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Electron optics column for a new MEMS-type transmission electron microscope

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Abstract. The concept of a miniature transmission electron microscope (TEM) on chip is presented. This idea assumes manufacturing of a silicon-glass multilayer device that contains a miniature electron gun, an electron optics column integrated with a high vacuum micropump, and a sample microchamber with a detector. In this article the field emission cathode, utilizing carbon nanotubes (CNT), and an electron optics column with Einzel lens, made of silicon, are both presented. The elements are assembled with the use of a 3D printed polymer holder and tested in a vacuum chamber. Effective emission and focusing of the electron beam have been achieved. This is the first of many elements of the miniature MEMS (Micro-Electro-Mechanical System) transmission electron microscope that must be tested before the whole working system can be manufactured.

Key words: MEMS, miniature TEM, electron emission, focusing of electron beam, electron optics column.

1. Introduction

Obtaining a focused beam of electrons is an important issue in developing miniature electron optics devices such as miniature X-ray generators [1], miniature electron lithography devices [2, 3], miniature spectrometers [4] and miniature electron microscopes [5–9]. As evidenced by literature, research on miniaturization of electron microscopes has been done for several years now. Work concerns mainly selected parts of the microscopes such as the electron gun and electron optics column. So far, these miniature devices (Fig. 1) work inside high vacuum chambers because there is problem with creating high vacuum inside their very small volume.

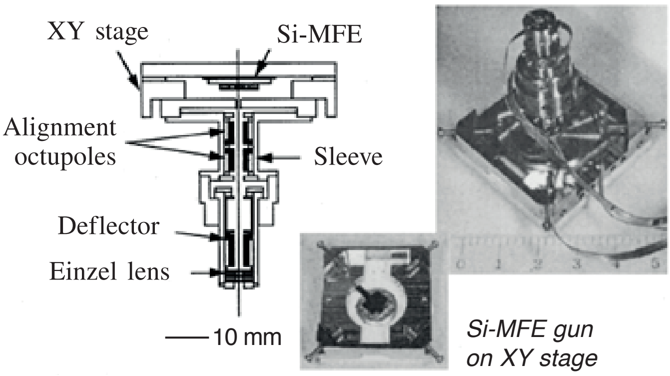

Fig. 1. Schematic image and photographs of the miniature electron optics column with a silicon field emission cathode [9]

This can be overcome with the use of a MEMS-type ion-sorption high vacuum pump [10], which has been developed by the team of Division of Microengineering and Photovoltaics, Faculty of Microsystem Electronics and Photonics, at the Wrocław University of Science and Technology. With to discovery of this device, a new concept of fabrication of MEMS-type TEM has been created [11]. It assumes that the microscope consists of a MEMS field emission electron gun connected to a MEMS ion-sorption micropump [10], which generates high vacuum inside the small (V ~ 0.25 cm^3) electron optics column. The column is bonded with a special fluidic microchamber, which introduces a sample into the electron beam's area of impact (Fig. 2).

The electron beam, emitted from the silicon cathode covered by carbon nanotubes, is focused by an Einzel lens on a very thin silicon nitride membrane (dimensions of 0.5×0.5 mm^2, 100 nm thick). This membrane separates the high vacuum part of the microscope (electron gun and electron optics column) from the

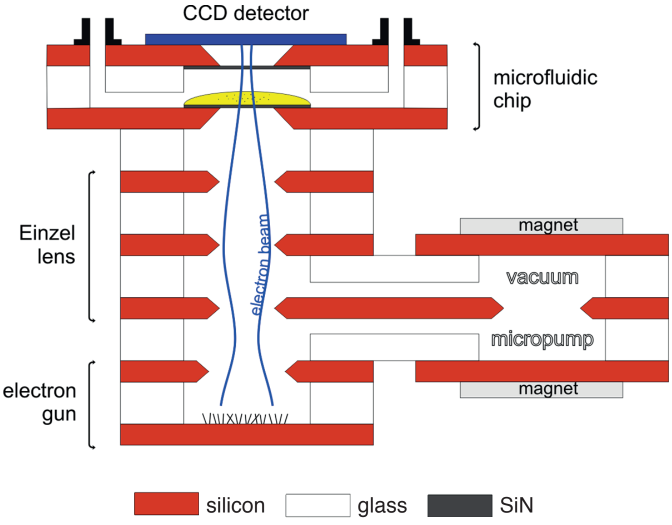

Fig. 2. Concept of miniature transmission electron microscope on-chip

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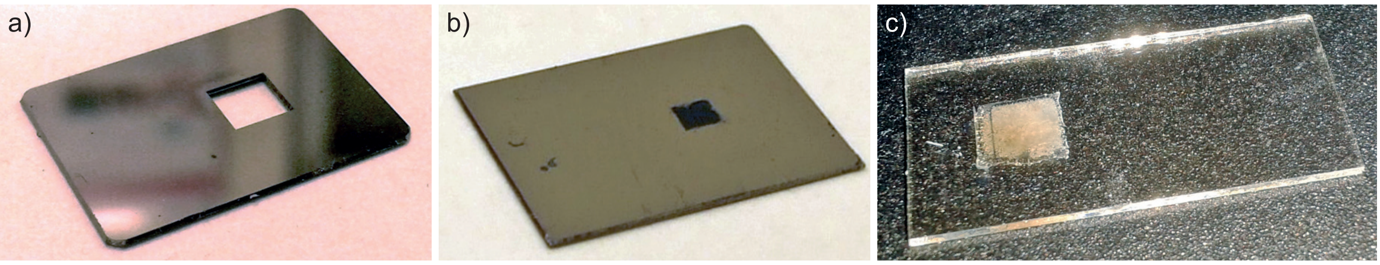

Fig. 3. Silicon and glass electrodes: a) silicon electrode with 3×3 mm^2 hole, b) silicon cathode with CNT layer (2×2 mm^2), c) glass screen with ITO layer and ZnS:Ag phosphor (5×5 mm^2)

sample microchamber (microfluidic chip). The electron beam is scattered on the sample and then detected by a CCD camera (detector) placed on top of the device. The microscope is made of silicon electrodes which are separated by borosilicate glass plates. All parts of the microscope are vacuum sealed by means of an anodic bonding process. Before a working MEMS transmission electron microscope is manufactured, some studies must be done concerning individual elements of this device.

In this paper we present and discuss of the manner for generating and focusing an electron beam in the device which we are developing. There are two basic solutions allowing to focus an electron beam in a small volume: this is done either by using the Einzel lens [12–15] and/or a deflector [16, 17]. In our concept the MEMS-type Einzel lens is utilized. Singular Einzel lens consists of three electrodes placed one after another, made of silicon substrates with a hole in the center. The potentials applied to the electrodes form the electric field on the path of the electrons to change their individual paths and to focus them on the surface of choice. This lens is integrated with a MEMS-type electron gun, which includes a CNT cathode and an extraction electrode (gate). To speed up and improve research, a special polymer holder made by means of the 3D printing technique was applied.

2. Technology

2.1. Electron column electrodes. All electrodes were made of 3", (100) oriented, n-type, low resistivity, 400 μm thick, double-side polished wafers. All electrodes, including the cathode, gate and Einzel lens, have the dimensions of 12×16 mm^2.

Silicon wafers used for the gate electrode and Einzel lens were wet-oxidized to obtain 1.5 μm thick dioxide. Following the photolithography process, the silicon wafer was anisotropically etched in a 10M KOH water solution at 80°C to form separate electrodes with square holes (3×3 mm^2) (Fig. 3a).

A layer of carbon nanotubes was deposited by means of the electrophoretic process ($U = 300$ V, $t = 2$ min) on the top surface of the silicon electrode made without a hole. The silicon plate was masked with polymer foil to deposit the CNT layer of different dimensions (1×1, 2×2, 3×3 mm^2) (Fig. 3b). The CNTs were suspended in isopropyl alcohol with a bit of $Mg(NO_3)_2$, and then sonicated (1 hour, 60°C).

The glass screen (anode) was made of borosilicate glass covered by an ITO layer (Kintec Co., Hong Kong). The glass (0.7 mm thick) was cut by a diamond blade to 12 mm wide and 25 mm long strips. ZnS:Ag phosphor was deposited on the ITO layer by means of the electrophoretic process. The ZnS:Ag powder was suspended in isopropyl alcohol with addition of $Mg(NO_3)_2$. The glass strip was masked with polymer foil to form an opening of 5×5 mm^2, where the phosphor was deposited at 200 V for 5 minutes (Fig. 3c).

2.2. Polymer holder. The test device was assembled by placing the silicon electrodes and the anode (glass screen or CCD camera) inside the dielectric polymer holder. The polymer holder was designed with use of the Autodesk Inventor software. It was projected to hold all electrodes with planar dimensions of 12×16 mm^2, one on top of another with the distance between them of 1.1 mm. This distance corresponds to the thickness of the borosilicate glass that is used in our concept of the MEMS microscope. The holder facilitated alignment of all electrodes, which is crucial for focusing the electron beam. It was made with the use of a ProJet 350 SD 3D printer (VisiJet M3 Crystal – printing material, VisiJet S300 – support material). Each printed monolayer had the thickness of 32 μm and the x–y dimensions quality was about 50 μm. The polymer holder was designed in two forms: to hold the glass screen (Fig. 4a) and to hold a CCD camera, been used for imaging the electron beam spot (Fig. 4b).

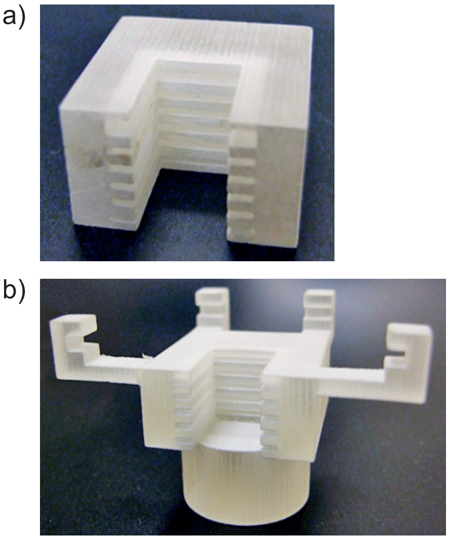

Fig. 4. Polymer holders: a) for mounting of the ITO/ZnS:Ag phosphor glass screen, b) for mounting of the CCD camera

3. Measurements

3.1. Electron gun tests. Measurements of electron emission properties of the MEMS electron gun were made inside a high vacuum reference chamber ($p = 10^{-5}$ hPa). This was done with the use of three electrodes: cathode, gate (with 3×3 mm^2 hole) and anode (flat silicon plate) (Fig. 5). The cathode was connected to a DC voltage supply and the other electrodes were grounded.

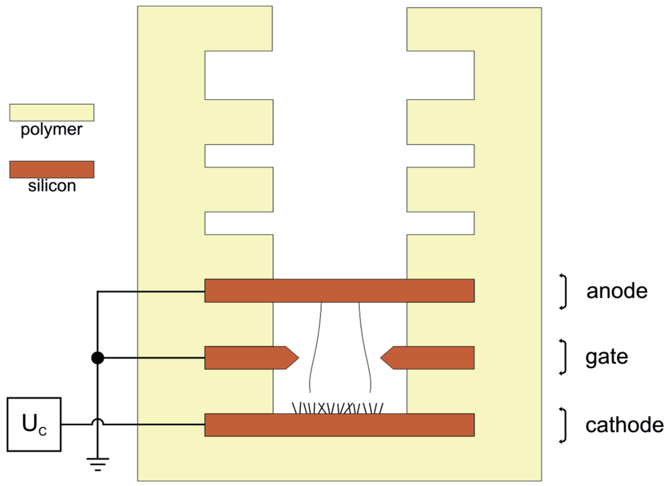

Fig. 5. Schematic image of emission current measurements

Electron emission was obtained for all the CNT cathodes prepared. Threshold voltage has been estimated at the level of 1100 V (Fig. 6). The highest value of anode current (I_A) was obtained for the largest size of the CNT layer. The value of electron current was satisfactory and the threshold voltage was relatively low, so this type of electron gun can be used as an electron source in miniature electron microscopes.

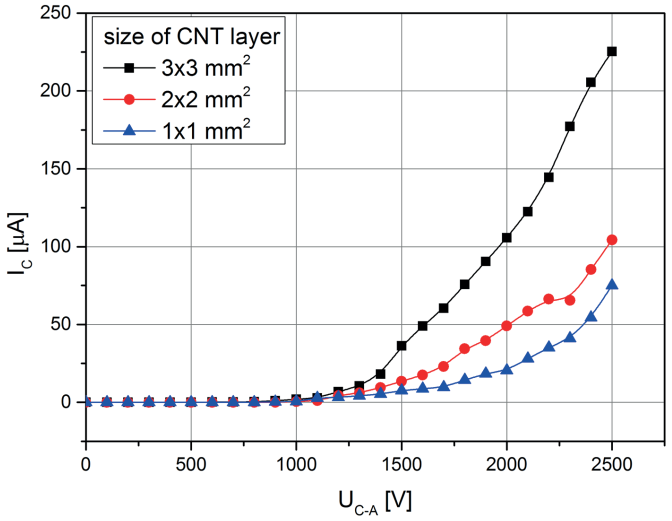

Fig. 6. Current-voltage characteristic of MEMS field emission electron gun for different sizes of the CNT cathode

3.2. Electron beam focusing. The shape of the electron beam spot was studied with all the electrodes, i.e. the cathode, gate and the three Einzel lens electrodes(central hole dimensions 3×3 mm^2), located inside the polymer holder (Fig. 7).

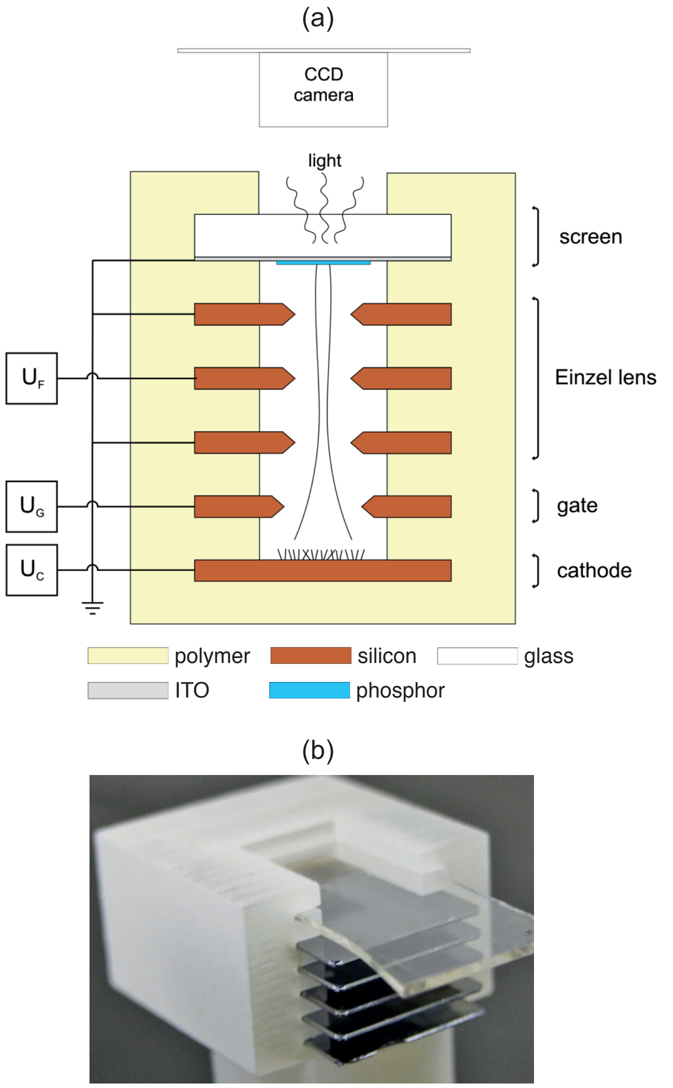

Fig. 7. Electron beam focusing measurements: a) schematic image, b) view of the hybrid silicon-glass-polymer electron optics column

The glass phosphor screen, which generates blue light when excited by the electron beam, was observed with the Bresser USB HANDY 2.0 MPix camera placed outside the high vacuum chamber.

The cathode with the 1×1 mm^2 CNT layer was used to deliberate on the results of electron emission measurements. Electron current for all test cathodes was satisfactory and obtaining the smallest electron beam spot size is likely to be from the smallest electron source. The cathode, gate and focusing electrode of the lens (center electrode) were connected to DC power supplies. The other two electrodes of the Einzel lens as well as the anode (screen) were grounded. After applying –2500 V voltage at the cathode, and –500 V at the gate elec-

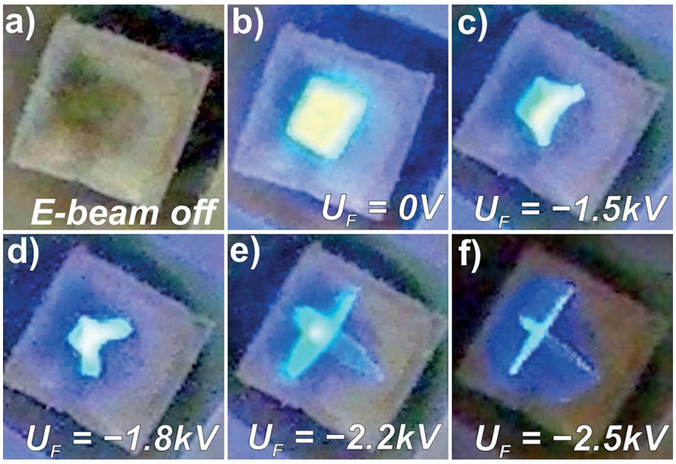

Fig. 8. Electron beam spot observed at the glass phosphor screen: a) electron gun turned off, b) $U_F = 0$ V, c) $U_F = -1.5$ kV, d) $U_F = -1.8$ kV, e) $U_F = -2.2$ kV, f) $U_F = -2.5$ kV; $U_C = -2.5$ kV, $U_G = -0.5$ kV

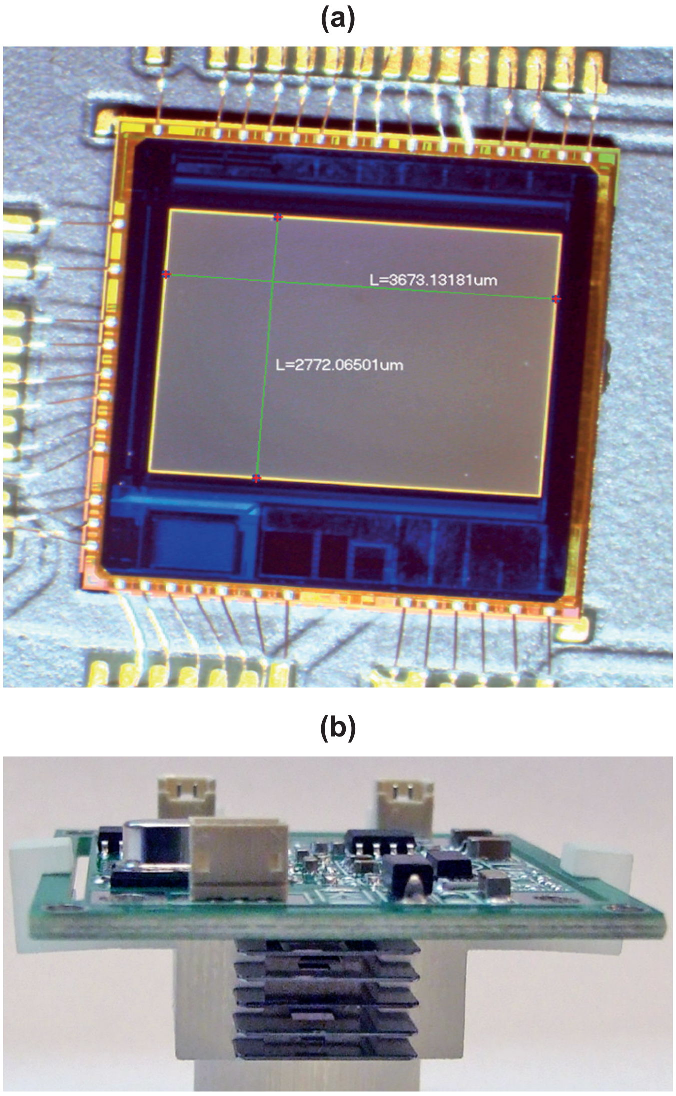

Fig. 9. CCD LC-1/4 Sony 480TVL Ex-view camera used as an electron detector: a) microscopic image of the CCD matrix, b) camera mounted in the polymer holder

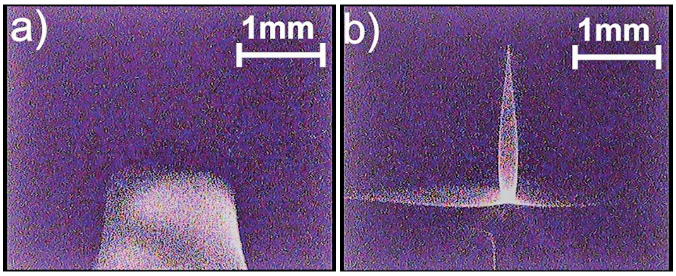

Fig. 10. Images of the electron beam spot recorded by the CCD camera for: a) unfocused beam ($U_F = 0$ V), b) focused beam ($U_F = -2.5$ kV); $U_C = -2.5$ kV, $U_G = -0.5$ kV

trode, blue light emitted from the screen was observed (Fig. 8). After turning the voltage of the focusing electrode from 0 V to –2500 V a change in the light spot could be seen. The shape of the spot was determined by the electric field generated by the square configuration of the holes in all electrodes. The irregular shape of the spot is due to the minor shift from the screen, as the electrodes seem to be placed correctly.

To estimate the quality of e-beam focusing, i.e. the focused beam spot size, the CCD camera (LC-1/4 Sony 480TVL Ex-view) with 3.68×2.77 mm^2 matrix (Fig. 9a) was mounted instead of the screen in the polymer holder (Fig. 9b). The camera was placed in a holder in such a way that the CCD matrix was at the same distance (1.1 mm) from the last Einzel electrode as the glass phosphor screen (Fig. 7b). The camera collects electron signal directly, producing an image of the electron beam spot. Knowing the dimensions of the CCD matrix (width = 3.67 mm, height = 2.77 mm, measured with an optical microscope) and the dimensions of the image (width = 720 pix, height = 576 pix), it is possible to estimate the dimensions of the focused beam spot. After measuring the width (or height) of the brightest area of the beam spot, which is about 32 pix, the calculated beam spot is about 0.163×0.163 mm^2 (Fig. 10). The estimation error depends on the precision of measuring the dimensions of the CCD matrix, and estimation of the brightest beam spot. The characteristic shape of the focused beam spot is determined by the square shape and dimensions of the holes in the lens and gate.

4. Conclusions

The new concept of a MEMS-type transmission electron microscope integrated on one chip is presented. It is a multi-layer silicon-glass structure containing an electron gun, an Einzel lens and an anode in the form of a silicon chip with a thin membrane. The proposed system will be tightly connected with the multi-layer silicon-glass vacuum micropump. A sample microchamber, in the form of a microfluidic device with a detector, will be integrated on top of such microscope structure. The device will be manufactured once all individual elements are developed.

The first step implementing this idea has already been done. The electron gun consisting of a field emission CNT cathode, gate and focusing electrodes was prepared by means of using the MEMS techniques. All electrodes were separated and adjusted with the use of a special polymer holder, which was made by means of the 3D printing method. The holder allowed for significant acceleration of research. The research was conducted inside a vacuum chamber, as this structure is under investigation and it isn't connected to the already described miniature MEMS vacuum micropump [10].

Electron field emission was obtained and first measurements of focusing capabilities of the Einzel lens, made of silicon substrate, were made. For cathode-gate distance of 1.1 mm, the threshold voltage of electron emission was about 1100 V. The Einzel lens showed acceptable focusing of the electron beam. It was observed that the shape of the beam spot was determined by the square shape of the central holes fabricated in the lens and gate. It was found that the focused beam spot of 0.163×0.163 mm^2 is small enough to be used to illuminate specimens in transmission electron microscopy. Research on the effects of different sizes and shapes of the electrodes holes will be carried out. Meanwhile, the results of this study are promising for developing the miniature MEMS-type transmission electron microscope in the future.

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