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Procedia Engineering 47 (2012) 684 – 687

Procedia Engineering

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ELSEVIER

Proc. Eurosensors XXVI, September 9-12, 2012, Kraków, Poland

Effects of the Focused Ion Beam parameters on nanopore milling in solid state membranes

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Abstract

This work describes a reliable nanofabrication technology of solid state nanopore arrays. The geometric parameters of pores are achieved and optimized according to the requirements of bioanalytical applications regarding conformation and size of the characteristic proteins in clinical diagnostics. Different structural configurations and material compositions of the nanopore structures were developed and characterised according to the variable biofunctionalisation strategies. The geometry of the fabricated pores were analysed as the statistical function of the focused ion beam milling parameters.

Keywords: solid state nanopore, focused ion beam, biochemical sensing

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1. Introduction

The application of the complex micro- and nanofabricated structures as sensing transducers becomes more relevant in the field of mechanical, chemical and biochemical sensors. As a result of novel and innovative biosensing principles new possibilities are being proposed in medical applications. These bioanalytical systems are expected to integrate the nanoscale transducers with sample preparation microfluidic systems also composing Lab-on-a-Chip devices. The chemically modified nanonopore based sensors can be applied to the detection of specific biomolecules through transport modulation determined

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doi:10.1016/j.proeng.2012.09.239

by molecule binding in pores. In spite of the extraordinary sensitivity of the principle, reliability and reproducibility of nanoscale fabrication processes are not adequately elaborated so far.

2. Fabrication of the nanopores

The characterized test structures were fabricated by the combination of silicon micromachining and subsequent nanofabrication processes. The composition of supporting membrane materials was selected such that low-stress mechanical structures are achieved. Functional layers of the nanopores were altered in order to provide proper surfaces for the possible receptor immobilization techniques. Accordingly, nanopores were drilled in both non-passivated and perfluoro-alkil passivated Silicon-Nitride and Gold layers respectively.

The multilayer membrane structures were developed by conventional MEMS technology including Low Pressure Chemical Vapour Deposition (LPCVD) of the supporting non-stoichiometric silicon-nitride (cca. 200nm thick), evaporation of titanium / gold layers (cca. 150nm thick) and anisotropic alkaline etching of silicon. The solid state nanopores were fabricated by Focused Ion Beam milling using accelerated Ga+ ions applying different milling currents and times as schematically demonstrated in Fig.1.

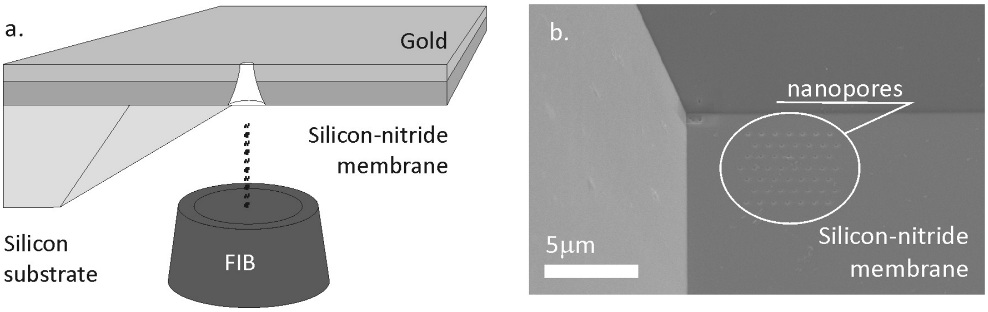


Fig. 1. Fabrication concept of the nanopores (a) in MEMS based membrane by Focused Ion Beam drilling and the realized nanopore array etched in the micromachined membrane layer (b).

The achieved nanopore geometries were characterized by scanning and transmission electron microscopy after revealing the nanopore cross section by cutting the membrane by Focused Ion Beam. The realized nanopore array and the cross sectional structure of a representative membrane structure and the nanopore are presented in Fig. 2.

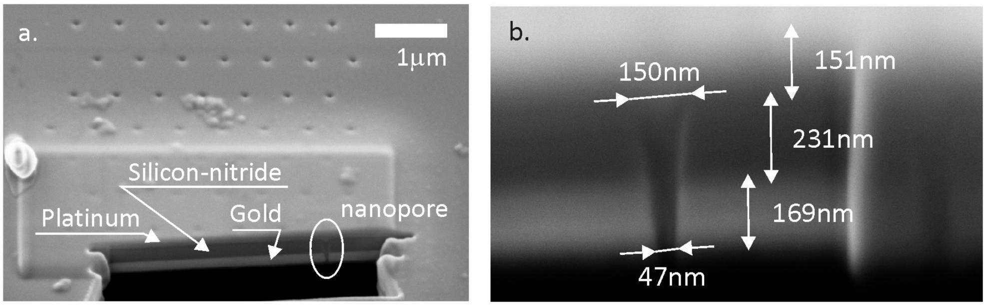


Fig. 2. Nanopore array fabricated in MEMS processed Silicon-nitride membrane.
The cross section of the membrane represents the layer structure (a.) and the nanopore geometry (b.) also.

3. Focused ion beam milling

The focused ion beam drilling process was characterized to establish a reliable fabrication technology regarding the accurate engineering of the pore geometry, particularly the pore diameter. The main issue was the tuning of the pore diameter by applying adequate fabrication parameters (milling time and ion current) in case of different material structures. The most interested membrane materials were the bare Silicon-Nitride and the Gold covered Silicon-Nitride. To achieve the lower limit of the pore diameter 5pA and 10pA milling currents were applied. The measured average pore diameters plotted against the milling time in the case of different milling currents and membrane structures are shown in Fig. 3. Note, that the nanopore diameters could have significant uncertainty due to the nanoscale fabrication process influenced by the material behaviour and proper focusing of the ion beam.

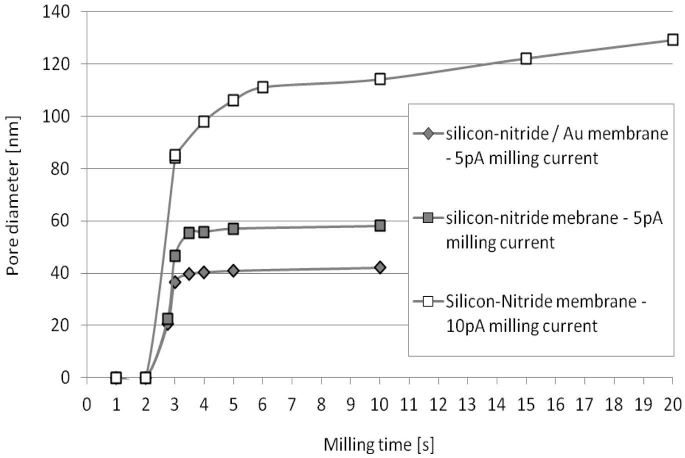
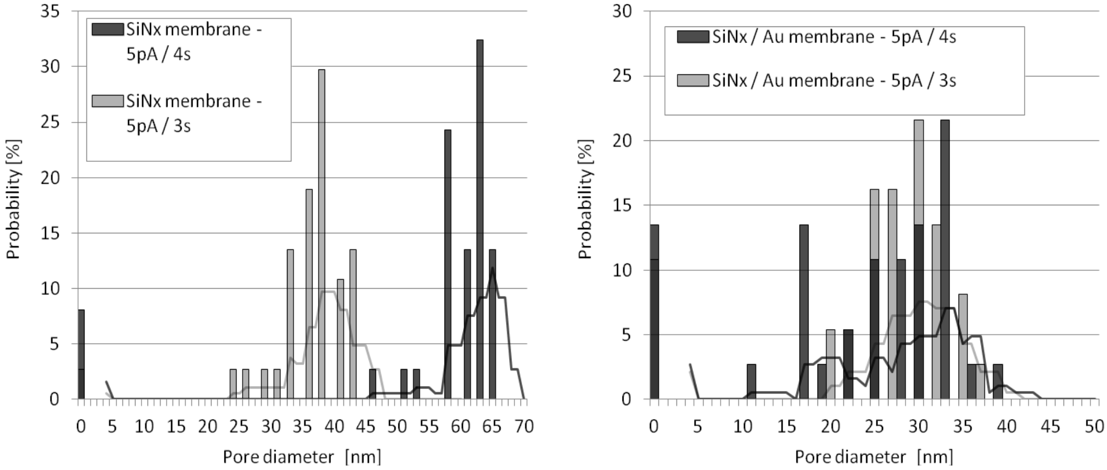


Fig. 3. The average pore diameter resulted by the ion milling of different layer structures as the function of the milling time applying 5 and 10pA ion current.

As clearly demonstrated in Figure 3 the evolved pore diameter is significantly depends on milling current, milling time, membrane thickness and structural materials also. In case of bare Silicon-Nitride membrane we observed almost doubled pore diameters when doubled (10pA) milling current was applied. The pore diameters were decreased in case of the Gold covered Silicon-Nitride membrane which can be explained by the higher thickness of the structure and the beneficial electrostatic influence of the metal layer. Due to the behaviour of the ion milling/sputtering process the complete perforation of the membrane occurs in a narrow time period, in case of these layer structure between 3 and 4 seconds.

4. Pore diameter distribution

For improvement the performance and reproducibility of the proposed biosensors solid state nanopore arrays were fabricated in the layer structures presented above. Advanced reproducibility of the nanopore structures can be achieved by applying multiple nanopores with exact statistical evaluation of pore diameters and shape. The nanopore arrays were characterized morphologically, and the statistical distribution of the nanopore diameters were measured (see Fig. 4). According to the previous experiments 5pA milling current were applied for 3 and 4 second to achieve the proposed pore geometry.



(a) (b)

Fig.4 . Statistical pore diameter distribution in different membrane structures (a: Silicon-Nitride, b: Gold coated Silicon-Nitride) drilled by Focused Ion Beam applying 5pA milling current for 3s and 4s.

Approximately 35nm and 60nm average inner pore diameters were measured in the bare Silicon-Nitride membrane in case of 3s and 4s milling time, respectively. Almost uniform, cca. 30nm and 35nm pore diameters were observed in case of Gold covered Silicon-Nitride membrane, applying the same milling currents. Note, that the probability of incomplete drilling cannot be neglected. We can conclude that the pore diameter changes more intensively in case of the bare dielectric layer in this time interval. However nanopores can be fabricated more reliably and with moderated diameter fluctuation in the metalized layer, almost independently from the milling time (over the characteristic perforation time).

5. Conclusions

The focused ion beam milling of micromachined membranes was characterized to establish reliable nanopore fabrication technology for nanoscale biosensing transducers. Bare and Gold covered Silicon-Nitride membranes were perforated by focused ion beam applying variable milling currents and times. The resulted pore geometries were analysed to define optimal milling parameters, and statistical limits (as pore diameter accuracy) caused by the uncertainties of nanoscale processes such as beam. The significant influence of the material composition was also clearly presented: the metallization of the dielectric membrane can improve the beam stability due to the reduced electrostatic fluctuations.

Acknowledgements

The partial support of the ENIAC JTI and the National Innovation Office (NIH) via CAJAL4EU project, the Hungarian National Research Fund (OTKA) via NF69262 and the János Bolyai fellowship of the Hungarian Academy of Sciences are gratefully acknowledged.

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