Experimental investigation of water-nitrogen flow in microchannel with high aspect ratio (with the 20 µm gap)

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Abstract. This study is devoted to the experimental investigation of two-phase water-nitrogen flow in the slit microchannel with a gap of 20 µm and a width of 10 mm. The technology of microchannel fabrication has been developed and described in detail. Experiments were conducted in adiabatic conditions. Using a modified schlieren system, four flow patterns have been observed and described: jet, bubble, churn, and annular. Flow pattern map was plotted according to obtained patterns. Moreover, a two-phase pressure drop was measured. Dependencies between two-phase pressure drop and superficial liquid and gas velocities have been investigated.

1. Introduction
The development of microfabrication technologies made the microchannels more in demand for different applications. Microchannels are widely used in compact heat exchangers due to their large area-to-volume ratio. Also, microchannels are applied in microfluidic systems for fluid and particles transport and effective mixing in these systems. Moreover, they are applied in biomedical micro-electromechanical systems (MEMS) to imitate the actual biological structures such as capillary vessels [1]. These microchannel applications lead to an increase in the number of publications on this topic over the past 20 years. However, rectangular microchannels with large aspect ratios remain poorly studied. This type of microchannel offers several advantages over square and round channels, such as less pressure drop at the same hydraulic diameter, and a larger surface area to volume ratio [2]. Furthermore, the flow structure differs from round, square, and rectangular channels with a small aspect ratio. The factors affecting the flow structure in different channels are described in the review [3].

Most of the publications in the literature are devoted to channels in the submillimeter range up to 100 µm. Moreover, the work [4] presents data for a circular microchannel with an inner diameter of 20 µm. In the work [5] minimum channels height reached 50 µm in a slit channel. However, decreasing microchannel height leads to certain problems such as an increase in pressure drop in a working range of flow rates and as a consequence – the problem of test section sealing. Therefore, new methods of microchannel manufacturing and sealing are required.
2. Experimental setup and measurements technique

Figure 1. Experimental setup.

For investigation of two-phase flow in 20 µm gap, a complicated design for the test section was developed. The test section consists of two parts: injection frame and flow cell. The injection frame was developed in SOLIDWORKS and printed using 3D-printing DLP technology by ANYCUBIC Photon printer. A more detailed description of 3D-printing methods is presented in the work [6]. For injection fluids, threaded holes have been made and threaded fittings have been tightened. Moreover, at the backside of the injection frame, threaded holes have been made for pressure sensors to measure pressure drop in the two-phase flow region. The flow cell consists of a silicon wafer with etched injection channels, the main two-phase microchannel, and two cover glass plates made of Borofloat 33 borosilicate glass. The overall dimensions of the glass plates are 75x20x2 mm, the silicon wafer is 75x20x0.48 mm.

Figure 2. 3D model of the test section.

The silicon wafer was etched in several stages. First, a photomask was made, then the silicon plate was prepared: a protective chromium mask was sprayed on, then a photoresist was applied, exposed and developed. Then, plasma-chemical etching of silicon was carried out to a depth of 20 µm in an inductively coupled plasma. The etching rate was 1.5 µm/min. After etching, the etching depth was checked on a profilometer. After testing, the mask and photoresist were removed. A detailed description of the features of etching microstructures for microfluidic chips is presented in the work [7].
After etching the microchannels in the silicon wafer, laser cutting of a 20 μm gap was performed to inject liquid perpendicular to the main microchannel. The overall dimensions of the two-phase region are 0.3x10x0.02 mm and those of the gas region are 0.25x10x0.02 mm.

Borofloat 33 cover glasses were preliminarily chemically treated before assembly – the glasses were washed in a toluene/acetone mixture, washed in distilled water, and dried in a thermostat.

![Figure 3. Formation of microstructures in a silicon substrate: a – preparation for etching (1-photoresist, 2-protective chromium mask, 3-silicon substrate), b – exposure of the photoresist through a photomask (4-UV radiation, 5-photomask), c – development of a photoresist, d – removal of the protective mask layer, e – etching of the substrate, f – removal of the mask and photoresist.](image)

For reliable sealing of the cell, the silicon wafer was connected to the cover plates from above and below by thermoanode bonding. Before the start of the sealing process, the holes for the liquid inlet and outlet were pierced in the lower cover plate. Figure 4 shows the bonding principle of silicon-glass thermoanode.

![Figure 4. Thermoanode bonding principle for sealing a silicon wafer with cover glass.](image)

The physical principle of anodic bonding is as follows. For one or another modification of glass properties, certain impurities in the form of oxides are introduced into the quartz mixture. In our case (for Borofloat 33) these were BO3, Al2O3, and sodium and potassium oxides. Under the impact of temperature, partial dissociation of oxides occurs. The latter oxides have the lowest activation energy for this process.

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\begin{align*}
    K_2O & \rightarrow 2K^+ + O^- + 288 \text{ kJ} \quad (1) \\
    Na_2O & \rightarrow 2N^+ + O^- + 299 \text{ kJ} \quad (2)
\end{align*}
\]

Further, under the impact of an electric field, ions are redistributed over the volume. Oxygen moves to the interface with silicon, where it is oxidized, forming a permanent bond with the glass.

Bonding regimes can be very different and depend on the equipment used. In our case, bonding was carried out at 430-450 degrees and a voltage of 800-1000 V and holding for 50 minutes when bonding the upper cover plate with a silicon wafer. Before bonding the bottom plate, the already bonded silicon module and the top glass plate were chemically treated. Further, the two-layer module was bonded with the lower cover glass at higher voltages of 1000-1200 V. Bonding of silicon wafer and cover glass was carried out in clean production rooms (cleanliness class of at least 7 ISO). The sealing area was more...
than 95% percent of the total contact area.

The injection frame and flow cell were connected to each other by gluing with Cosmofen CA-12 cyanoacrylate glue. Figure 2 shows a 3D model of the finished sealed flow cell and connected injection frame.

The experimental setup is shown in Figure 1. High-purity nitrogen was used as a gas. Gas was supplied from a gas tank (1); the flow rate was regulated using a high-precision Bronkhorst flow regulator (2). Milli-Q® water was used as a working liquid. The liquid was injected into the microchannel using a Cole-Parmer® high-precision syringe pump (3). As mentioned above, the test section consisted of two parts: injection frame (4) and flow cell (5). Additionally, in the injection frame, two threaded holes (12, 13) were made for pressure drop measurement. The pressure was measured using industrial BD pressure sensors. To study the gas-liquid interaction, the schlieren method was used, which is described in detail in [9]. Light from a LED source (6) passed through the lens (7) forming a parallel beam. Then the light passed through a beam splitter (8) and the upper plate of the flow cell. After that, the light was reflected from the interface, the light passed through a beam splitter (8) and a lens (10), falling into the camera (11). Thus, the camera captured an image of a two-phase flow, where the interface was clearly visible. The camera and Bronkhorst flow regulator were controlled using a computer (9). The frame rate was 60 Hz. The resulting images were processed in MATLAB®. Colorful images were obtained in the jet, bubble, churn, and annular flow patterns. The following flow characteristics were detected: films on the upper wall of the microchannel, dry areas, and liquid bridges.

3. Results and discussion
At low superficial velocities of gas and liquid, a jet flow pattern was observed. This pattern is characterized by separated flows of liquid and gas in the form of jets. The jets alternate over the entire width of the channel, with gas flowing predominantly along the lateral parts of the channel, while liquid predominantly occupies the central part of the channel. Figure 5 demonstrates the schlieren image of jet flow pattern at \( U_{sl} = 0.083 \text{ m/s} \) and \( U_{sg} = 0.583 \text{ m/s} \) respectively.

![Figure 5. Jet flow pattern at \( U_{sl} = 0.083 \text{ m/s} \) and \( U_{sg} = 0.583 \text{ m/s} \).](image)

At high superficial velocities of liquid and low and high superficial velocities of gas, a bubble flow pattern was observed. This pattern is characterized by bubbles, whose size depends on the ratio between liquid and gas superficial velocities. Also, interference patterns were observed which means that the observed films were about 0.5 µm […] Upper film on the bubbles can drain during the flow due to poor wettability. Figure 6 demonstrates the bubble flow pattern at \( U_{sl} = 0.116 \text{ m/s} \) and \( U_{sg} = 0.125 \text{ m/s} \) respectively.

![Figure 6. Bubble flow pattern at \( U_{sl} = 0.116 \text{ m/s} \) and \( U_{sg} = 0.125 \text{ m/s} \).](image)

At high superficial velocities of liquid and middle superficial velocities of gas, a churn flow pattern was observed. This pattern also was observed at low superficial velocities of liquid and high superficial velocities of gas. This pattern is characterized by merged bubbles, separated by horizontal fluid bridges of arbitrary shape. Figure 7 demonstrates churn flow pattern at \( U_{sl} = 0.333 \text{ m/s} \) and \( U_{sg} = 1.66 \text{ m/s} \).
respectively.

**Figure 7.** Churn flow pattern at $U_{sl}=0.333$ m/s and $U_{sg}=1.66$ m/s.

At high superficial velocities of liquid and gas, an annular flow pattern was observed. This pattern is characterized by film closed along the cross-section of the channel. This film also can drain during the flow due to poor wettability. The film also was separated by liquid bridges. Figure 8 demonstrates annular flow pattern at $U_{sl} = 0.333$ m/s and $U_{sg} = 5$ m/s respectively.

**Figure 8.** Annular flow pattern at $U_{sl}=0.333$ m/s and $U_{sg}=5$ m/s.

Figure 9 shows a flow pattern map, where $U_{sl}$ was chosen as abscissa and $U_{sg}$ as ordinate. Figure 10 (a) and (b) show the dependencies between pressure gradient and superficial velocities of liquid and gas respectively.

**Figure 9.** Flow pattern map.
Figure 10. Pressure gradient versus superficial liquid velocity (a) and gas velocity (b).

It is clearly seen that pressure drop increases almost linearly with increasing liquid superficial velocity. However, at a fixed superficial velocity of liquid, with increasing gas superficial velocity, the same trend was not observed. In this case, pressure increases more sharply at low superficial velocities of gas.

4. Conclusion
For the first time, an investigation of two-phase flow in the slit microchannel with a gap of 20 µm has been carried out. To investigate this channel, a new concept of the test section was offered, implemented, and described. The technology of thermoanode bonding allowed creating a hermetically sealed permanent connection between covered glasses and microstructures plate designed for high pressures. By using the schlieren optical system, jet, bubble, churn and annular flow patterns have been identified. All patterns have been described. The dependencies between measured pressure gradient and gas and liquid superficial velocities have been investigated. It is shown, that pressure drop increases almost linearly with increasing liquid superficial velocity. At a fixed superficial velocity of liquid, with increasing gas superficial velocity, pressure gradient increases more sharply at low superficial velocities of gas.

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