ESRF	**Experiment title: X-ray imaging of porous silicon overlayers and membranes to characterize the first fabrication steps of gas sensor devices**	**Experiment number:** HS-259
Beamline:	**Date of experiment:** from: 26 June 1997 to: 30 June 1997	**Date of report:**
Shifts:	**Local contact(s):** Milita Silvia	***Received at ESRF:*** 2 9 AOUT 1997

Names and affiliations of applicants (* indicates experimentalists):

*Marco Servidori, CNR-Istituto LAMEL, Via Gobetti 101, I-40129 Bologna (Italy)

*Silvia Milita, ESRF, 38043 Grenoble Cedex (France)

* Post-dot scientist and PhD students at ID19 beamline

Report:

A set of samples has been prepared at LAMEL Institute from p+ (B-doped, 0.01 Ωcm resistivity) <OOl> silicon wafers by technological processes involving masking, lithography, chemical and electrochemical etching. Membranes of porous silicon have then been obtained with the following features:

1. porosity of 55%, 65% and 75%,
2. membrane sizes from 1x1 to 2.5x2.5 mm2,
3. membrane thickness of 25 pm.

Another set of identical samples have been subjected to nitridation by halogen lamp rapid thermal treatment in NH_3 atmosphere in order to passivate the porous structure against aging. The purpose of the investigation was to visualize and quantitatively determine the deformation state of the membranes before and after the nitridation process and to compare the images taken in the following two ways:

<u>A. ***monochromatic beam***</u>**.** Symmetric 400 Laue and 004 Bragg topographs have been taken on all samples with a beam conditioned by a vertical double 111 Si monochromator and a 004 Si crystal, the last having its diffraction plane perpendicular to that of the double monochromator. The samples have been mounted in parallel configuration with respect to the 004 Si crystal, so that the topographs were π images. This geometry was chosen to avoid the

beam-chromaticity-induced vertical translation of the image on the sample surface, as a consequence of the sample rotation and hence to keep the small membranes into the illuminated area. In fact, several images had to be taken at different working points on the rocking curves. The energy used was 17 keV.

B. white *beam*. Another set of Laue images was taken in white beam with a narrow slit in front of the sample (section images). The diffraction plane of the samples was vertical so that the topographs were σ images.

All the images, recorded on Kodak high resolution films, showed essentially a strong orientation contrast inside the membranes, indicating a remarkable bending of the membranes themselves. At a first sight, it seems that the nitridated membranes do not have curvatures larger than those without covering with silicon nitride films. This aspect can be very important for the fabrication of the final devices, where mechanical stresses are expected to play a role during the working cycles under pulsed temperature.

Typical images are reported below, the first (Fig. 1, magnification 10x) being a monochromatic 004 Bragg topograph of a not nitridated membrane with 55% porosity, while Fig. 2 (magnification 10x) is a white beam Laue section image of the same membrane. The membrane bending is easily seen in Fig.2. From the different sets of reflecting planes in the section images, the distortion fields inside the membranes will be determined and compared with those resulting from the monochromatic topographs. This analysis is in progress.

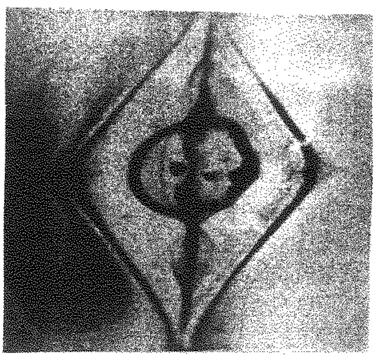

Fig. 1

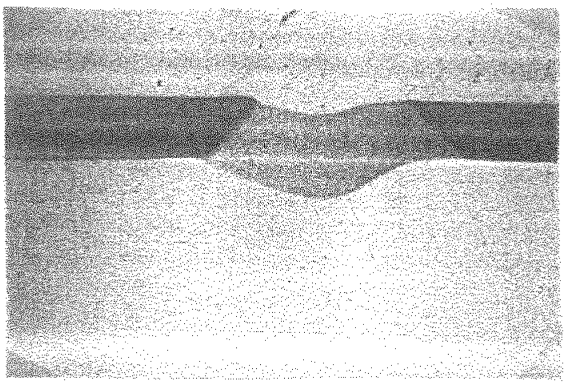

Fig. 2

The 12 shifts used for this investigation were allocated also for in-situ phase contrast and diffraction topography of sensor devices under pulsed thermal cycles (experiment HS-258). However, this part of work has not been done because of the difficulties encountered by the colleagues of LAMEL Institute in fabricating the devices in due time. These experiments will be subjects of proposals which will be submitted in the future.