Technology for cleaning glass substrates in glow discharge plasma for microfluidics applications

D.M. Rabotyazheva

Abstract. The production of microfluidic chips is increasing every year because the technology using the chip allows you to make an express test to obtain the analysis, as well as economically viable. Often microfluidic chips are based on glass in which the microchannels are formed. Accurate production of a microfluidic device requires careful preparation of the glass substrate to improve adhesion for further application of functional layers. The choice of cleaning mode strongly affects the output product, so a clear understanding of what factors affect the quality of cleaning in HF plasma is needed. Mathematical modeling of dependence between plasma power of the glow discharge, time and gas medium first of all gives understanding which cleaning mode is optimal and which parameter affects stronger very relevant for development of this potentially promising technology of quality products.

1 Introduction

In the current development of micro-, nano- and microelectronics, contamination of substrate surfaces is becoming commensurate with the size of the micro- and nanostructures being formed, which leads to the need for thorough cleaning and tight control over the degree of surface cleanliness.

Microfluidic chips have quickly gained widespread adoption at the point of care, offering advantages of smaller sample volume, fast detection, high throughput, low contamination and easy integration.

Microfluidic chips often consist of a polymer cap assembly and a glass substrate with microchannels (Fig. 1), produced by photolithography and wet etching. Therefore, it is important to prepare the glass substrate before applying the functional layers.

*Corresponding author: pryanichnikovadm@student.bmstu.ru

© The Authors, published by EDP Sciences. This is an open access article distributed under the terms of the Creative Commons Attribution License 4.0 (https://creativecommons.org/licenses/by/4.0/).
The implementation of plasma treatment in microfluidic devices represents a promising approach to the development of compact, relatively inexpensive, sensitive and effective diagnostic tools for clinical practice.

2 Materials and methods

Plasma treatment of glass is currently a common method in various fields of industry [1]. Plasma treatment can achieve the best cleaning of surfaces compared to other methods. An important element of research for a microfluidic chip is the preparation of a glass substrate prior to subsequent operations [2], developed at the Department of ME11 of the Bauman Moscow State Technical University. An experiment, using the MSS plasma unit (Fig. 2) and a goniometer to measure the wetting angle (Fig. 3), deriving a mathematical model to determine the best processing mode within the ME11 laboratory, showed good results for their subsequent use.

Fig. 1. Schematic diagram of a microfluidic chip

Fig. 2. Purification scheme in glow discharge plasma: 1 - substrate, 2 - electrode, 3 - plasma, 4 - gas injection, 5 - pumping
Thus, the plasma treatment of glass substrate surface before coating in vacuum leads not only to removal of surface impurities, such as organic impurities, oxides and physical inclusions, but also to modification of surface properties towards increasing its adsorption and adhesion activity, i.e. to creating active adsorption and adhesion centers of atomized particles on substrate surface, and to surface hardening [3]. The most effective way to prepare the glass substrate surface for subsequent operations in a vacuum chamber is the method combining degreasing and glow discharge treatment. Alcohol was used as a solvent for degreasing. As a result of desorption, the contaminant molecules pass from the surface to be cleaned into the near-surface liquid layer and then diffuse into its volume.

Contaminants on the surface of substrates are classified, as a rule, according to their physical and chemical properties:

- Physical contaminants (dust particles, lint, abrasives, silicates, silica dust and other foreign particles);
- Contaminants chemically unrelated to the surface of wafers and substrates;
- Contaminants chemically bound to the surface of wafers and substrates (oxides, nitrides and other compounds);
- Organic contaminants (fats, oils, silicones and other impurities);
- Water-soluble contaminants (salts, acids, etchant residues, fluxes, etc.);
- Gases adsorbed by the surface of wafers and substrates.

Various types of contaminants can be present on the surface of the wafers at the same time. The most difficult to remove are organic and chemically bound contaminants, as well as contaminants from abrasive materials, polar gases and ions embedded in the surface layer of the plates.

Using a pure gas environment is not the optimal cleaning method, so a mixture of gases should be created in a vacuum chamber. Argon is an inert and chemically neutral gas and is effectively used for physical cleaning. The oxygen environment is good for removing organic contaminants, resulting in gaseous compounds that are evacuated by the vacuum chamber. The mixture of oxygen and argon gases will not only physically dislodge contaminants, but also chemically clean the surface from contaminants.

Analysis of scientific articles has shown that the parameters of the glow discharge plasma on which the efficiency of substrate cleaning depends are:

- Processing time;
- Discharge power;
- Residual gas pressure in the chamber;
- Location of the substrate (this determines the treatment zone);
- Composition of the gas medium.

These parameters are related quantities that interact with each other.
3 Results

The main purpose of the experiment is to develop a mathematical model adequately describing the influence of process parameters on the value of the wetting angle. Simulation of the selected process is necessary to determine which of the input factors most significantly affect the output parameter, and which influence the output parameter to a lesser extent.

Table 1. Results of glass cleaning on the MSS unit

<table>
<thead>
<tr>
<th>№</th>
<th>Time, sec</th>
<th>Power, Watt</th>
<th>Ar concentration in O₂, %</th>
<th>Angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>60</td>
<td>100</td>
<td>15</td>
<td>10,4</td>
</tr>
<tr>
<td>2</td>
<td>180</td>
<td>100</td>
<td>15</td>
<td>4,9</td>
</tr>
<tr>
<td>3</td>
<td>60</td>
<td>200</td>
<td>15</td>
<td>8,3</td>
</tr>
<tr>
<td>4</td>
<td>180</td>
<td>200</td>
<td>15</td>
<td>13,9</td>
</tr>
<tr>
<td>5</td>
<td>60</td>
<td>200</td>
<td>30</td>
<td>12,4</td>
</tr>
<tr>
<td>6</td>
<td>180</td>
<td>100</td>
<td>30</td>
<td>7,2</td>
</tr>
<tr>
<td>7</td>
<td>60</td>
<td>100</td>
<td>30</td>
<td>12,9</td>
</tr>
<tr>
<td>8</td>
<td>180</td>
<td>200</td>
<td>30</td>
<td>4,3</td>
</tr>
</tbody>
</table>

The pressure in the working chamber was set depending on the stable operation of the generator and monitored by a wide-range sensor. Power was adjusted and set on the generator unit. Concentration of working gas was set by the gas flow regulator and controlled by the control scale on it.

It should be noted that the studied parameter in this experiment is the wetting angle of the substrate surface. The treated surface is inherently hydrophilic (Θ<90°). A quantitative measure of surface wettability is the wetting angle (Fig. 4) - the angle between the tangent to the surface of a liquid drop at the point of contact of the three phases (solid, liquid and gaseous) and the surface of a solid, measured inside the liquid phase [5].

The wetting angle was measured using a goniometer. The procedure for measuring the wetting angle is as follows: the sample under study is placed on the goniometer slide, a drop of distilled water is applied to the sample surface using a special syringe [4].

Fig. 4. Drop profile projection

For better understanding of the relationship between the factors under study and the output parameter of the cleaning quality dependence on the composition of the gas medium, time and power of low-pressure HF plasma, a mathematical model was made:

\[ y = 9.2903 - 1.7022X_1 + 2.2452X_2 - 0.0909X_3 + 0.9789X_1X_2 - 1.7535X_1X_3 - 1.7535X_2X_3 - 1.7949X_1X_2X_3 \]

where

- \( X_1 \) - normalized substrate processing time of 60 to 180 seconds;
- \( X_2 \) - normalized percentage of Ar in the O₂ gas medium from 15% to 30%;
- \( X_3 \) - normalized power of HF discharge from 100 watts to 200 watts;
- \( y \) - wetting angle.
Also, an analysis of the ion purification rate from the ion current density was carried out, which gives a direct understanding of the interaction of these parameters. Ion purification is realized using argon Ar+ noble gas ions, which atomize atoms of the target material due to the high (over 1 keV) energy of the ions.

In ion purification, the purification rate (physical atomization of the material) is:

\[
\rho \frac{Ae}{ICNqSMjV} = \rho
\]

where:
- \( j_i = 12,1..16,1 \, \text{А}/\text{m}^2 \) – ion current density;
- \( S = 1 \, \text{atom/ion} \) – atomization factor;
- \( M = 40 \, \text{kg/kmol} \) – molar mass;
- \( q_e = 1,6 \cdot 10^{-19} \, \text{Кл} \) – electron charge;
- \( N_A = 6,02 \cdot 10^{26} \) – Avogadro number;
- \( \rho = 2500 \, \text{kg/m}^3 \) – density of the material.

The calculation is as follows:

\[
V_{IC} = \frac{j_iSM}{q,N_A \rho}
\]

\[
V_{IC} = \frac{j \cdot 1 \cdot 40}{1,6 \cdot 10^{-19} \cdot 6,02 \cdot 10^{26} \cdot 2500} = 0,166 \cdot 10^{-9} \, j \, \text{m/sec} = 0,166 j \, \text{nm/sec}
\]

The density of the ionic current is determined by the formula:

\[
j = \frac{I}{S}, \text{А}/\text{m}^2
\]

where the current strength varied from 0.75 to 1 A; \( S=0,062 \, \text{m}^2 \) – cross-sectional area. Based on the formula, ion current density will be determined by the interval from 12,1 to 16,1 А/м².

On the basis of calculation, a graph of ion cleaning in glow discharge plasma was plotted:

**Fig. 4.** Graph of ionic purification

### 4 Conclusion

As a result of investigation of the influence of low-pressure HF plasma on the surface of glass substrates, eight variants of MSS unit operation modes were selected, based on their...
results, a mathematical model of the surface wetting angle was derived. From the experimental data obtained, we can conclude that the smallest angle of wettability of the surface has the mode at 30% argon in oxygen medium, the maximum power of 200 watts delivered to the electrodes and a time of 180 seconds. At these process parameters, the wetting angle is 4.3°.

Also, it can be noted that mode 4 at maximum power and time, but minimal argon content in the oxygen environment indicates that the physical knockout of particles has a greater impact on the surface to be treated.

5 Acknowledgements

The research was carried out as part of the ME11 research project at the MSS facility.

References


3. А. Г. Лучкин, Г. С. Лучкин, Cleaning the surface of substrates for coating by vacuum-plasma methods, UDC 533.599, 621.793, 539.23.


5. В. А. Софир, Н. Л. Казанский, В. А. Колпаков, А. И. Колпаков, В. В. Полипин, Method of measuring substrate surface purity, No 2380684 (2008)
