Proceedings

SAFE 2009

12th Annual Workshop on Semiconductor Advances for Future Electronics and Sensors

ProRISC 2009

20th Annual Workshop on Circuits, Systems and Signal Processing

November 26-27, 2009
Veldhoven, The Netherlands

ProRISC

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Fabrication of mechano-optical sensors for hydrogen gas

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Abstract— **We present results related to the fabrication of a novel and highly sensitive mechano-optical sensor for hydrogen gas, based on microcantilevers, supplied with a selective gas absorbing layer (Pd), suspended above a Si_3N_4 grated waveguide (GWG). Integrated microcantilever-GWG devices have been fabricated successfully using MEMS techniques. Several technical problems encountered during the preparation of such integrated devices (i.e., grating production, surface roughness, facet quality) will be discussed and solutions to address these issues will be given as well.**

Index Terms— **micro-cantilevers, grated waveguide (GWG), laser interference lithography (LIL), mechano-optical sensors, RIE, TMAH.**

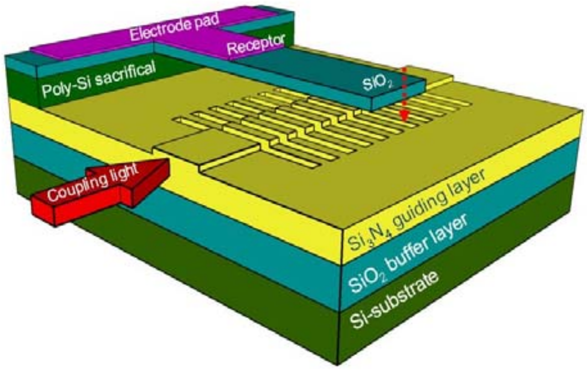

Fig. 1. The grated waveguide-cantilever device

I. Introduction

Waveguide gratings are often referred to as one-dimensional (1D) photonic crystals, which have a periodic variation of the dielectric constant along the propagation direction. An important property of a grated waveguide (GWG) is the occurrence of fringes in the transmission spectrum near the stop-band edges. It is well known that these oscillations are due to Fabry-Perot resonances of Bloch modes propagating in the cavity defined by the grated section [1]. Based on this property of GWGs, a demonstration of the potential of such structures for sensing of index changes was reported using a cavity with a high quality factor (high Q) [2]. In addition, the potential of micro-cantilevers to convert concentration changes efficiently into displacements was also demonstrated [3-5]. For these reasons, we were motivated to integrate a GWG and a microcantilever into one chip as a novel compact mechano-optical sensor for hydrogen gas. Such a sensor enables to detect the concentration of hydrogen gas through the change of nanodisplacements of the microcantilever, which is monitored optically by shifts of resonance peaks of the transmission spectrum [6]. A picture of the envisioned device is given in Fig.1. The receptor layer applied on top of the cantilever is Pd, which is selective for hydrogen gas. In this paper we present the fabrication process, developed for the functional optimization of the integrated chip.

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II. Fabrication Procedure

The process flow chart of the fabrication of the device is shown in Fig. 2. An 8-µm thick SiO_2 buffer layer was grown on a (100) Si wafer, using thermal oxidation. Next, a 275-nm thick Si_3N_4 core layer was deposited using the stoichiometric LPCVD technique. The refractive indices of Si_3N_4 and SiO_2 are 1.981 and 1.445, respectively. A 5-µm wide ridge waveguide, with 5 nm ridge height, was defined using photolithography, and etched into the Si_3N_4 layer using the BHF wet-etching process. The 490-nm period gratings were defined with laser interference lithography (LIL), using a Lloyd's-mirror-setup, producing a pattern size $2.7\times10\ cm^2$. For our application, small alignment patterns needed to be added at strategic positions in order to be able to align gratings and cantilevers perpendicular to each other. A photolithographic mask was used to define the size (number of periods) and position of the gratings. The grating patterns were transferred into the Si_3N_4 layer using reactive ion etching (RIE).

In a next step, a 400 nm sacrificial poly-Si layer and an 800 nm TEOS SiO_2 layer were deposited using LPCVD techniques. Microcantilever patterns were defined on a TEOS SiO_2 layer by conventional photolithography and by removing unnecessary SiO_2 areas using RIE. Then, metallic layers, viz. a 10 nm Cr adhesion layer and a 50 nm Pd receptor layer, were sputter-deposited at room temperature, and patterned using a lift-off process. Finally, microcantilevers were released using a 5% TMAH wet-etching solution at 70^oC to remove the sacrificial poly-Si layer, followed by a freeze-drying process.

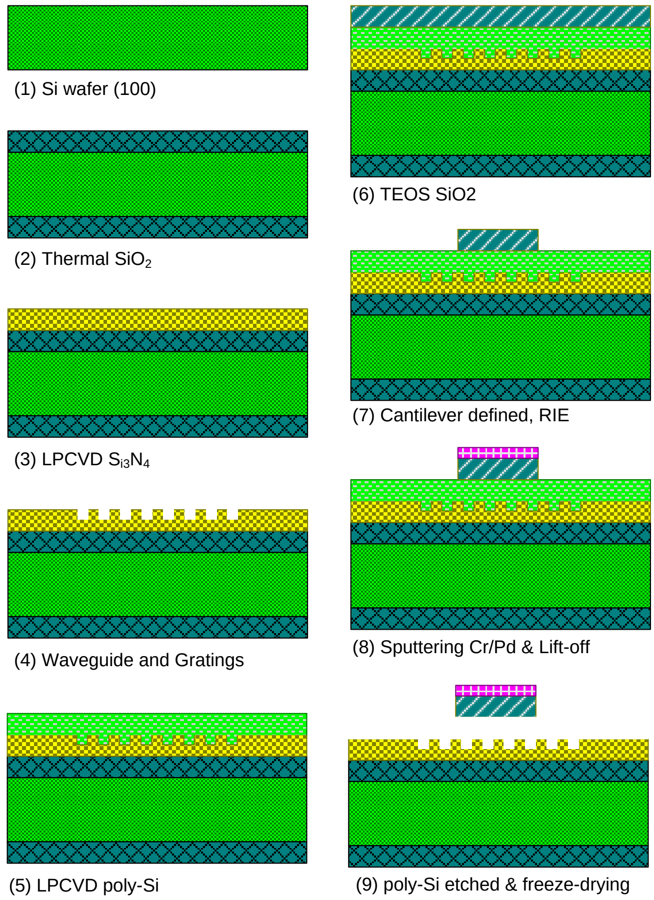


Fig.2. Process flow chart of fabrication of an integrated mechano-optical sensing device

III. Results and Discussion

Several processing steps needed to be investigated and optimized in order to obtain good-quality devices.

The LIL nanolithographic process used for creating the resist pattern for the gratings should preferably produce gratings with 50% duty-cycle. The duty cycle of the resist pattern increases with the exposure dose. The 50% target duty cycle was attained with a 3.3-mJ/cm^2 dose using a 20-s exposure time.

The RIE process for transferring the grating pattern into the Si_3N_4 layer was optimized for uniformity and aspect ratio. As a result, the Si_3N_4 gratings were etched in an O_2(10 sccm):CHF_3(100 sccm) plasma at 40 mTorr, 250 W, for 2 min. Figure 3 shows an SEM image of the fabricated grating with aspect ratio, period and uniformity as desired.

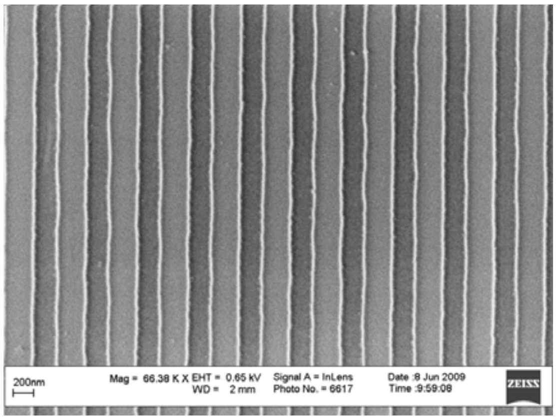
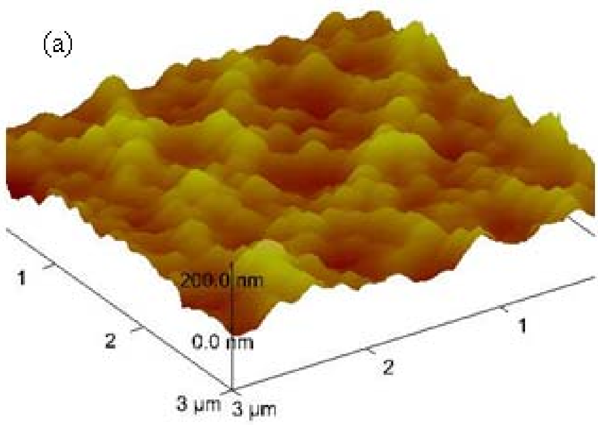


Fig.3. SEM image of a grating with period of 490 nm, fabricated by laser interference lithography (LIL)

Two possible methods for selective removal of the sacrificial poly-Si layer, needed to release the microcantilevers, have been investigated, viz. SF_6 plasma dry-etching and TMAH wet-etching. In particular the impact of etching on the wave-guiding material (i.e., Si_3N_4 underneath the poly-Si) was investigated using AFM topographic surface measurements of the Si_3N_4 layer. Figure 4 shows the surface roughness of the Si_3N_4 films caused by the dry-etching and wet-etching processes. The RMS roughness values (R_q) in both cases are 25.6 nm and 0.46 nm, respectively. This result means that the TMAH wet-etching solution did not damage the Si_3N_4 surface. On the contrary, the SF_6 plasma dry-etching did destroy the Si_3N_4 ridge waveguide, which has a height of 5 nm. Therefore, the TMAH wet-etching is a good candidate and a proper selection for releasing the microcantilevers in our fabrication process.



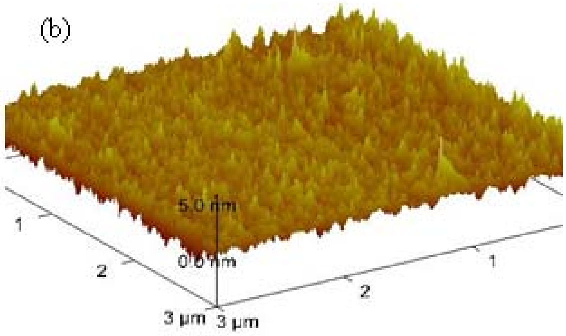


Fig.4. AFM topographic images of Si_3N_4 surface roughness, affected by (a) SF_6 dry-etching (R_q=25.6nm) and (b) TMAH wet-etching (R_q= 0.46 nm)

Microcantilevers with various dimensions were released by etching the sacrificial poly-Si underneath. Figure 5 shows an SEM image of a 50-µm long cantilever. Due to residual stress between SiO_2 and metallic layers, the microcantilevers showed an initial bending after release (1 µm at the tip in this case). Although the initial bending could be compensated by applying a DC voltage to the metal pad we are currently investigating how to minimize it.

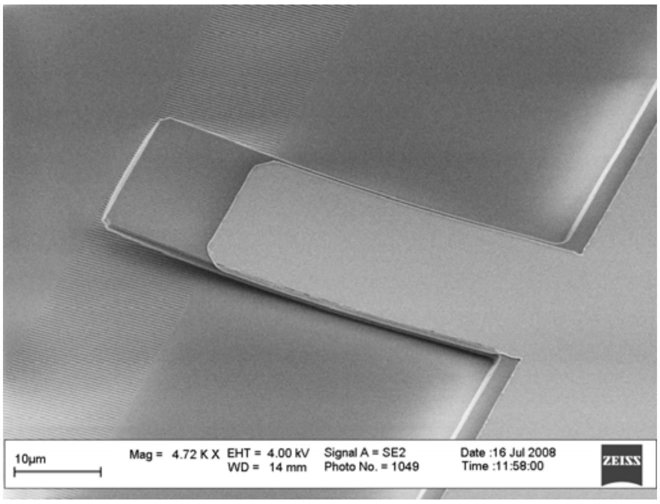


Fig. 5. SEM image of the fabricated device with a suspended cantilever above a 490 nm periodic grating

For the optical characterization, sensor chips need to be diced from the wafer. This step was carried out by cleaving the wafer which introduced very rough end facets, which reduces the efficiency of coupling light into the chip. Therefore, a new technique has been developed for the fabrication of smooth end faces of the chip. The technique consists of the following steps (as shown in Fig. 6.):

a. Reactive ion etching (RIE) to define the end facet for the waveguiding part of the structure
b. Dicing of the chip at a safe distance from the above end-facet. (At this stage the cantilevers have not yet been released and hence are not susceptible to damage by the relatively rough dicing process.)
c. TMAH etching of Si, simultaneously releasing the cantilevers and removing part of the Si substrate to separate the chips, thus enabling fibre coupling.

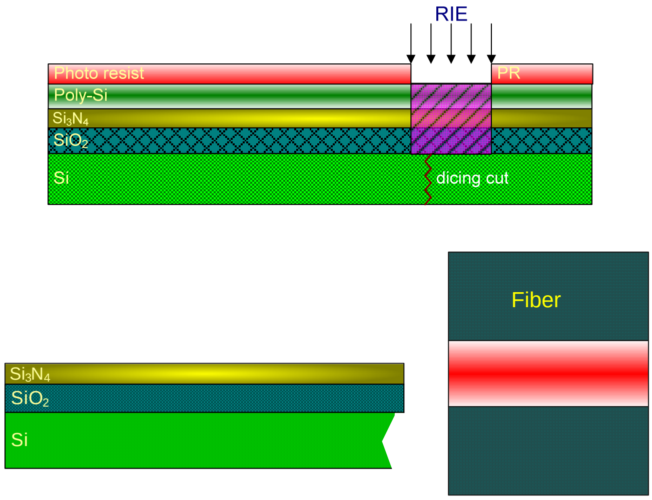


Fig. 6. *Illustration of a new technique for improving the facet quality: (upper) the RIE and dicing steps, which are followed by TMAH etching of Si (lower)*

Figure 7 shows the optical microscopic images of facets achieved by a previously used cleaving method and by a new technique, demonstrating a significant improvement. The results of the optical characterization and sensing detection will be published elsewhere.

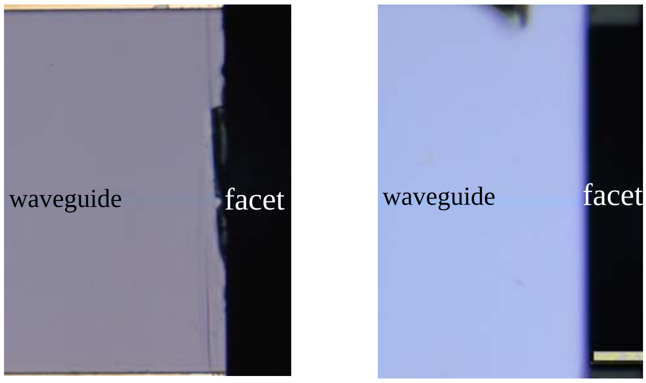


Fig.7. Optical microscopic images of facets: (left) a rough and cracked cleaved facet and (right) a smooth facet obtained by the new technique

IV. CONCLUSIONS

We have presented the fabrication of a mechano-optical sensor for hydrogen gas, based on microcantilevers suspended above a grated waveguide. An optimized process that allows the fabrication of such GWG-cantilever integration has been developed. Integrated devices with good-quality gratings and low initial bending cantilevers were realized, and a new technique for improving the quality of the end facet has been developed successfully.

ACKNOWLEDGMENT

The authors would like to thank the IOMS and MESA+ staff members for technical support. This research is financially supported by MEMSland, a project of the Point One program funded by the Ministry of Economic Affairs and the STW Technology Foundation through project TOE. 6596.

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