

# Micromachining of stabilised porous silicon with several morphological features

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A micromachining process on stabilised luminescent porous silicon (PS) using KOH was performed, for the purpose of using it in microstructures fabrication. A selective etching process using KOH aqueous solutions was developed to isolate luminescent PS regions with controlled geometrical features. As anodized PS and stabilised micromachined PS were analysed using photoluminescence and cathodoluminescence tools. The micromachining technique developed in this work is compatible with the standard MOS technology, which is to be applied to fabricate silicon luminescent microstructures.

*Keywords:* Porous Silicon, Silicon Micromachining, Photoluminescence, Cathodoluminescence

## 1. Introduction

Hitherto porous silicon (PS) is the most promising material to develop solid state sensors with a wide variety of applications. Mainly light-emitting PS is believed to be a fundamental building block for a new generation of optoelectronic integrated circuits (OEIC's) fully compatible with silicon-based microelectronics. Some light-emitting structures [1], wave-guides [2], optical cavities [3], etc., have been fabricated using luminescent PS, suggesting that silicon-based OEIC's might become reality in the very near future.

On the other hand it is well known the problem of the PL instability [4] related with ageing and post-processing, this subject remains unsolved. Some instability is characterised by a blueshift of the peak position and the degradation of the peak intensity [4]. Part of the reason is the ambiguity of the PL mechanism. Among numerous models introduced to explain the mechanism underlying the PL, the quantum confinement model seems to be the most acceptable one [5]. Recently Zhu et. al. [4] has got stability of PL in iron-passivated porous silicon, but iron is an element incompatible with IC's processing.

Usually the PL analysis is developed on the whole PS/crystalline silicon (c-Si) samples and the electroluminescence (EL) experiments are based on vertical structures Metal/PS/c-Si/Metal, were the top metal area defines the effective size of the electroluminescent device. This fact means that rarely luminescent PS is etched (after the anodization) to define geometric patterns or to confine the luminescent area, because PL instability is induced when etching post-anodization is developed. On the other hand lithographic steps have been realised previous to the selective anodization procedure, which is controlled by means of selective surface wafer masking, but in this case frequently occur undesirable lateral anodization of the patterns.

In this work we propose a new micromachining technique of stabilised porous silicon. Optical lithography and KOH

etching are utilised to develop micromachining of luminescent structures whose potential use are horizontal electroluminescent devices, and other isolated structures for optical cavities.

The post-anodization micromachined structures were analysed using room temperature PL and CL tools.

## 2. Experimental details

The rotating and temperature controlled system, recently reported [6], was employed to prepare samples with different morphologies. The substrates were P-type (14-20  $\Omega$ -cm) silicon wafers with a (100) mirror surface. After peroxide-based cleaning, the wafers were thermally oxidised at 1,100 °C during 120 minutes with an O<sub>2</sub>/TCE gas compound, and thermal annealing at 1,000 °C in nitrogen ambient was performed. After the thermal oxidation, the back-side oxide was etched with a HF aqueous solution and then using Boron Spin on Dopant this surface was P<sup>+</sup> doped. Finally, 2  $\mu$ m thick aluminium film was e-gun evaporated in order to get proper metallic contacts.

Previous to anodization, the front-side oxide was etched with the same HF solution. The silicon surfaces were anodised according to the following scheme :

The electrolyte was 49% concentrated and 30% HF aqueous solution. The samples were prepared using a current density of 20 mA/cm<sup>2</sup> during 10 and 20 minutes. Several samples (size ~ 1 cm<sup>2</sup>) were anodized at 2, 10, 20 and 40 °C, under controlled rotation of the electrolyte vessel, this arrangement produces a controlled surface structure described in a previous paper [6].

## 3. Results

### Lithography

Lithography is the process of transferring patterns of geometric shapes on a mask to a thin layer of radiation-sensitive material covering the surface of a semiconductor

wafer. These patterns define the various regions in an IC such as the implantation regions, the contact windows, and the bonding pad areas. This process was based on the commercial negative photoresist to develop several microstructures in luminescent PS.

As is known, KOH aqueous solutions are utilised to etch polysilicon films (poly), usually a thin oxide film is used to define the poly patterns, typically to develop the gate electrode of the MOS IC's. We have observed similar etching mechanism on both, polysilicon and PS, when KOH selective etching is performed. From these considerations, some geometric (square, ring, circle) and arbitrary patterns were developed over the surface of several luminescent PS samples.

The negative resist was effective to mask for several minutes selective etching of PS using KOH 40 wt.% at 40 °C. We have worked with 10 and 20  $\mu\text{m}$  PS thickness. As a function on the anodization conditions, after a few minutes these films were totally etched. When the etching step was completed, negative photoresist was striped-off using a  $\text{H}_2\text{SO}_4/\text{H}_2\text{O}_2$  solution. Fig. 1 shows a photograph of the luminescent microstructures.

In our experiments it is important remark the following ; after the complete lithographic step and KOH etching the PL disappears, but performing a final etching in HF aqueous solution the PL is recovered ; the PS microstructures still remain the same PL characteristics after anodization. This stabilised PL and the controlled structure of the PS are the key step to fabricate upgraded optical cavities [3, 7].

### Photoluminescence

The PL spectra were obtained in air at room temperature by using a Perkin Elmer LS50B instrument. The Fig. 2 shows typical PL spectra of the as anodized samples. The measurement were carried out after the samples were left in

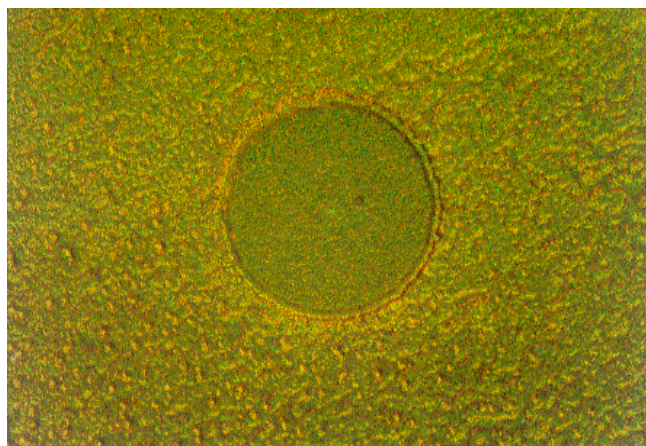


Fig. 1 Optical microscope surface image of the micromachined stabilised PS. The circle is composed by a luminescent PS 10 microns thick film.

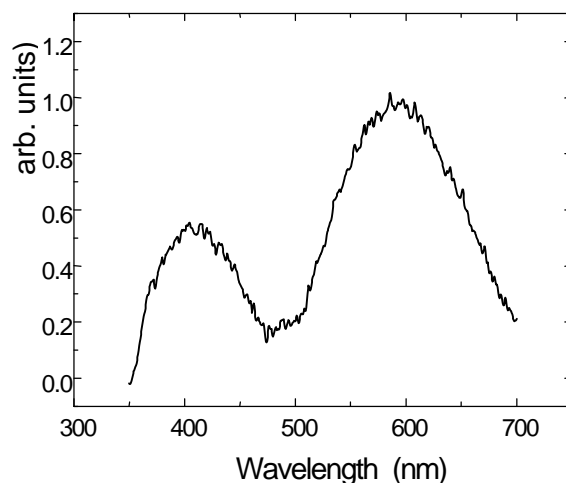


Fig. 2 Room temperature PL spectra of PS structures.

ambient air for at least three months. The PL spectra exhibited a peak at 600 nm and another component at 400 nm. Stabilised and micromachined samples show the same PL spectra.

### CathodoLuminescence

The CL spectra were obtained at room temperature. The exciting electron beam conditions were 10 - 25 KeV with a beam current of 0.5  $\mu\text{A}$ , and a beam spot diameter of 2-3 mm. As shown in Fig. 3, corresponding to an arbitrary PS pattern, the CL spectrum was dominated by bands at 425, 475, and 660 nm. There was no shift in the peak bands with the variation in the electron excitation energy.

After the KOH selective etching, in some samples a thin PS film still remains and it shows the same CL spectra. At the same time, CL emission was not detected on the silicon substrate. These facts indicate the following : a) The observed CL is originated from the PS layer and b) A very uniform emission (and uniform structure) of the PS from the surface up to the bottom is evidenced.

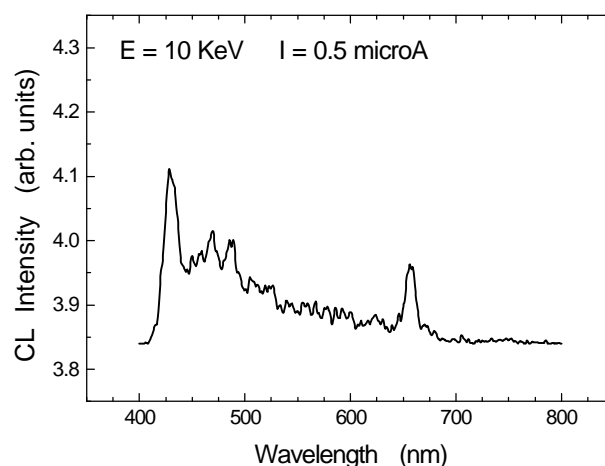


Fig. 3 Room temperature CL spectra of stabilised PS structures. The arbitrary patterns were micromachined with KOH.

#### 4. Conclusions

A micromachining process on stabilised porous silicon using KOH was performed, for the purpose of using it in microsensor devices. A selective etching process using KOH solutions was developed to isolate luminescent PS regions with controlled geometrical features. The micromachined technique developed in this work is compatible with the standard MOS technology, which is to be applied in silicon luminescent microstructures fabrication.

#### Acknowledgements

We are very grateful to Israel Fuentes Tapia and Everardo Alberto Chávez Cortes for technical support. This work was supported by CONACYT-México.

#### References

- [1] K. D. Hirschman, L. Tsybeskov, S. P. Duttagupta, and P. M. Fauchet, *Nature*, **384**, 338 (1996).
- [2] H. F. Arrand, T. M. Benson, A. Loni, M. G. Krueger, M. Thoenissen, and H. Lueth, *Electronics Lett.*, **33**, 1724-1725 (1997).
- [3] M. Cazzanelli, C. Vinegoni, and L. Pavesi, *J. of Appl. Phys.*, **85**, 1760-1764 (1999).
- [4] D. Zhu, L. Zheng, X. Li, and Y. Zhang, *J. of Appl. Phys.*, **86**, 692-694 (1999).
- [5] V. Lehmann and U. Gosele, *Appl. Phys. Lett.*, **58**, 856-858 (1991).
- [6] R. Osorio, C. Vázquez, W. Calleja, D. D. Allred, and C. Falcony, *Thin Solid Films*, **338**, 100-104 (1999).
- [7] A. Arena, S. Patane, G. Saitta, S. Savasta, and R. Girlanda, *Appl. Phys. Lett.*, **72**, 2571-2573 (1998).