fig.1.

Starting from the bottom, the device comprises three heating/cooling blocks with receptacle holes for electrically powered cartridges. The temperature profile or the heat flux at the bottom of the channel can be imposed with a controlled electrical power station.

Above the heating layer the channel foil including the inlet and the outlet holes and a three wall micro channel is mounted. This is the actual test section and can be easily removed and substituted.

The third layer is a PTFE foil including a recess for the installation of a sensor chip. This represents the inner measurement system and consists of a silicon substrate (80x8 mm²) where an array of thin film membranes has been realized. On top of the membranes a series of temperature and pressure sensors have been manufactured. Figure 2 shows the sensor chip layout.

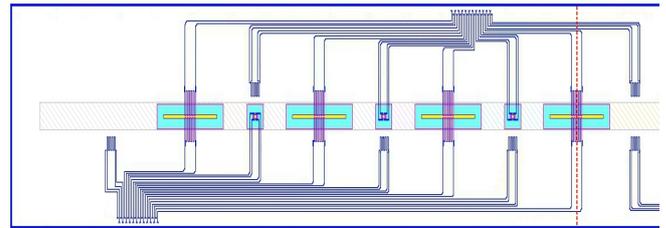


Figure 2: Sensor chip (only half) with membranes (yellow rectangles) and temperature and pressure sensors (on the long and the short membranes respectively).

The pressure sensors have a typical Wheatstone bridge configuration realized with polysilicon-aluminum contacts. A detail of a pressure sensor is showed in Fig.3.

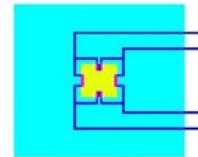


Figure 3: Details of a pressure sensor realized on the top of the thin film membrane (yellow).

The temperature sensors (details are shown in Fig. 4) are thermo couples consisting of ten polysilicon-aluminum junctions (thermopiles). They register the temperature difference between the center of the membrane and the beginning of the sensor leads (ΔT). Every membrane comprises two thermo couples (one per side), allowing a redundant measure and at the same time a symmetric heat conduction profile from the connection leads.

The choice of manufacturing thermopile-like sensors instead of thermistors is justified by the fact that with thermistors a relevant bias current should have been fed. This current would have also caused joule-heating phenomena on the polysilicon thin film membrane (which have a high thermal conductivity). The temperature increase would have been of the same order of magnitude of the temperature to be measured, leading to a non-negligible error on the final reading.

Outside the membranes, a series of thermistors, registering the temperature of the polysilicon substrate, have been realized. These signals can be used as references for the calculation of the absolute temperature inside the channel, from the differential temperature values registered by the thermopiles.

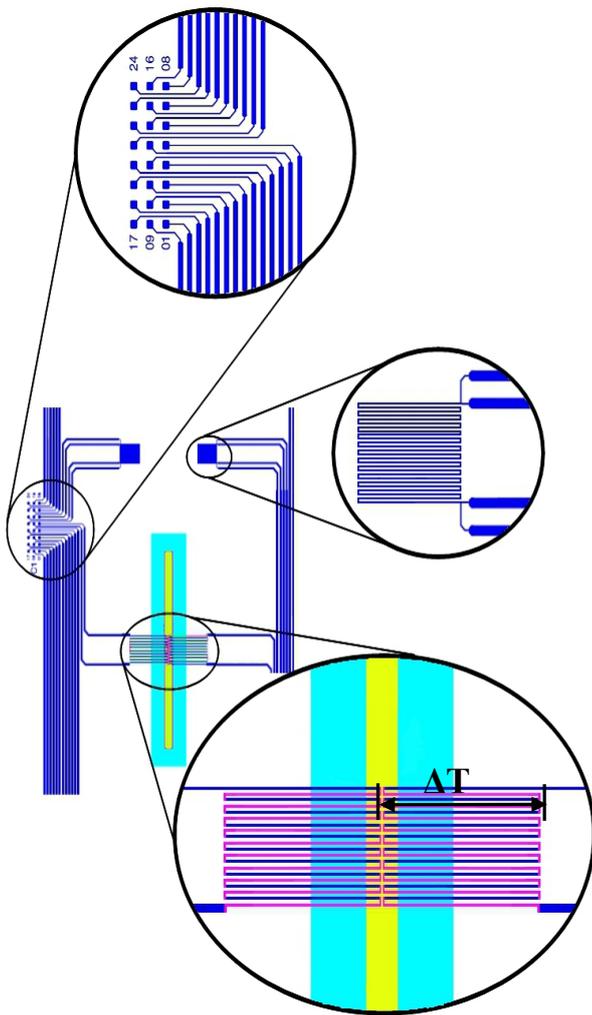


Figure 4: Details of the temperature sensors. From the top: contacts for the cable connections; thermistors for the temperature reference; thermopiles on thin film membranes with a series of junctions between polysilicon (magenta) and aluminum (blue).

The silicon chip, once mounted, constitutes also the upper cover of the channel (i.e. the channel fourth wall). The sensors face the inner part of it, and thus grant direct access to the local information of the gas flow.

The main feature of the described internal measuring principle is the application of the well known silicon fabrication technologies to a flexible experimental design. Thanks to the multi layer configuration, the whole structure can be opened and the channel foil can be easily changed. As a result, the test sections can be manufactured in principle from any material to be characterized (i.e. metal, ceramic and polymers) and tests are not limited to silicon, as it is the case in previous applications with integrated micro sensors.

Sensor fabrication process

Starting from a silicon substrate low pressure chemical vapor deposition process (LPCVD) is used for the deposition of silicon nitride and a low temperature oxide serving as insulation layers.



A layer of n-doped polysilicon is deposited on top of the oxide and is subsequently annealed to activate the dopants. The polysilicon is patterned using photolithography and reactive ion etching (RIE).



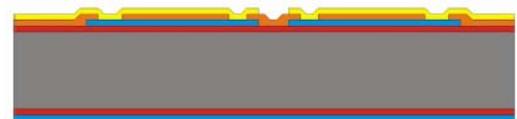
Plasma-enhanced chemical vapor deposition (PECVD) is used to apply a silicon oxide covering which constitutes the passivation layer between polysilicon and aluminum.



The contacts sites on the passivation layer are then opened with RIE.



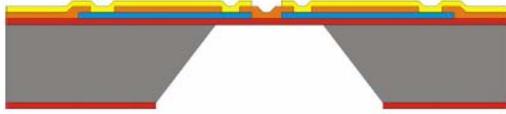
AlSi is deposited with sputtering above the passivation layer and is then patterned with photolithography and wet etching. A process of thermal annealing is performed in order to activate the contacts between polysilicon and aluminum.



On the rear side of the wafer, the nitride and oxide layer are patterned with photolithography and RIE.



As a final step the thin film membranes are realized by KOH anisotropic wet chemical etching.



The different chips can finally be diced and separated.

Measuring loop

The internal measuring system, realized on the silicon chip, is connected to the outside of the device via flexible polyimide cables. These are inserted in ZIF connectors (zero insertion force) on printed circuit boards (PCB) to which the data acquisition system is linked.

To achieve different levels of rarefaction and to perform tests in a wide range of operating conditions, the device needs to be operated below atmospheric pressure. Therefore, it is necessary to provide a vacuum insulated environment for placing of the whole structure.

To avoid the construction of a big vacuum volume, a small chamber, closed by a flange system, has been realized (Fig. 5).

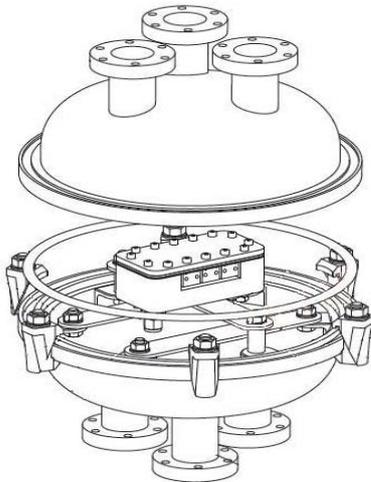


Figure 5: System for the vacuum insulation of the device.

Only the device and the sensor connections are included inside the vacuum chamber, while the rest of the experimental set-up and measuring system is kept on the outside. This configuration reduces the complexity of the testing loop, grants an easier accessibility for the handling and the monitoring of the device, and also allows a good thermal and vacuum insulation.

Finally, the device and the vacuum flange system are installed in an experimental loop, which includes conventional inlet-outlet temperature and pressure measurements, mass flow meters and a vacuum pump (Fig. 6).

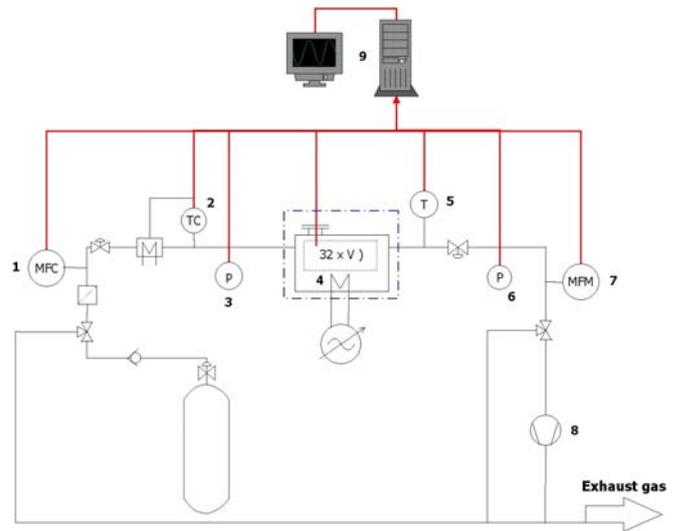


Figure 6: Scheme of the experimental loop. Mass flow controller (1); inlet temperature controller (2); inlet pressure measurement (3); flange vacuum system with the device and the integrated sensors (4); outlet temperature measurement (5); outlet pressure measurement (6); mass flow meter (7); vacuum pump (8).

FUTURE EXPERIMENTS

The experimental campaign requires two main steps. The first is the roughness characterization of the different test sections, and the second consists of the actual tests with the described setup.

The surface quality analysis can be done at first with SEM imaging for a qualitative characterization. Subsequently, white light interferometry or tactile measurements can be employed for the quantification of the actual roughness profile.

As a first step, micro channels fabricated on metal foils will be tested. 400 μm wide and 100 μm high channels will be manufactured from oxygen free copper, stainless steel and aluminum (with progressively decreasing surface qualities). In addition, also wet chemically etched polymer micro channels will be tested, these last having a typical semi-elliptical cross section (with a 400 μm upper opening). The four combinations of materials and manufacturing processes lead to very different characteristic roughnesses, giving a first general overview on roughness effects.

The rarefaction degree inside the micro channel can be directly set with the vacuum pump imposing the inlet and the outlet pressures (regulated by needle valves), and thus determining the inlet and outlet Knudsen number, once the dimensions of the channel are known.

The resulting internal pressure profiles can be measured and compared with the analytical ones and with other experimental data from literature. The analytical non-dimensional pressure profile for a compressible slip gas flow can be calculated with Eqn. (1) [11].

$$\tilde{p}(\tilde{z}) = -6\sigma Kn'_0 + \frac{1}{\sqrt{(6\sigma Kn'_0)^2 + (1+12\sigma Kn'_0)\tilde{z} + (\Pi^2 + 12\sigma Kn'_0\Pi)(1-\tilde{z})}} \quad (1)$$

The pressure p is non-dimensionalized by dividing it by the outlet value p_0 , while the streamwise coordinate z is divided by the channel length l . The non-dimensional quantities are showed with a $\tilde{\sim}$ symbol. The modified Knudsen number Kn' is defined here as the ratio between the gas mean free path and the minimum channel dimension.

The internal pressure measurements serve to calculate the local friction factor, which can be determined for different roughness levels. The friction factor for an isothermal compressible flow in a constant area duct is expressed by Eqn. (2) [9].

$$f_x = \frac{D}{\Delta L} \left[\left(\frac{1 - \left(1 - \frac{(p_{i,x} - p_{i,x+1})^2}{p_{i,x}^2} \right)}{\left(\frac{\dot{m}\sqrt{RT}}{A p_{i,x}} \right)^2} \right) - 2 \ln \left(\frac{1}{1 - \frac{(p_{i,x} - p_{i,x+1})}{p_{i,x}}} \right) \right] \quad (2)$$

The second part of the experimental analysis requires a thermal characterization of the flow, which is done by imposing either a given temperature profile or a given heat flux at the bottom wall of the device. The local temperature profile is recorded by the internal sensors and can be used to determine the Nusselt numbers. Data are reduced with equation (3).

$$Nu_x = \frac{q''_{w,i} D}{(T_{w,i} - T_{x,i}) \kappa} \quad (3)$$

The obtained results can be compared to theoretical correlations for conventional channels and also to the results of previous studies.

By analyzing how the different features vary with changing channel materials and characteristic roughness, a direct correlation between testing conditions, surface properties and gas behavior can be derived.

CONCLUSIONS

An experimental design for the characterization of rarefied gas flows in micro channels has been presented. The device is equipped with internal temperature and pressure sensors developed with silicon micro fabrication technology. Unlike previous attempts, this system is not limited to silicon or glass micro channels, thanks to a flexible multilayer configuration. The test sections can be easily substituted, allowing the characterization of different channel materials.

The integration of roughness characterization and local measurements makes possible an understanding of the gas-wall interactions and clarifies their actual influence on the gas transport properties. The effects on the flow of manufacturing

processes, channel material and resulting surface roughness can be investigated.

With the presented experimental approach it is possible to extend the actual knowledge on rarefied gases in MEMS including the interactions between the different scaling effects (i.e. compressibility, rarefaction, roughness and gas-wall interactions). By comparing the obtained experimental results with the available literature data, the existing theories can be verified or new model can be eventually proposed. Moreover, the test results could be useful for the validation of future numerical approaches on gas-wall interactions and roughness effects analysis.

ACKNOWLEDGMENTS

The research leading to these results has received funding from the European Community's Seventh Framework Program (FP7/2007-2013) under grant agreement n° 215504.

The authors wish to thank Dr. P. Ruther from IMRET Freiburg for his work in the design and fabrication of the integrated micro sensors.

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