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Characterization of the ionization process inside a miniature glow-discharge micropump

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Abstract. The article presents a detailed characterization of the ionization process taking place inside a miniature MEMS-type ion-sorption vacuum pump, based on the Penning pump architecture. Influence of a variety of parameters on the discharge current has been investigated. These include: magnetic field, type of material used for the electrodes as well as horizontal and vertical dimensions of the micropump. It was found that the micropump works efficiently as long as the magnetic field is higher than 0.3 T, and the pumping cell's dimensions exceed $1\times1\times1$ mm^3. Best results have been obtained for copper and aluminum cathodes and low resistivity silicon anodes.

Key words: ion-sorption micropump, MEMS-type micropump, gas discharge, ionization.

1. Introduction

Different types of ion-sorption pumps have been developed and manufactured over the years, most intensively in the last century [1–3]. In this type of pumps, gas particles are ionized and absorbed by a sputtered getter material, usually titanium. The ionization process takes place during the collisions of gas molecules with high energy electrons. The biggest challenge is to ionize gases in high vacuum, in which free electron path becomes significantly larger as compared with pump dimensions (even above few kilometers) and the probability of electron-gas particle collisions decreases. To solve this problem, in most types of ion-sorption pumps electrons are forced to oscillate inside a confined volume, which is usually done by means of proper electric field configuration or by utilizing a magnetic field.

The most popular ion-sorption pump is the so-called Penning pump [4]. It consist of two planar titanium plates working as cathodes and a cylindrical anode located between the cathodes in honeycomb architecture.

The electrodes are placed in a strong magnetic field. In each pumping cell a gas discharge is ignited. Electrons cruise around due to the presence of the Lorentz force and ionize gas particles. Thus generated ions hit cathode surfaces and cause secondary electron emission, which leads to a self-continued process of gas discharge. During the bombardment of high energy ions, cathode material is sputtered. In the middle of the cathode the material is removed – a fresh, unpassivated, highly reactive layer is uncovered, and it can adsorb chemically active molecules. Sputtered atoms are deposited onto the periphery of the cathode and onto the anode, where they can additionally cover up gas molecules and atoms of noble gases.

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A lot of research has been done on characterizing these pumps and on improving their properties, for example eliminating argon instability (problem of reversible pumping of noble gases [5]). Penning pumps are now very efficient, commercially available and often used as the last stage of a pumping system which allows generating ultra-high vacuum of up to 10^{-11} hPa [6]. It appeared that no more research in this field was required but the situation changed a few years ago, when a new possible application of the ion-sorption pumps was proposed.

It was noticed that there is a serious problem with providing stable high vacuum conditions for miniature vacuum devices (vacuum MEMS, vacuum nanoelectronics devices) [7–9]. In a very small volume ($V > 1$ cm^3, large surface to volume ratio) due to outgassing, degassing and leakages, one cannot obtain high vacuum [10], and the use of getters only partially solves the problem [11]. The chance of generating high vacuum was seen in miniaturization of ion-sorption pumps. The first concept of a miniature orbitron pump was proposed by Koops in 2004 [12] and some attempts of practical construction of miniature ion-sputter pumps were presented by the group from University of Michigan [13, 14], but the first really working miniature ion-sorption pumps were only developed by the authors of this article in 2013–2014 [15–17]. Up till now, best results have been obtained by the use of the so-called glow-discharge micropump, partially based on the Penning pump (Fig. 1a) [17].

The micropump consisted of silicon electrodes (covered with a thin Ti layer) separated by borosilicate glass spacers. Magnetic field was introduced by two small permanent magnets located outside the silicon-glass structure. Typical size of conventional Penning pumps including large magnets and housing can reach several centimeters in each dimension, and the pumping cell is a few centimeters wide and a few centimeters high [6]. In comparison, the total size of the newly elaborated micropump is $20\times12\times10$ mm^3, including magnets, and the micropump cell is $5\times5\times3.4$ mm^3 [17]. Magnetic flux

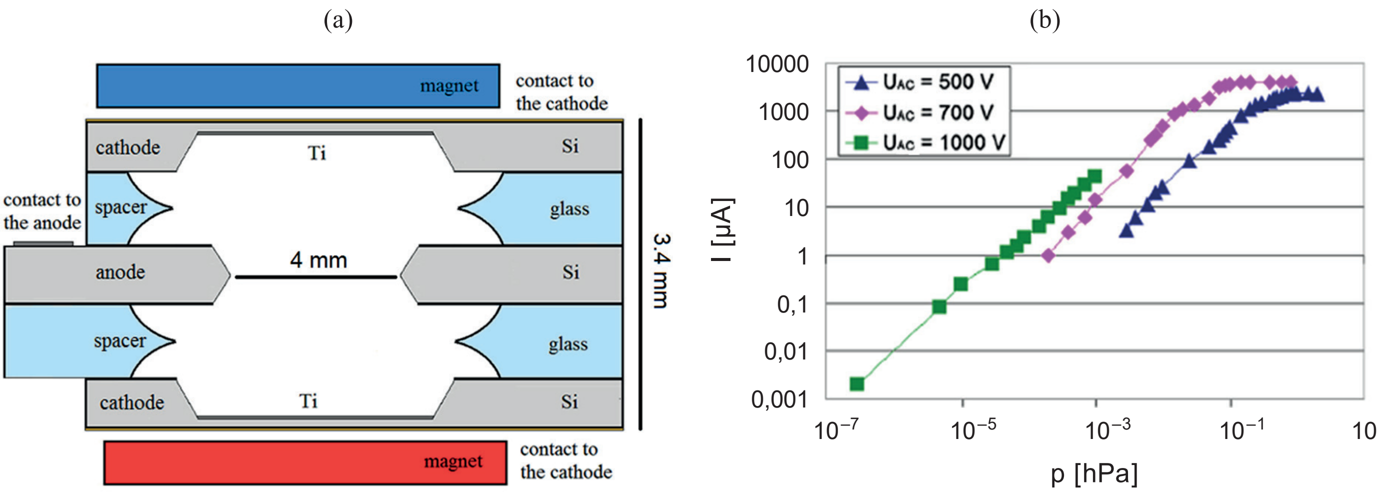

Fig. 1. Glow discharge ion-sorption micropump: a) construction (not in scale), b) relation between discharge current and pressure for different bias voltage, measured in a reference vacuum chamber [17]

density B in large pumps equals about 0.2–0.3 T, and inside the micropump it can be as high as 0.6 T, although much smaller neodymium magnets are used (weight: 2 g, as compared with even a few kilograms). This is attributed to the fact that the slit between them is much smaller.

The basic characterization of the micropump has already been done. The glow discharge inside the pump is ignited by spontaneous electrons and maintained by the avalanche ionization process. During electron-particle collisions positive ions and additional electrons are created, which can ionize other particles. Discharge current has a value from several milliamperes in low vacuum (~1 hPa) to several nanoamperes in high vacuum (Fig. 1b). In about 30 minutes an encapsulated structure (microchamber volume of 0.08 cm^3) can be pumped down to the pressure of about 10^{-7} hPa.

However, many questions were left without an answer. As for large-scale Penning pumps, more research had to be conducted also for the micropump. In this article, we present detailed characterization of the elaborated device, showing the influence of different (geometrical, electrical, material, etc.) parameters on its operation. It is worth underlining that this is the first time when the ionization process taking place in a microcavity in medium and high vacuum is thoroughly examined.

2. Results and discussion

Many different parameters of the recently elaborated micropump have been changed and their influence on its properties has been measured. First of all, minimum magnetic field necessary to confine charged particles and to ignite a glow discharge has been determined as a function of pressure. The influence of cathode and anode material on the pump operation has also been examined. Moreover, it has been investigated what is the minimum size of the pump chamber is and it has been determined whether there is a limit to its miniaturization.

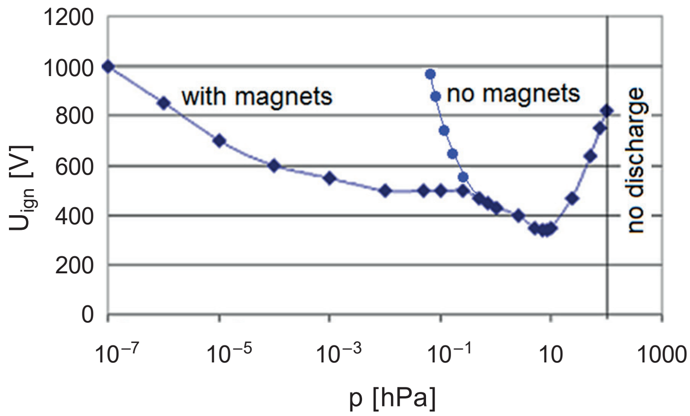

Fig. 2. Relation between ignition voltage and pressure, measured in a reference pressure chamber with and without external magnets (0.6 T)

2.1. Influence of a magnetic field on micropump properties. Magnetic field is one of the most important parameters influencing micropump operation. Without it, any discharge inside a pumping cell cannot be ignited below 10^{-1} hPa (Fig. 2a). At this pressure a free path of electrons becomes similar to the inter-electrode distance, and collisions of electrons with gas particles are less frequent. Mechanism of increasing their path must be applied and this is ensured by introducing the magnetic field.

The larger the magnetic flux density, the smaller the electron orbit and longer the trajectory, which leads to much frequent collisions. The critical value is obtained when diameter of the orbit is smaller than the diameter of the pumping cell. This parameter could be derived from the equation on Larmor radius[1], but one needs to take into an account also the presence of a cloud of positive and negative charges and changes of the

[1] $r = \sqrt{\frac{2m}{q}}\frac{\sqrt{V}}{B}$, where m is a mass of a particle and q its charge, V – applied voltage, and B – magnetic flux density

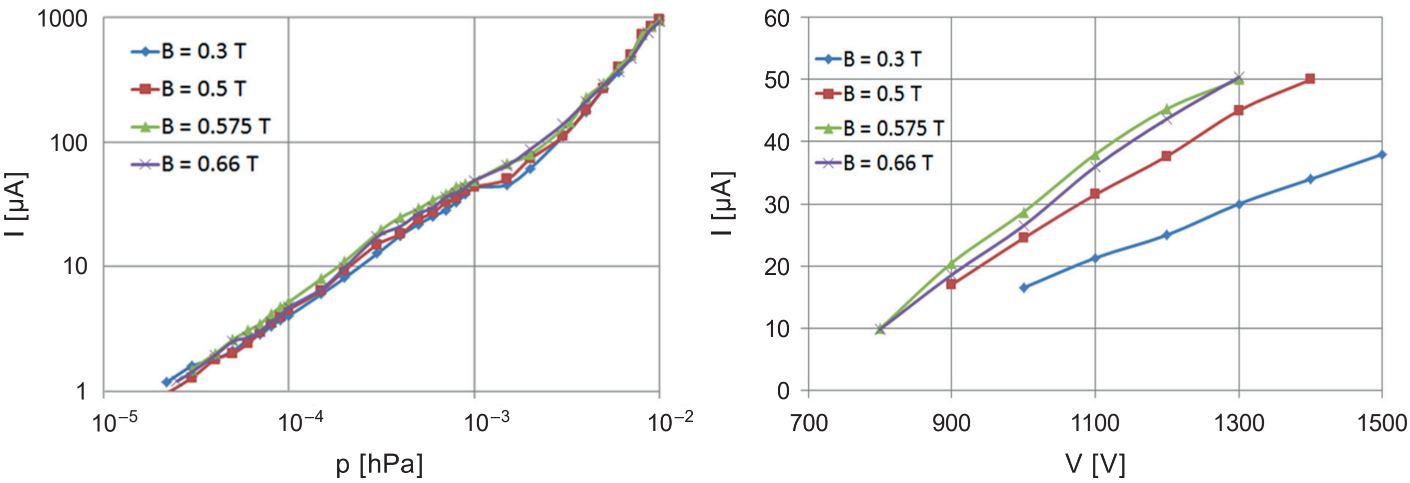

Fig. 3. Influence of magnetic flux density on micropump ion current: a) as a function of pressure; V_{AC} = 1 kV, b) as a function of anode-cathode voltage; p = 5×10^{-4} hPa

electron path caused by collisions, which makes simple equations more complex, and exact value more difficult to predict. However, the critical magnetic flux density can be determined more easily in practice.

Different neodymium magnets (providing different magnetic fields, from 0.19 to 0.66 T) were placed on both sides of a microchamber and the current *vs* pressure characteristics were then measured (Fig. 3). For the two smallest magnets (0.19 and 0.23 T), it was impossible to ignite the discharge below 5×10^{-3} hPa, even though voltage was increased up to 2 kV. To obtain almost proportional *I-p* curve in wide pressure range, magnetic flux density had to exceed at least 0.3 T (in this case even lower voltage, i.e. 1kV, was sufficient to maintain a discharge). The field equal to 0.3 T can be treated as the critical value, but small changes in characteristics appeared also above this value. For two weaker fields (0.3 and 0.5 T) a slight deviation of *I–p* curve from linearity occurred above 1×10^{-3} hPa and currents for the same applied voltages presented on the I–V curves were 1.5–2 times lower than for higher fields (0.575 and 0.66 T). Taking into account that larger magnetic fields require larger magnets, the best compromise is to choose the ones which ensure 0.5 T. They are 8 mm wide, 3 mm thick, and weigh 1 g. Exactly these magnets were utilized in all other measurements.

2.2. Influence of the dimensions on micropump properties. In the second experimental stage micropump cells of various dimensions were tested. The first geometrical parameter was the thickness of the glass spacer – it determined the total pumping cell height, since the anode and cathodes were always made of 400 μm thick silicon substrate. Current-pressure characteristics were measured for spacer thickness from 0.3 to 1.1 mm at constant anode-cathode voltage of 1 kV (Fig. 4).

The biggest difference in *I–p* characteristics was noticed between the spacers smaller and larger than 0.7 mm. If the distance was too small, voltage of 1 kV was insufficient to maintain a discharge in high vacuum – for 0.5 mm the current disappeared at 7.5×10^{-4} hPa and for 0.3 mm – at 2×10^{-3} hPa. However, even in this case it was possible to ionize gas particles but with at least 1.2 kV (Fig. 5). For wider spacings gas discharge was observed in the whole measurement range (V_{AC} = 1 kV). The *I–p* curves did not change much between 0.7 and 1.1 mm thick spacers, but slightly higher current values were noticed for smaller distances. This fact can be attributed to the changes of magnetic field inside the structure. In all experiments the same magnets (3 mm thick) were used, but with decreasing the distance between the electrodes, magnetic flux density slightly increased.

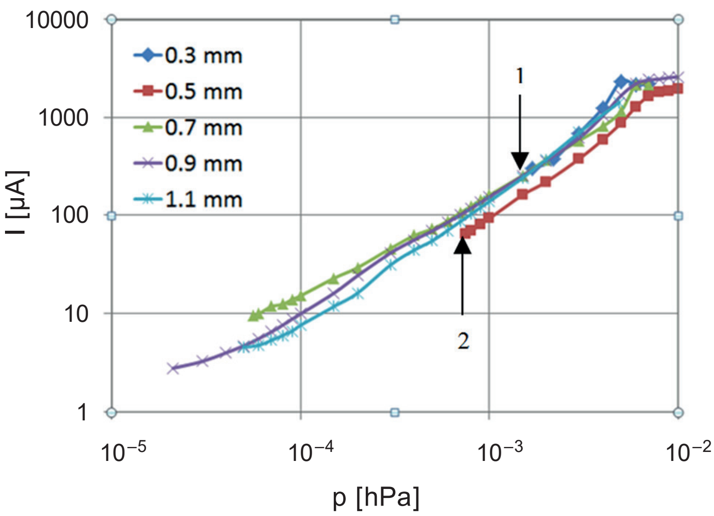

Fig. 4. Influence of glass spacers thickness on micropump ion current as a function of pressure; VAC = kV, arrows 1 and 2 show the pressure for which the discharge current becomes extinguished

The second essential geometrical parameter (other than spacer thickness) is the size of the central square hole made in the anode. It is an equivalent of the diameter of the cylindrical anode in classic Penning pumps. Test structures with holes of

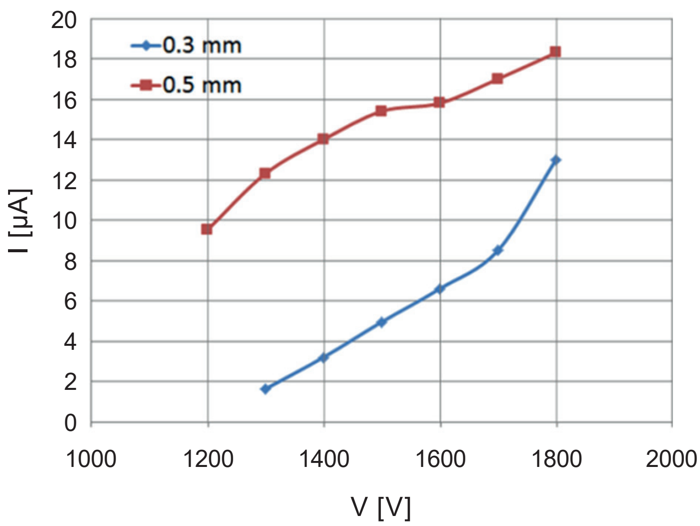

Fig. 5. Influence of spacer thickness on the value of micropump ion current as a function of anode-cathode voltage; p = 6×10^{-5} hPa

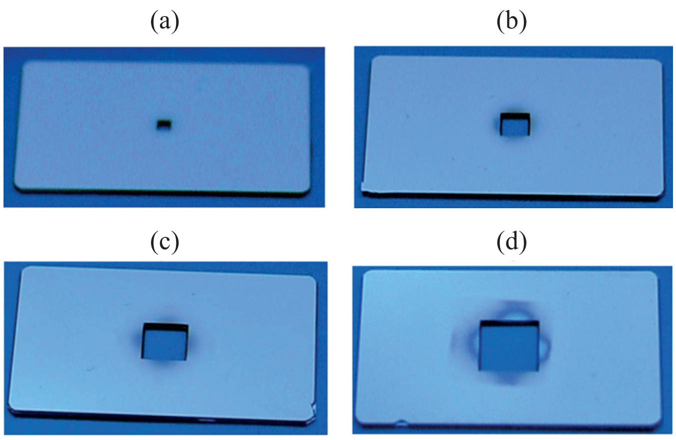

Fig. 6. Silicon anodes with different square hole sizes: a) 1×1 mm^2, b) 2×2 mm^2, c) 3×3 mm^2, d) 4×4 mm^2; material deposited near the hole edges during micropump operation is visible

1×1, 2×2, 3×3 and 4×4 mm^2 in size have been manufactured and tested in the reference vacuum chamber (Fig. 6).

The results obtained were quite surprising. It appeared that the best results were not obtained for the largest hole, but for the smaller ones, i.e. those sized 3×3 mm^2 and 2×2 mm^2 (Fig. 7).

The 2×2 mm^2 anode also exhibited the highest currents in the high vacuum range and can thus be treated as an optimum structure. The explanation for this can be attributed to the distribution of the electric field – it is the most symmetric when horizontal and vertical dimensions of the pumping cell are similar.

Another interesting observation is the shift in *I–p* curve for the anode with the 4×4 mm^2 hole. Current is proportional to pressure in lower and higher vacuum ranges, but a sudden jump in current value appears at about 4×10^{-4} hPa. Glow inside a discharge cell increases at this point and covers a large part of the pumping cell. This behavior appeared repeatable in subsequent measurements. This can be attributed to some changes in electromagnetic field and space charge distribution, but this effect must be studied more carefully in future experiments.

Current-pressure curve for the 1×1 mm^2 hole exhibits the lowest current values, but the relation is proportional. The current-voltage characteristics for this structure are more interesting (Fig. 8). Many repeatable local minima and maxima are visible on the curves for all the pressures examined. Voltage ranges with negative plasma resistivity appear. This must be attributed to the fact that for a certain combination of magnetic and electric field, and geometrical parameters of the microchamber the conditions become more or, alternatively, less favorable for maintaining an electric discharge.

2.3. Influence of the cathode material on micropump properties. In addition to the impact of geometrical parameters on micropump performance, material properties have also been examined. First, cathode material was tested. We expected that different materials will emit different numbers of secondary electrons, and will sputter to a different degree. Moreover, different materials may react chemically with various gases and have different absorption ratios.

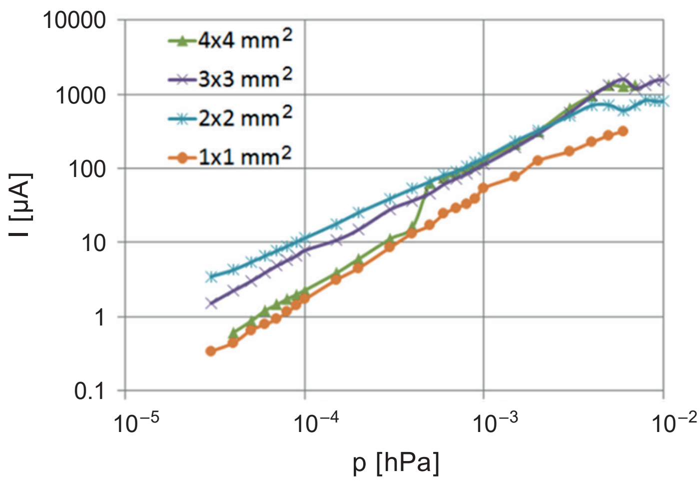

Fig. 7. Value of the micropump ion current as a function of the pressure for four sizes of the hole in the anode (V_{AC} = 1 kV)

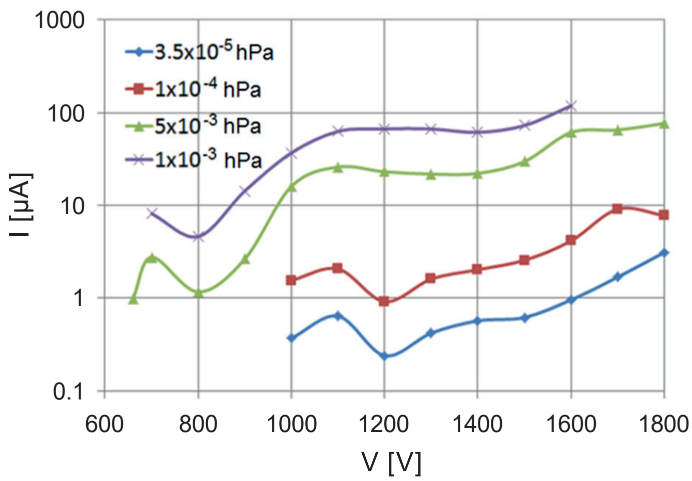

Fig. 8. Micropump ion current as a function of anode-cathode voltage for the 1×1 mm^2 hole in the anode; glass spacer thickness: 1.1 mm

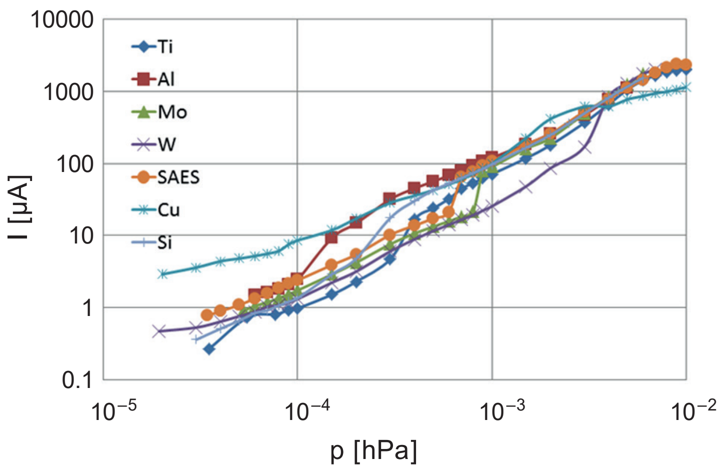

Fig. 9. Micropump ion current as a function of pressure for various cathode materials; $V_{AC} = 1$ kV, anode with 4×4 mm^2 hole, glass spacer thickness: 1.1 mm

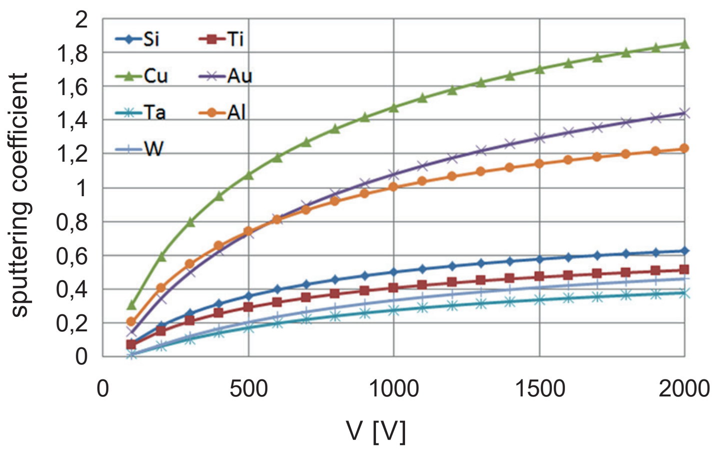

Fig. 10. Sputtering coefficient for different materials as a function of voltage

Measurements showed that the *I–p* characteristics obtained for various materials did not differ significantly (Fig. 9).

Current-pressure curves look similar, mostly in the low and high vacuum range. They are more or less proportional, but a sudden shift in current value can be noticed between 1×10^{-4} and 5×10^{-3} hPa. It occurs at different pressure for different materials. Nevertheless, the values change not more than 1 order of magnitude.

The highest values of the current were obtained for copper and aluminum layers and lowest – for tungsten and titanium. This correlates quite well with sputtering coefficients of these materials (Fig. 10) [18] – the more efficient the sputtering, the higher the current. Along with the gas molecules, the sputtered atoms can also take part in the discharge. The effects of sputtering are visible also on cathode surfaces. Some of them were sputtered to a higher degree and some to a lower one (Fig. 11). For copper and gold cathodes within 30 minutes of micropump operation we could even observe short circuits caused by covering the glass spacers surface with a thin metallic layer.

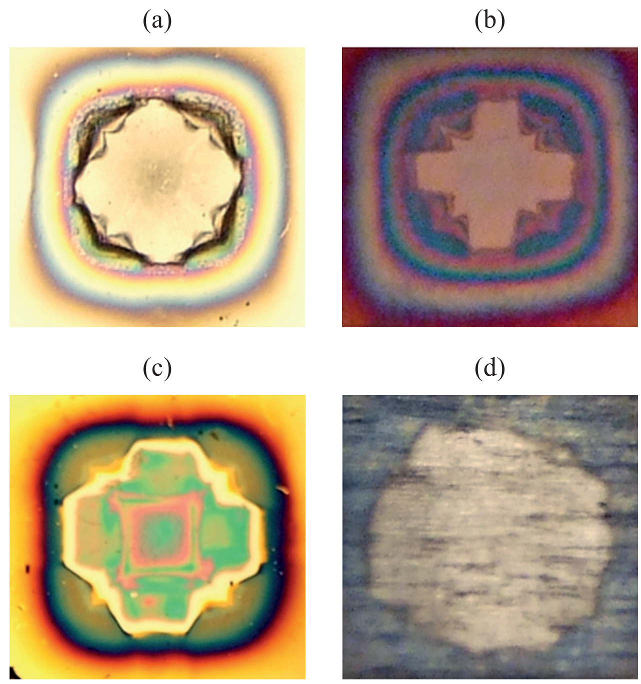

Fig. 11. Images of cathode surfaces after about 30 minutes of micropump operation: a) made of silicon, b) made of copper, c) made of gold, d) made of tungsten; $V_{AC} = 1$ kV, anode with 4×4 mm^2 hole, glass spacer thickness: 1.1 mm

2.4. Influence of silicon anode resistivity on the ion current. It was believed that the most important parameter would be the cathode material, because this electrode is bombarded with the ions and most of the electrons are generated there. The role of the anode is only to draw the electrons, which do not have sufficient mass to sputter the material.

To verify this fact, two types of silicon anodes were examined, one made of high resistivity ($>1\ \Omega \cdot$cm) and one made of low resistivity ($0.001\ \Omega \cdot$cm) substrates (Fig. 12). Surprisingly, it appeared that the differences in characteris-

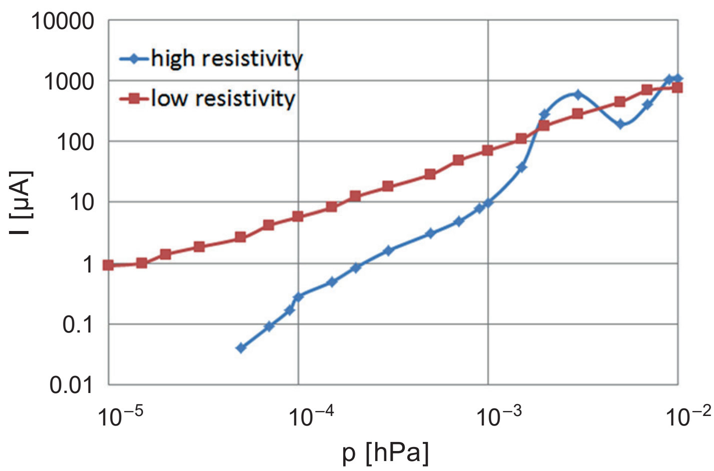

Fig. 12. Comparison between high and low resistivity anodes; $U_{AC} = 1$ kV, anode with 4×4 mm^2 hole, glass spacer thickness: 1.1 mm

tics in both cases were significant. For the same voltage and pressure (VAC = 1 kV, p = 5×10^{-5} hPa), the discharge current changed about 50-fold. Especially in the high vacuum range, the high resistivity samples exhibited completely unsatisfactory performance – they would only work in medium vacuum. The dramatic difference between the two types of anodes is difficult to explain. It is unlikely to be attributed to serial resistance and voltage drop across the wafer, because it is 70 times smaller than serial resistance used in the circuit during measurements (100 kΩ), and total voltage drop should not exceed in high vacuum 1 V. This behavior needs further examination.

After noticing that anode resistivity plays such a significant role, two more aspects were investigated. Does it matter if n-type or p-type silicon is chosen, and how they behave as compared with metallic anodes? However, this time differences reached only a few percent, so the effect is negligible. The second question was if cathode resistivity is also important, but also in this case the influence was very small.

3. Conclusion

This article describes the results of a miniature ion-sorption pump characterization. The influence of magnetic and electric field as well as of geometrical and material parameters of the pump on the ionization process has been carefully investigated.

It appeared that to provide sufficient conditions to ignite and maintain a discharge inside the micropump cell, four factors are crucial – applied voltage, which should be adjusted to the pressure level (not smaller than 400 V), magnetic flux density, which should be higher than 0.3 T, type of material used for the anode, which must have low resistivity, and the size of the hole in the anode, which should be at least 1×1 mm^2. Otherwise the micropump cannot work efficiently in high vacuum. The other parameters can be arbitrarily adjusted. It is possible to apply any type of cathode material in the micropump, but to obtain the highest efficiency it is better to choose a material with a high sputtering coefficient and a high ability of chemical and physical adsorption (like Cu or Al). It is possible to use any thickness of spacers, but the best results are obtained for at least 0.7 mm. Finally, the exact size of the hole in the anode and the exact value of the magnetic flux density is not crucial, but a 2×2 mm^2 hole and a 0.5 T field ensure the highest value of ion currents.

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