Experimental characterization of water flow through smooth rectangular microchannels

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This article presents experimental results obtained in water flows through smooth rectangular microchannels. The experimental setup used in the present study enabled the investigation of both very small length scales (21–4.5 μm) and a wide range of Reynolds numbers (0.1–300). The evolution of the friction coefficient was inferred from pressure drop versus flow-rate measurements for two types of water with different electrical conductivities. The channels were made of a silicon engraved substrate anodically bonded to a Pyrex cover. In these structures, pressure losses were measured internally with micromachined Cu-Ni strain gauges. When compared to macroscale correlations, the results demonstrate that in smooth silicon-Pyrex microchannels larger than 4 μm in height, the friction law is correctly predicted by the Navier-Stokes equations with the classical no-slip boundary conditions, regardless of the water electrical conductivity ($>0.1 \mu\text{S cm}^{-1}$).

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Several fundamental studies, over the last 15 years, have explored the basic characteristics of momentum and heat transfer for liquids flowing in microstructures. This research has been motivated by their future use in many industrial applications. The previously published works related to this paper are based on pressure drop versus flow-rate measurements performed in microchannels, both in the laminar regime^{1–7} and in the transitional regime.^{8–13} These experimental studies have questioned the validity of using the classical flow theory for liquids in ducts of hydraulic diameter smaller than 500 μm . Up to the last two or three years, the published literature showed very dispersed results on the friction coefficient in microchannels (see review papers^{2,13,14}). These discrepancies were interpreted by some investigators as the result of early transition to turbulence or electrokinetics interactions at the solid-liquid interface. However, these hypotheses do not match all the published results and the great variety of trends observed remains difficult to interpret. The deviations from the classical theory may also have been, in part, caused by erroneous comparisons between results obtained in smooth and rough microducts.^{6,8,9,12,15} This paper is focused on the hydrodynamics of liquid flows in smooth wall microchannels (relative roughness $<0.5\%$).

In duct flows, pressure losses are evaluated in terms of the Fanning friction factor

$$f = \tau_w / (1/2 \rho U^2), \quad (1)$$

where the wall shear stress τ_w is deduced from pressure loss measurements, ρ is the density, and U is the fluid bulk velocity. For the fully developed laminar regime in conventional ducts, the product $f\text{Re}$ (Reynolds number Re of the flow based on U and the hydraulic diameter D_h) is a constant referred to as the Poiseuille number Po , which only depends on the cross-sectional geometry of the duct.¹⁶ Table I summarizes the available friction coefficient measurements for laminar liquid flows in smooth microducts. These works are displayed in decreasing order of the internal length scale (smallest dimension of the cross-sectional geometry). Much work has been done on flows in smooth microducts with an inner scale larger than 30 μm . Of special interest are the most recent studies which, for the greater part, support the classical flow theory.^{3,6,7,11–13} A noticeable exception stands with the experimental investigation of Brutin and Tadriss.⁴ The authors reported up to a 27% excess in Po for distilled and tap water flows in fused silica microtubes of diameters ranging from 530 to 50 μm and interpreted this deviation as the result of electrokinetic effects. Subsequently, Phares and Smedley⁶ calculated that Brutin and Tadriss's⁴ observations were not consistent with the conventional model of the electroviscous effect. Their conclusion was supported by pressure drop measurements performed with water samples of different ionic concentrations flowing in smooth polyimide microtubes from 152 to 119 μm . According to a recent comment of Brutin and Tadriss,¹⁷ additional charge-related surface interactions may account for the observed behavior.

Fewer experiments have been performed for an inner

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TABLE I. Summary of available friction coefficient measurements for laminar liquid flows in smooth microducts:

Authors	Dimensions (μm)	Fluid	Duct	Observations
Pfund <i>et al.</i> (Ref. 10)	521, 263, 128	Water	Polycarbonate/polyimide microchannels	+10% to +20% in Po
Phares <i>et al.</i> (Ref. 6)	152, 119	De-ionized and tap water, saline solution	Polyimide microtubes	Agreement with classical flow theory
Gao <i>et al.</i> (Ref. 11)	500, 400..., 100	Demineralized water	Bronze microchannels	Agreement with classical flow theory
Li <i>et al.</i> (Ref. 12)	205–80	De-ionized water	Glass and silicon microtubes	Agreement with classical flow theory
Brutin <i>et al.</i> (Ref. 4)	530–50	Distilled and tap water	Fused silica microtubes	0% to +27% in Po depending on the fluid's ionic properties
Sharp <i>et al.</i> (Ref. 13)	247–50	Deionized water, 1-propanol, glycerol solution	Glass microtubes	Agreement with classical flow theory
Kohl <i>et al.</i> (Ref. 7)	98–24	Water	Silicon microchannels	Agreement with classical flow theory
Judy <i>et al.</i> (Ref. 3)	150–20	Distilled water, methanol, isopropanol	Fused silica microtubes	Agreement with classical flow theory
Papautsky <i>et al.</i> (Ref. 2)	30, 20	Water	Nickel micropipettes	+10% to +20% in Po
Ren <i>et al.</i> (Ref. 5)	40.5, 28.2, 14.1	De-ionized water and KCl solutions	Silicon microchannels	+0% to +20% in Po
Pfahler <i>et al.</i> (Ref. 1)	53, 1.7, 0.8	N-propanol	Silicon-Pyrex microchannels	–50% to +500% in Po

scale smaller than 30 μm and their results are rather conflicting. Both reduction¹ and increase^{1,2,5} from the conventional value have been reported for the measured Poiseuille number. A physical phenomenon that could possibly influence pressure-driven liquid flows in microstructures is the presence of an electrical double layer (EDL) at a solid/liquid interface. The theory^{18,19} shows that the interaction between pressure-driven liquid flows and electrostatic forces in the EDL leads to an apparent increased fluid viscosity. A necessary condition for the occurrence of a significant electroviscous effect is a low electrokinetic radius ($r_{\text{elec}} = D_h/\lambda_D$, λ_D =liquid Debye length). Since the Debye length of a liquid increases when its ionic concentration decreases, the above condition corresponds to dilute ionic solutions in narrow microducts. Recently, Phares and Smedley⁶ calculated that the electroviscous effect may become significant for de-ionized water flows in microducts smaller than 50 μm . In particular, flows of de-ionized water in tubes 4 μm in diameter are expected to exhibit an apparent viscosity more than two times larger than the fluid bulk viscosity. To our knowledge, only Ren *et al.*⁵ have experimentally detected an electroviscous effect for ultrafiltered de-ionized water flowing in a 14.1- μm -deep microchannel. However, their results have not been confirmed and should therefore be considered cautiously. It is clear from the above review that refined measurements of Poiseuille numbers are still helpful to have a better insight in microscale flows and the related EDL effects.

We present results obtained in well-controlled experiments that allow varying the ionic composition of the fluid and consequently, the electrokinetic radius of the flow in microchannels of very small size (21–4.5 μm in height). The experiments were conducted in large aspect ratio microchannels, made of silicon and Pyrex, and equipped with two local

micromachined pressure probes.²⁰ The advantage upon inlet/outlet pressure measurements is to avoid making assumptions on the minor losses that occur at the channel inlet and outlet. To our knowledge, there has been only one recently published work⁷ on internal measurements in such small structures. The manufacturing of these devices consists of four main steps:

- (i) Two square ($3 \times 3 \text{ mm}^2$) deflecting membranes of about 100 μm in thickness are chemically etched with KOH at 80 °C on the top face of a $15 \times 20 \text{ mm}^2$ silicon substrate, double polished and coated with a 100-nm silicon nitride (Si_3N_4) layer.
- (ii) A 400-nm aluminum layer is then evaporated on the top face; a microchannel, inlet and outlet sumps, and two 30- μm wide pressure taps connecting the membranes to the microchannel are lithographed together in the aluminum layer. These patterns are then reactively ion etched through the aluminum mask.
- (iii) Aluminum and Si_3N_4 are subsequently removed from the top face of the sample. Figure 1 shows a scanning electron microscopy (SEM) view of a sample at this step of the fabrication process. After that, a Pyrex cover is anodically bonded to the silicon wafer in order to cap the channel.
- (iv) Cu–Ni strain gauges, 350-nm in thickness, forming a Wheatstone bridge are sputtered and lithographed on the back side of the deflecting membranes still coated with Si_3N_4 .

Details on the microfabrication principles, the characterization of the integrated pressure transducers and the calibration procedures can be found in Ref. 20. The microdevices were included to an experimental loop composed of a high-

FIG. 1. Micrograph of a silicon substrate (microchannel $n^\circ 1$ connected to a pair of deflecting membranes). Dimensions in Table II.

pressure expansion tank (up to 25 bars), a $0.45\text{-}\mu\text{m}$ filter, a Pt 100 temperature probe for determining the outlet temperature of the fluid, two lock-in amplifiers (NF Electronic Instruments, 5610 B), a high precision pressure transmitter (Keller-33 $\times$, 30 bars, 0.025%) used for the strain gauges calibration procedure, and a high-precision balance (Kern-GS, 410 g, 0.001 g). The accuracy of the micromachined strain gauges was estimated to be $\pm 7.5 \cdot 10^{-3}$ bar. The flow rates were measured by using the classical weighing method and ranged from $2 \times 10^{-6} \text{ l min}^{-1}$ to 0.01 l min^{-1} . The measuring instruments were all connected to a PC microcomputer. The microchannel height was measured in the following two ways: Firstly, a Tencor profilometer determined the engraved depth of the silicon substrate before bonding the Pyrex cap. For each sample, the measurements were done along four transversal lines placed in between the two local pressure taps. Secondly, the microchannels were analyzed by postmortem observations of their transverse cross section. The silicon-Pyrex ensemble was cleaved, sputter-coated with a few nanometers layer of gold, and inspected in a scanning electron microscope (Fig. 2). This verification ensured that the channel geometry was not affected by the anodic bonding operation. Both methods yielded similar results. The Tencor profilometer was also used to examine the surface finish of the silicon engraved stripes and the Pyrex substrate. The roughness height denoted k was found to be less than 5 nm for the Pyrex substrate. It was more variable for the silicon engraved walls: the scans revealed basic irregularities of amplitude smaller than 5 nm and isolated roughness elements of 30 nm in height. The active channel walls were dielectric and probably hydrophilic during the measurements. In fact, the flattening of water drops deposited on both substrates prior to

FIG. 2. Cross-sectional scanning electron microscope view of a silicon-Pyrex microchannel.

TABLE II. Geometrical characteristics of the smooth silicon-Pyrex microchannels ($k < 0.05 \mu\text{m}$).

n°	h (μm)	L (mm)	L_p (mm)	w (mm)	$d\text{Po}/\text{Po}$
1	$20.5(\pm 0.1)$	6	1.5	1	$\pm 7.7\%$
2	$14.3(\pm 0.1)$	6	1.5	1	$\pm 8.3\%$
3	$7.5(\pm 0.2)$	6	1.5	3	$\pm 14\%$
4	$4.58(\pm 0.05)$	2	0.5	0.215	$\pm 9.9\%$

the anodic bonding operation was clearly observed. This is most probably due to the previous immersion of the substrates in nitric acid, which is known to result in superficial coating by a thin layer of hydrophilic SiO_2 . Table II gives a summary of the geometrical characteristics of the different microchannels tested in this work. h is the distance between the Pyrex wall and the silicon wall. L , L_p and w are, respectively, the total channel length, the distance between the two local pressure taps and the width of the channel. The working fluid was stored in the Butyl II-R vessel of an expansion tank. The vessel was entirely filled with water and air was vacuumed out of the whole fluid loop before running the experiments. This prevented gas from being trapped in the pressure probe cavities. To measure very low flow-rates, the outlet reservoir was covered with a finely pierced latex sheet and, in addition, a film of oil sheltered the free surface to limit water evaporation. The very small evaporation rate ($10^{-7} \text{ l min}^{-1}$) was taken into account in the flow-rate determination.

The friction coefficient was calculated from the measured pressure drop ΔP between the two local pressure taps:

$$f = \Delta P D_h / 2 \rho U^2 L_p. \quad (2)$$

The location of the first pressure tap x_1 was chosen to be far from the channel entrance [$x_1^+ = (x_1/D_h)(1/\text{Re}) > 0.2$ in all of the experiments]. Therefore, the results were compared to the fully developed laminar regime in conventional channels:¹⁶

$$f = 24/\text{Re} \quad \text{or} \quad \text{Po} = f\text{Re} = 24. \quad (3)$$

The Po number was deduced from the measured quantities by

$$\text{Po} = 2(\Delta P L_p) / (w h^3 / q \nu), \quad (4)$$

where q stands for the mass flow rate and ν is the kinematic viscosity of the fluid. The physical properties of water were determined at the fluid outlet temperature. A careful analysis of the experimental uncertainties is critical for the determination of apparent deviation from the classical theory. The uncertainties associated with Po were estimated by using Eq. (4). It should be emphasized that the inaccuracy in the evaluation of h is the major source of error in the determination of Po. The uncertainty on ν was 2% ($\pm 1^\circ \text{C}$ for the mean bulk temperature). The uncertainties on both the pressure losses and the mass flow-rate measurements depend on Re and reached 3% for the upper limit of the measuring range. However, more relevant values for the series of measurements were, respectively, 1.5% and 0.75%. The uncertainties on the width and the length of the channel were both estimated at

FIG. 3. Poiseuille number Po vs Re for smooth silicon-Pyrex microchannels ($h=20.5$, 14.3 , 7.5 , and $4.58 \mu\text{m}$). Open symbols: water conductivity $<0.1 \mu\text{S cm}^{-1}$. Solid symbols: water conductivity $=70 \mu\text{S cm}^{-1}$. $\times$: water conductivity $=7 \mu\text{S cm}^{-1}$.

1%. The global uncertainty on the Poiseuille number ranged from $\pm 7.7\%$ to $\pm 14\%$ for the microchannels $n^\circ 1$ – $n^\circ 4$ (Table II). In order to detect a possible electroviscous effect, experiments were first conducted with de-ionized water (electrical conductivity of $<0.1 \mu\text{S cm}^{-1}$) then with a solution of tap water (1/4) in de-ionized water (3/4) (electrical conductivity of $70 \mu\text{S cm}^{-1}$). An equivalent ion concentration I (mol/l), can be deduced from these measurements using the ionic conductance of Na^+ and Cl^- ions. This method yields $-\log([I]) > 6.1$ for the de-ionized water and $-\log([I]) = 3.2$ for the dilute tap water. According to Phares and Smedley's calculations,⁶ significant electroviscous effects (more than 20% increase of the apparent viscosity) may arise in ducts of inner scale smaller than $10 \mu\text{m}$ for water with a negative log of molarity larger than 6 [$-\log([I]) > 6$].

The results presented in Fig. 3 show that Po remains independent of Re at the small scales investigated in this work. Moreover, Fig. 3 clearly demonstrates that the electrical conductivity of water does not play a significant role on the characteristics of the flows studied here. A similar conclusion was drawn by Phares and Smedley⁶ but for microtubes larger than $150 \mu\text{m}$ in diameter. On the other hand, the data presented in Fig. 3 differ from the results of Brutin and Tadrist^{4,17} obtained in microtubes from 530 to $50 \mu\text{m}$ in diameter. A noticeable difference between their experimental facility and the present one is that these authors used high-pressure gas to force water through the fluid loop. Considering the very long duration of their experimentations ("21 h for a 33.4-mm-long $50\text{-}\mu\text{m}$ capillary"⁴), it is likely that the working fluid was saturated with gas. Due to the high-pressure losses occurring in these flows, it is possible that the dissolved gas has expanded to form bubbles in the downstream part of the microtubes where the pressure becomes low, resulting in an artificial increase of the friction losses. In 2001, Ren *et al.*⁵ found a 20% increase in Po for de-ionized water flowing in a silicon microchannel $14.1 \mu\text{m}$ in height compared to aqueous solutions of a higher ionic concentration. Figure 3 shows that we were not able to reproduce their results in silicon-Pyrex microchannels of similar or smaller heights. Differences in the measuring techniques are highly suspected to be responsible for such a discrepancy.⁶

The results of these experiments have proved that the

friction factor is correctly predicted by the classical Navier-Stokes equations at the small scales investigated in this study. No scale effect was found for water flows in rectangular smooth microchannels of height ranging from 21 to $4.5 \mu\text{m}$ and for Reynolds number varying between 0.1 and 300 . In fact, the friction factors measured with two types of water are effectively quite similar. These results suggest that deviations from the conventional theory observed in other experiments with analogous conditions cannot be explained by the electroviscous effect. Moreover, it can be noted that the actual importance of the electroviscous effect is overestimated by Phares and Smedley's calculations⁶ for practical situations where de-ionized water flows in silicon-Pyrex microchannels.

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