### **Short Communication**

# Experimental Investigation of the Distribution of Energy Deposited by FIB in Ion-beam Lithography

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The first and rigorous sensitivity comparison of the most used positive-tone resist (PMMA 950K) exposure to both electrons and gallium ions in a wide range of exposure doses at the same beam energy was carried out. It was found that the PMMA 950K resist has a positive sensitivity of  $0.15~\mu\text{C/cm}^2$ , which is about three orders of magnitude more sensitive to gallium ions than to electrons, all at the same conditions. At high Ga exposure doses, as well as with electron exposure, negative sensitivity is observed. The depth of the resist after etching in a solvent depending on the exposure dose was also studied, and based on this an analytical model using the absorbed energy density in the form of a displaced Gaussian, that allows one to restore the resist contrast and the energy length from experimental data, was proposed. The model accurately describes the both experimental and simulation results. It was shown that the contrast for the PMMA 950K resist is  $\gamma \sim 3.1$  for the energies of gallium ions and the energy length is  $L_e = 43~\text{nm}$ .

**Keywords**: Ion-beam lithography, Resist, FIB (Focused ion beam), Poly (methyl methacrylate) (PMMA), Gallium ion, Sensitivity, Contrast.

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#### 1. INTRODUCTION

For various reasons, ion-beam lithography is less known than electron-beam lithography. Ion-beam lithography mainly uses beams of light ions, such as protons and helium ions [1-5], and heavy ions are used for chemical and radiation modification of the surface [6, 7]. It is well known that an ion beam has much weaker angular scattering in matter than an electron beam, moreover, secondary particles have very short ranges, and these circumstances allow ion-beam lithography to overcome the resolution limit of electron-beam lithography. Since the energy loss of ions in the polymer resist is several orders of magnitude higher than that of electrons, then the sensitivity of the resist to the ion-beam is higher and exposure is much faster, and this is a rather important technological parameter.

Thus, based on the foregoing, the task was set to experimentally study the interaction of Ga<sup>+</sup> ions (heavy compared to H<sup>+</sup> and He<sup>+</sup>) with the most used resist PMMA 950K to obtain information on the distribution of energy deposited in the resist and the main parameters characterizing the lithographic process.

#### 2. EXPERIMENTAL DETAILS

Exposures were carried out in SEM Quanta 3D 200i dual beam system with Nanomaker pattern generator system. The currents of electrons and ion beams were measured by using Faraday cup. The deviation of the beam currents during exposure did not exceed 2 %. The PMMA 950K resist was spin coated on the Si wafer with the speed of 6500 rpm and 3000 rpm for 30 s, the thickness was about 40-70 nm and 140 nm, respectively. The sample was pre-baked on a hot plate at 180 °C for 180 s. Next, the structure of the so-called dose wedge

was exposed by focused Ga<sup>+</sup> ions in a wide range of doses at 30 keV. After exposure to the ion-beam, sample was developed at MIBK: IPA developer (1:3) for 8 s and 5 s dip in stopper (IPA). Topography of dose wedges was performed using AFM.

### 3. RESULTS AND DISCUSSION

The sensitivity of the PMMA 950K resist to electron and gallium ion exposure was compared. The comparison presented in Table 1 shows that the positive sensitivity to ion-beam exposure is about  $0.15~\mu\text{C/cm}^2$  and it is three orders of magnitude higher compared with typical sensitivity to electron-beam exposure at the same conditions. It should be noted that at high doses of exposure, as in the case of electron-beam exposure, a region of negative sensitivity is also observed. This comparison was carried out on a resist with a thickness of 50 nm. Gallium ion exposure of the resist with a dose of up to  $10~\mu\text{C/cm}^2$  did not lead to a significant sputtering of the resist.

The second series of the experimental study concerned the investigation of etching depth of the (thick) resist depending on the radiation dose (Fig. 1, blue dots). It is found that, in contrast to electron-beam, the distribution of energy deposited by Ga<sup>+</sup> FIB is strongly inhomogeneous in depth, which results in strongly inhomogeneous etching rate as a function of depth and radiation dose.

Table 1 – Experimental sensitivity of positive tone resist (PMMA 950K) to 30 keV electron and gallium ion irradiations

	Positive, [μC/cm²]	Negative, [μC/cm²]
Electrons	~ 150	~ 5000
Ions	~ 0.15	~ 2.2

These data served as the basis for attempts to restore quantitatively the spatial distribution of the energy losses of fast ions in matter.

The fitting procedure is based on the formula (1), in which the dissolution rate of the resist V was related to the density of energy  $E_{\rm emp}$  absorbed in the z layer, the exposure dose D and the contrast of the resist  $\gamma$ :

$$\frac{V}{V_0} = \left(\frac{DE_{\text{emp}}(z)}{D_0}\right)^{\gamma},\tag{1}$$

where  $V_0$  is the dissolution rate of the resist at the exposure dose  $D_0$ .

Expressing D as a function of z, we obtain the following relation:

$$D = D_0 \left( \int_0^Z \frac{dz}{\left( E_{\text{emp}}(z) \right)^{\gamma}} \right)^{\frac{1}{\gamma}}.$$
 (2)

The density of the absorbed energy was described by the empirical formula (3) in the form of a displaced Gaussian obtained in [8, 9] by the Monte Carlo simulation (TRIM program)

$$E_{\rm emp}(z) = A \exp\left(-\frac{\left(\frac{z}{L_e} - B\right)^2}{2C^2}\right). \tag{3}$$

There is a good agreement between the experimental and calculated data (Fig. 1). It is possible to restore from the fitting the contrast of the resist as  $\gamma = 3.1$  and the mean free path of ions in the PMMA 950K  $L_e = 43$  nm. This value is approximately 20 % less than the energy length obtained by modeling (53 nm). It is not yet clear whether this difference is due to the imperfection of the formulas (interaction cross sections of TRIM) embedded in the modeling algorithm, or the difference is due to the simplicity of the development model.

An important feature of ion-beam lithography is noted in the discussion. Compared with electron-beam lithography, a strong dependence of the sensitivity of the resist on its thickness is observed. With an increase in

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thickness from 10 nm to 70 nm, the sensitivity measured in units of  $[\mu C/cm^2]$  changes by an order of magnitude remaining, however, surprisingly high in comparison to the sensitivity to electron irradiation.

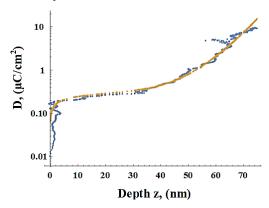


Fig. 1 – Comparison of the experimental etching depth (blue dots) with the etching simulation (yellow dots) allows to determine the contrast value of the resist and the energy length of  $\rm Ga^+$  ions in the PMMA 950K

#### 4. CONCLUSIONS

It was shown that the sensitivity of the positive tone resist PMMA 950K to gallium ions with an energy of 30 keV is approximately  $0.15~\mu\text{C/cm}^2$ , which is 1000 times higher, and the negative sensitivity is more than 2200 times higher than that to electrons at the same energy.

We developed an analytical model for extracting the main parameters characterizing the lithographic process from the experimental curve dependence of the exposure dose on the depth D(z). There has been shown good agreement between the analytical model and experimental data. Thus, in addition to the practical significance of the measured characteristics, the presented method opens up an experimental method for studying the interaction processes of fast ions with matter.

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## Експериментальне дослідження розподілу енергії при осадженні сфокусованим іонним променем в іонно-променевій літографії

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Було проведено перше і ретельне порівняння чутливості найбільш часто використовуваного покриття РММА 950К до впливу як електронів, так і іонів галію в широкому діапазоні доз опромінення при однаковій енергії пучка. Було встановлено, що покриття РММА 950К має позитивну чутливість  $0.15~\rm mkK.n/cm^2$ , і приблизно на три порядки більш чутливе до іонів галію, ніж до електронів в тих самих умовах. При високих дозах опромінення іонами галію, а також при опроміненні електронами спостерігається негативна чутливість. Також вивчали глибину травлення покриття РММА 950К після його травлення в розчиннику залежно від дози опромінення. Виходячи з цього, була запропонована аналітична модель з використанням густини поглинутої енергії у формі зміщеного гаусіана, яка дозволила відновити контрастність покриття РММА 950К та енергетичну довжину за експериментальними даними. Запропонована модель точно описує як експериментальні результати, так і результати моделювання. Було показано, що контрастність для покриття РММА 950К становить  $\gamma \sim 3,1$  для енергій іонів галію, а енергетична довжина є рівною  $L_e = 43~\rm hm$ .

**Ключові слова**: Іонно-променева літографія, Покриття, FIB (сфокусований іонний промінь), Полі-(метилметакрилат) (РММА), Іон галію, Чутливість, Контрастність.