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COMMUNICATION

Functional nano-structuring of thin silicon nitride membranes

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The paper describes the development and production of a nano-optical device consisting of a nano-perforated layer of silicon nitride stretched in a single-crystal silicon frame using electron beam lithography (EBL) and reactive ion etching (RIE) techniques. Procedures for transferring nanostructures to the nitride layer are described, starting with the preparation of a metallic mask layer by physical vapor deposition (PVD), high-resolution pattern recording technique using EBL and the transfer of the motif into the functional layer using the RIE technique. Theoretical aspects are summarized including technological issues, achieved results and application potential of patterned silicon nitride membranes.

Keywords: membrane, nano optical device, electron optics, electron beam lithography, silicon nitride, reactive ion etching, silicon etching, microfabrication

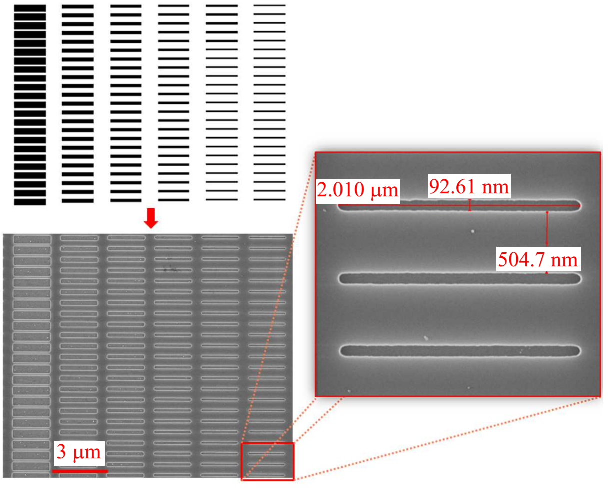

Fig. 1. Pictures of test design with different pattern sizes and SEM images of the pattern after RIE of silicon nitride

1 Introduction

Silicon nitride (SiN) layers are typically used in semiconductor industry and IC industry as insulating or masking layers in MOS technology [1]. SiN films are commonly used as well as a base material for membrane fabrication that can be used in various microelectronic and micro optic devices (MEMS, MOMS), *eg* as a capacitive pressure sensor with a Si_3N_4 diaphragm [2].

The basic procedure of membrane fabrication is well known [3][4]. Typically, it is obtained by wet anisotropic etching of bulk monocrystalline (100) silicone coated with a silicon nitride layer. What brings new technological challenges is the development of the fabrication process, which deals with the above-mentioned IC functional micro- and nanopatterning of the membrane surface. Such innovative membrane-based devices can have both new functionality and possible applications, *eg* , modification of electromagnetic radiation or of the charged particle beams distribution, phase or spectrum. Recently, a series of research activities and publications have been devoted to the development of these functional elements, eg, micro-optical elements for Helium [5], electron microscopy [6], laser and light optic [7][8], as well as nanosieves for molecular separation in microfluidics or for bioengineering [9].

In this article, we propose a fabrication method of a patterned membrane based Nano Optical Device (NOD) with application in charged particle optics. The membrane is not used just as a support for functional structures built on top (not IC based), but they are used as a bulk material to prepare functional relief structures directly in the SiN layer itself. To do so, different etching techniques have to be adapted within the fabrication steps: plasmatic etching (PE), reactive-ion etching (RIE) and anisotropic wet silicon etching. High-resolution e-beam lithography (EBL) is necessary due to the resolution required for patterning of planar structures in the membrane.

2 Design and fabrication of the test structures

The design of test structure consists of a series of lines on an area of approximately $20 \times 20\,\mu\mathrm{m}^2$ (Fig. 1).

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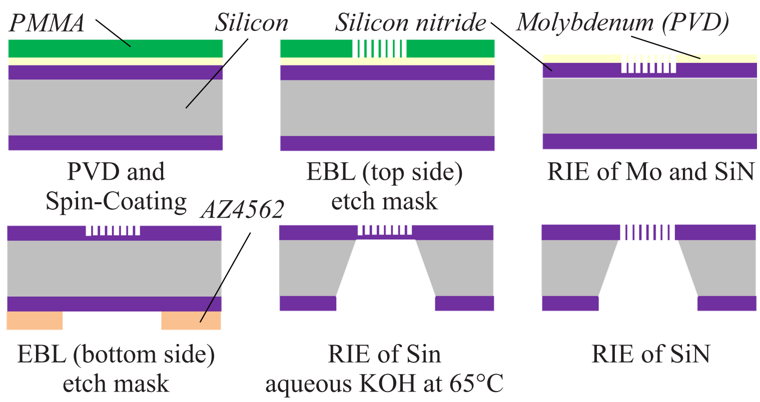

Fig. 2. Schematic of fabrication processes of silicon nitride patterned membranes.the thickness of silicon nitride is 100 nm, the thickness of molybdenum is 20 nm.with extra RIE etching at the end of the process to finish holey pattern in the membrane, the final thickness of the silicon nitride membrane is 75 nm

We used it to recognize the parameters of motif transfer in individual fabrication steps: EBL soft mask, RIE hard mask and RIE SiN. The parameters of the lines in the test structure were as follows: line width 50, 70, 100, 150, 200 and 400 nm, line length 2000 nm, line periodicity 600 nm.

The sample with the test structure was prepared from a monocrystalline silicon wafer (100) with 100 nm of silicon nitride (LPCVD low-tension) on both sides and 20 nm of molybdenum (Mo) prepared by PVD. For etching of Mo and SiN, we used an RIE unit with inductively coupled plasma (ICP) of Oxford Instruments. This test structure was measured and evaluated using an atomic force microscope (AFM) and an electron microscope (SEM) between the etching steps to estimate how the line motif would expand during hard mask preparation and then during motif transfer across the hard mask into the nitride layer.

The critical part was to find correct parameters of RIE for motif transfer with low edge extension, high selectivity, and low etching speed.

With respect to our previous experience [10][11], we have chosen the $SF_6 + Ar$ and $CF_4 + CHF_3$ recipes for molybdenum mask and silicon nitride etching. From the measurements of the sample with the test structure in SEM, AFM and profilometry we were able to estimate good starting parameters for the RIE process. For the Mo hard mask preparation we used a recipe with SF_6 (50 sccm) + Ar (40 sccm) gas mixure, ICP power 3000 W and 10 W RIE power at 60 °C. This recipe exhibits an average etching rate of molybdenum 2 nm/s. Although the selectivity of Mo/PMMA etching is not high, it is sufficient for the purpose. For the silicon nitride etching through the hard mask we used a recipe with CF_4 (10 sccm) + CHF_3 (40 sccm) gases, ICP power 1000 W, 50 W RIE power and working temperature 60 °C. This recipe exhibits an average SiN etching rate of 2.5 nm/s and at the same time, the hard mask (Mo) etch rate is about 20 times lower.

3 Fabrication procedure characterization

After initial study with the test structure, we have proposed the technological process of fabrication of a real nano-structured membrane. The fabrication processes are schematically illustrated in Fig. 2. and are as follows:

Top-side lithography operations

- PVD deposition of Mo layer (hard mask)
- coating of the PMMA resist
- pattern recording by EBL at EBPG5000+ES (soft mask)
- plasmatic etching of the PMMA in O_2
- RIE of the Mo layer trough the PMMA mask
- RIE of the SiN layer trough the Mo mask
- plasmatic ashing of PMMA in O_2 (dry removal of PMMA)
- wet removal of the Mo layer (H_3PO_4:HNO_3:H2O).
- protective coating with AZ4562 resist Back-side lithography operations
- coating of the AZ4562 resist on back-side
- pattern recording by EBL at EBPG5000+ES (soft mask)
- RIE of the SiN layer trough the AZ4562 mask
- wet removal resist
- wet etching of silicon in aqueous KOH solution at 65 °CFinalization
- RIE of the SiN layer without mask

In order to ensure the correct recording of the structure into the membrane, a number of measurement operations by AFM, SEM and a probe profilometery have to be included. These measurements take place especially between individual etching steps by RIE and during the mask preparation steps.

4 Design of the membrane based nano-optical device

The design of the nano optical device (NOD is inspired by the concept published in [12]. The NOD we have de-

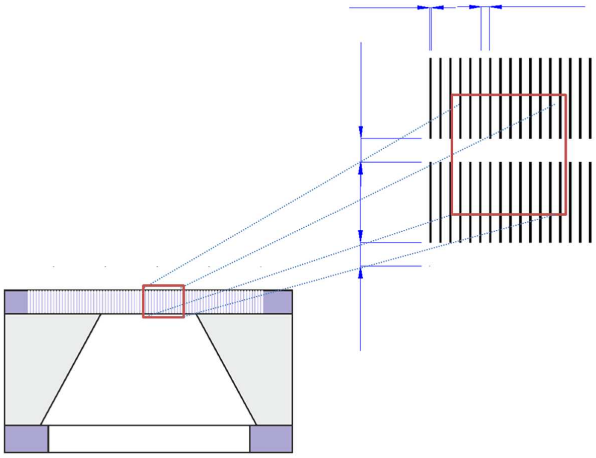

Fig. 3. Schematic illustration of NOD silicon chip with holey grating pattern transferred directly in the nitride membrane.grating parameters: linewidth 70 nm, periodicity 250 nm, length 2000 nm

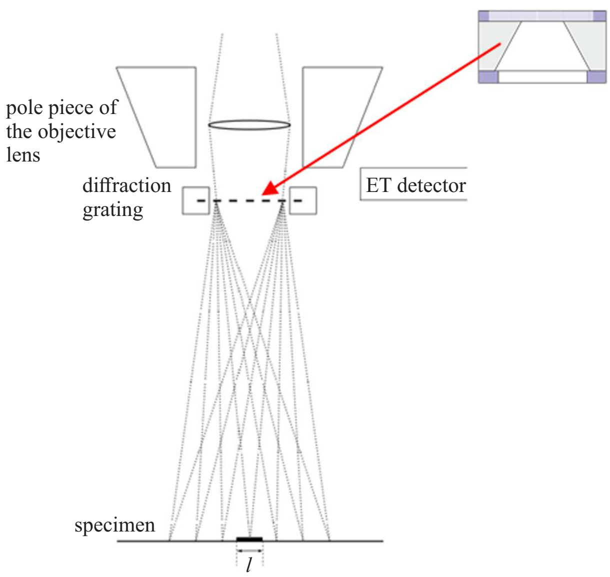

Fig. 4. Scheme of the experiment with membrane base NOD in the SEM magellan 400

signed (see Fig. 3) purposefully for experimental study of electron beam diffraction in a scanning electron microscope. The device was developed as part of research into the possibility of phase manipulation in the primary electron beam in transmission or scanning electron microscopes.

One of the most promising areas in which this research is directed is the possibility of generating vortex beams. The application potential is, for example, probing of magnetic materials or for electron spin-polarized beam microscopy [13].

To fabricate the designed NOD, we used the technology we have developed and described above in this paper. The NOD is a silicon chip with external dimensions not exceeding 44 mm^2. In the middle of the chip there is a 100×100 μm^2 window with a 70 nm thick nitride membrane. The membrane area and the area around (1×1 mm2) contains high-density rectangular shape nano-perforation with a size of 70 nm $\times$ 2000 nm, see Fig. 5.

The membrane surface is metalized with a thin layer of platinum to avoid charging. This device functions in the electron microscope as a screen with over 20 k nano apertures on which diffraction occurs of the passing electron beam.

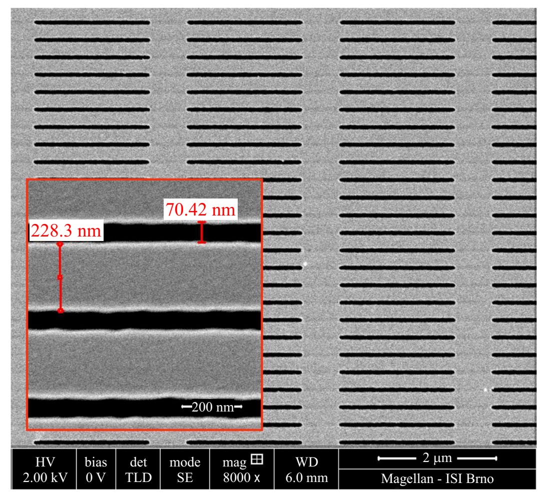

Fig. 5. SEM image of the final holey grating structure fabricated in the thin silicon nitride membrane.size of the rectangle shape opening in the membrane is 70 nm $\times$ 2000 nm

5 Conclusions

In our work we have described processes used for functional nano-structuring of nitride based freestanding membranes. We point out that both electron beam lithography and selective etching in a RIE based system are very promising in terms of transfer of sub 100 nm size functional structure into the membrane. During the preparation of the test sample, we suggested possible ways to fine-tune the etching operation in RIE and we proposed recipes for molybdenum hard mask and silicon nitride etching.

Based on several experiments and on our expertise in lithography, we were able to design a functional technological process for the preparation of nanostructured membranes for various applications. The functionality of the presented fabrication procedure was verified with the preparation of a membrane based NOD that was successfully used in the study of electron beam diffraction in the SEM/TEM [14]. We expect a further development and new promising applications for patterned silicon membranes in SEM/TEM system, eg, phase plates, vortex-generating structures as well as in other fields as laser, EUV, optical fibers light processing and s engineering.

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