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Geometry of nanopore devices fabricated by electron beam lithography: Simulations and experimental comparisons

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ABSTRACT

Fabrication of solid-state nanopore sensors for individual biomolecule analysis is a growing topic of interest. A number of recent studies demonstrate that engineered nanopore devices (ENDs) can be fabricated by electron beam lithography (EBL) with high density (on the order of 10 devices per cm^2). The internal pore geometry of ENDs is a critical characteristic of the devices, and often obtained by some combination of SEM, TEM, AFM, and conductance measurements. However, experimental data alone is not sufficient to understand the nanopore geometry under a broad set of fabrication conditions. It is necessary to also examine the physical basis underlying the EBL-based fabrication of ENDs. In this work, the internal pore geometry of ENDs is calculated from electron energy distributions in EBL while investigating the effects of dose, operating blur, substrate, and dosing pattern. The photoresist is ZEP-520 on silicon or silicon nitride substrates. It is found that higher beam blur and lower dose cause a greater degree of pore tapering, with the most prominent tapering observed in sub-10 nm pores. Nanopores in silicon nitride tapered more than those in silicon. The results also demonstrate that a combination of blur and dose can be chosen to achieve a target tapering angle and pore size at a given depth in the substrate. Because the pore tapering angle is non-uniform, the ICP (inductively coupled plasma) etch depth may also be used to tune pore size and geometry following EBL. The resist sensitivity is shown to increase with beam blur for pore sizes larger than 10 nm. By comparing to our experimental data, it is found that beam intensity measured by the EBL instrument may not translate to the operating blur, as is often assumed in EBL simulations. Secondary electrons were found to be responsible for pore tapering and beam broadening in the resist.

1. Introduction

Nanopore biosensors fabricated on silicon substrates have emerged as a robust and adaptable platform for interrogating individual biomolecules as they translocate through the pore under an applied driving force such as an electric field [1]. These solid-state "engineered nanopore devices" (ENDs) have been fabricated using focused ion beam (FIB) [2], transmission electron microscopy (TEM) [3], and electron beam lithography (EBL) [4,5] techniques. EBL is attractive for END fabrication because it is a fully wafer scalable process [6] shown to produce arrays of pores at a time [5]. This is important for realizing high-throughput operation in a real sensing environment.

Thus far, EBL in the context of ENDs has been studied experimentally with the intent of producing and characterizing nanopores with diameters less than 100 nm in a reproducible manner. These studies have revealed strong correlations between dose and pore geometry as well as qualitative insights into the fabrication process. Nonetheless, there are a number of issues that cannot be addressed through experimentation alone. From the literature, the smallest pores produced by EBL were of size 10 nm [5]. It is unclear whether this apparent lower bound is a fundamental physical constraint or the result of an empirical limitation. In addition, pores observed in SEM appeared to have a tapering internal structure, the exact morphology of which could not be fully determined. The source of this observed effect possibly lies in the electron scattering behavior. Another interesting observation was that pores produced in ZEP photoresists on silicon nitride appeared measurably smaller than pores in ZEP on silicon at the same dose [5]. These pores were produced under identical conditions of development and etching, which may indicate that the source of the discrepancy arises from different electron scattering behavior in silicon and silicon nitride.

The objective of this work is to evaluate in detail the internal geometry of ENDs in the context of a physical model of the EBL process. Pore morphology is of critical importance to the function of ENDs. EBL processing is commonly simulated by attributing main chain scission of the resist to the electron energy distribution in the film, the latter being calculated using a Monte Carlo approach [6–11]. Prior literature focused on geometries, pattern densities, and length scales which are outside of the scope of ENDs.

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Much of the previous work was also dedicated to proximity effect correction, which is not significant for ENDs due to the large spacing (on the scale of millimeters) between individual nanopore features.

The approach taken in this work is as follows. First, the electron energy distributions in the substrate are calculated. These distributions are then used to simulate the pore geometry for a broad range of process parameters, including the dose, shot pattern, beam blur, and substrate. The study is performed with substrates and operating conditions used in our previous experimental work [5] to make comparisons with empirical data. This allows evaluation and explanation of the experimental results obtained in previous literature, and makes possible a predictive 'design' capability for END fabrication. We also use our model to study the effects and importance of secondary electron emission, which is hypothesized to play a role in determining the nanopore morphology.

2. Methods

2.1. Assumptions of the model

Prior to studying electron scattering behavior, it is important to note some key considerations as determined from literature. First, the primary difference between exposing single and multiple shots is that single-shot dosing relies to a greater extent on backscattered electrons whereas multiple shot dosing relies more on forward-scattered electrons to produce the same nominal pore size [12]. In addition, since forward scattering characteristically occurs at smaller angles than backscattering, the geometry of a single shot pore is hypothesized to 'taper' to a lesser degree than a pore patterned with multiple shots. Another important consideration is the role of the 'proximity effect' [12]. The distance between adjacent nanopore devices is 2.47 mm, well outside the range of the proximity effect (on the order of microns). However, in determining experimental pore size distributions [5], most of the pores viewed in SEM were surrounded by features only about 500 nm away on all four sides. These would certainly experience some proximity effects, although the experiments suggest that it is nominal. Nonetheless, care must be taken in drawing comparisons between the experimental and simulated data.

The symmetry intrinsic to ENDs allows certain simplifying assumptions especially in the case of the single-shot model. For example, the energy distribution of a single shot (in cylindrical coordinates) clearly depends on radial distance r and depth z in the film, but is independent of the sweep angle θ due to the angular symmetry. This consideration significantly reduces the simulation effort. From our experimental work we found that pores as small as 8 nm were achieved in ZEP [5], smaller than most resolvable line widths that have been reported [13]. This may stem from the fact that lines inherently consist of multiple shots as well as a contribution of nearby shots from adjacent lines. The proximity effects are significant and limiting, but this is not a restriction for the present case wherein circular pores, and not lines, are being produced.

ZEP is well known to behave differently under different development conditions, specifically marked by changes in resist sensitivity [14,15]. In designing the simulations, the dose sensitivity is assigned in part by comparison to experimental data [5]. Because the development conditions were identical for every wafer, this factor is then eliminated as a variable. In summary, the following assumptions were made in simulating the internal pore geometry: (1) the electron energy distribution and subsequent pore geometry is angularly symmetric, (2) the pore geometry depends primarily on the energy deposition profile in the resist due to electron scattering in the film, (3) the proximity effect is negligible, (4) the etch rate in ICP is uniform and only occurs in the perpendicular z direction, and (5) experimental pores surrounded by boxes were comparable in size to isolated pores, allowing a reasonable basis for comparing simulated and experimental pore size distributions.

2.2. Simulation methods

In modeling electron scattering behavior, it is assumed that the atoms in the substrate are distributed randomly and with uniform

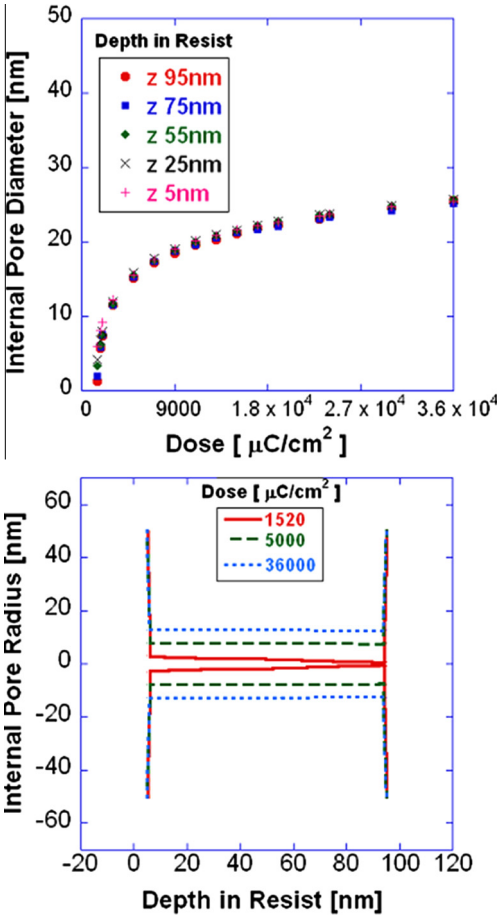

Fig. 1. Simulated internal pore diameter of single shot pores at different doses in 100 nm ZEP on Si assuming a beam blur of 9.9 nm.

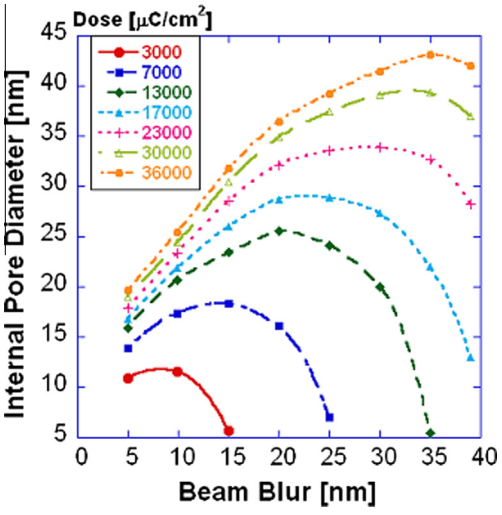

Fig. 2. Simulated internal pore diameter at a depth of 95 nm in the film (100 nm ZEP on Si) as a function of beam blur at multiple doses.