

Contribution to the Study of Thermal Conductivity of Porous Silicon Used In Thermal Sensors

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Abstract—The porous silicon (PS), formed from the anodization of a p^+ type substrate silicon, consists of a network organized in a pseudo-column as structure of multiple side ramifications. Structural micro-topology can be interpreted as the fraction of the interconnected solid phase contributing to thermal transport. The reduction of dimensions of silicon of each nanocrystallite during the oxidation induced a reduction in thermal conductivity. Integration of thermal sensors in the Microsystems silicon requires an effective insulation of the sensor element. Indeed, the low thermal conductivity of PS consists in a very promising way in the fabrication of integrated thermal Microsystems. In this work we are interesting in the measurements of thermal conductivity (on the surface and in depth) of PS by the micro-Raman spectroscopy. The thermal conductivity is studied according to the parameters of anodization (initial doping and current density). We also, determine porosity of samples by spectroellipsometry.

Keywords—micro-Raman spectroscopy, mono-crystal silicon, porous silicon, thermal conductivity

I. INTRODUCTION

POROUS silicon (PS) is obtained by electrochemical attack of single crystalline silicon [1]. PS is a tuneable material in respect of its pore size distributions depending on its formation conditions. From the macro-porous silicon (with thickness bigger than 0.1 micrometer or 0.1 nanometer) to the meso-porous silicon (about 2 to 100 nanometer of thickness) to the nano-porous silicon (thickness smaller than 10 nanometer), the range of its applications varies. This material is used in photonics and optoelectronics [2], quantum electronics [3], silicon-on insulator technology [4] and very recently in sensors [5]. Since the last decade, PS has also been investigated in the area of photovoltaic [6] as an antireflection coating and as a sacrificial layer for the layer transfer process (LTP) [7]–[8]. This material presents a thermal conductivity near to thermal conductivity of silicon dioxide [9]. It is an excellent candidate to ensure the thermal insulation for the micro sensors on silicon because it ensures the mechanical stability of the microstructure [10].

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For this reason, PS layers have been effectively used as material for local thermal isolation on bulk silicon [11], [12] and as material for the fabrication of micro-hotplates for low-power thermal sensors [13], [14]. Nevertheless, before implementing the PS sensor, it is necessary to understand the thermal transport in such nanostructure and the experimental study of its thermal conductivity, according to its characteristic parameters.

In this work, we are interesting in anodization of silicon to release the principal factors, acting on the morphology in-depth of the layer. We present porosity results obtained by spectro-ellipsometry and thermal conductivity of PS layers by micro-Raman spectroscopy. The analysis of surface and in-depth distribution of the crystallites composing the material is presented.

II. PROCEDURE EXPERIMENTAL

The PS samples were prepared by electrochemical anodic etching of p -type, (100)-oriented, $0.01\text{--}0.025\ \Omega\cdot\text{cm}^{-1}$ silicon wafers, in a solution of HF (48%): $\text{C}_2\text{H}_5\text{OH}$ of 1:1. Current densities range were 5 and 75 mA/cm^2 during 10 min. The current density and the time attack selected permit to obtain different layers with different porosities and different thicknesses. After the electrochemical treatment, samples were rinsed in deionized water and were dried under nitrogen flow.

Thereafter, a thermal oxidation at low temperature (300°C) under dry atmosphere of O_2 is applied on these samples in order to stabilize the whole structure mechanically and to allow carrying out following treatments [15]. We called this stage “pre-oxidation”.

Meso-porous layers of $175\text{-}\mu\text{m}$ thickness were obtained by complex methods then were oxidized under dry atmosphere at different temperature (between 300°C and 700°C).

We determine porosity of the materials by the spectroellipsometry technique.

The thermal characterization was carried out by micro-Raman spectroscopy in order to determine the size of crystallites of PS as well as thermal conductivity according to the parameters of anodization.

III. RESULTS AND DISCUSSION

The analysis by spectroellipsometry permit to determine the indexes of refraction (n , k). From n and k , we deduce the

value of samples porosity. We use the model of Bruggmann as given by Aspnes [16]. The results are summarized in table 1 as follows:

TABLE I

SAMPLES POROSITY OBTAINED FROM SPECTROELLIPSOMETRY

Curent density (mA/cm ²)	Porosity (%)
1	17
5	22

The results presented in table 2 of the crystallites size are obtained from the measurement of the width of middle height of Raman peak function [17].

TABLE II

CRYSTALLITES SIZES OBTAINED FROM THE RAMAN ANALYSES

Porosity (%)	Crystallites size (nm)
20	9,5
48	8,7

The exploitation of Raman spectra can lead to the determination of crystallites size only when the incidental power laser is low for not provokes the heating of the surface (which would modify the position and the shape of the spectra) (Fig. 1).

The method of measurement that we developed is founded on two effects complementary to the micro-Raman spectroscopy. The first is the use of the laser beam localized on the surface of material, like local source of heating. The rise in the temperature resulting depends mainly on thermal conductivity from the sample. The second effect is the displacement of the peak Raman with the temperature of the sample, which makes it possible to deduce the local rise in the temperature from material.

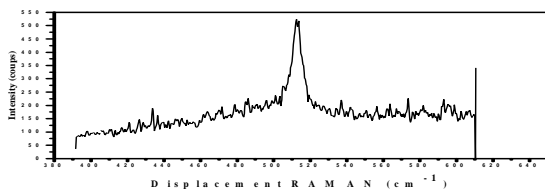


Fig. 1 Raman spectra of the sample (J=5 mA/cm², t=10mn)

By using the linear model suggested by Nonnenmacher [18], the thermal conductivity K_s of a material in thin layer can be measured by (1):

$$Ks = 2P / \pi a(T_j - T_s) \tag{1}$$

P the power of heating, T_j the temperature of surface and T_s the temperature of the substrate, a the diameter of the laser spot.

We obtained the following results. The porosity is directly, related to the nano-structure of material. It is the principal parameter acting on thermal conductivity. Fig. 2 shows that thermal conductivity decreases when porosity increases. This is explained by the effect of reduction of the effective size of the nano-crystals silicon; when porosity increases, crystallites size decreases as well as surfaces of interconnection between crystallites what more reduces the thermal way by conduction in the silicon skeleton.

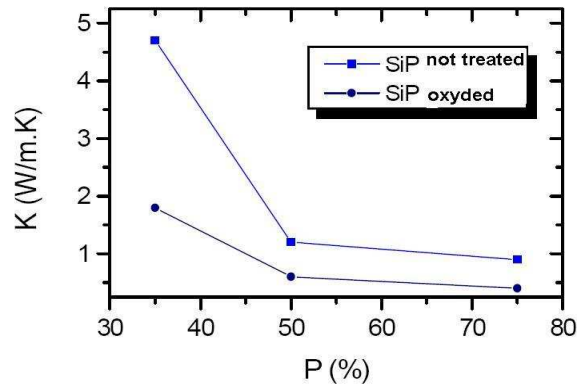


Fig. 2 Thermal conductivity of meso PS samples oxidized 1h under dry O₂

The variations of thermal conductivity with the temperature of porous silicon are presented in Fig. 3 for three porosities. Each sample has a thickness of 50 μm. Their thermal conductivity is measured after anodization and after oxidation during 1 hour, in dried O₂ at 150°C, 300°C and at last at 450°C. Whatever their porosity, the thermal conductivity of the samples follows the same evolution. Conductivity decrease initially until reaching a minimum then increases slightly

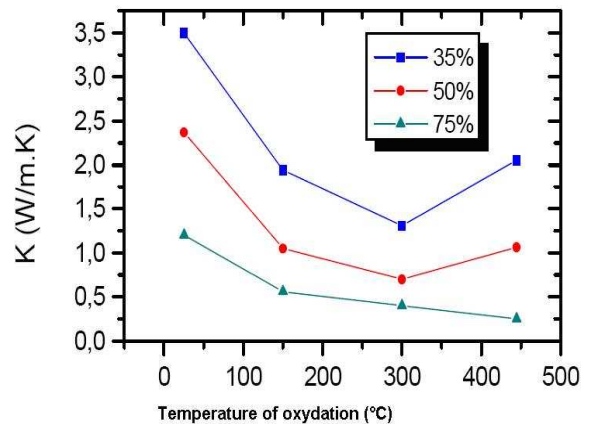


Fig. 3 Thermal conductivity of meso-PS

IV. CONCLUSION

In this study we examine porous silicon samples by spectroellipsometry which inform us about the porosity of the layers. The micro-Raman spectroscopy enabled us to obtain thermal conductivity of porous silicon. This thermal conductivity of PS was studied according to the parameters of anodization. Thermal conductivity strongly decreases when the layer porosity increases. Oxidation under dried O₂ induced a

strong decrease of conductivity to reach a minimum. The thermal conductivity of PS is quite lower than that of mono-crystal silicon. Thermal transport in the porous silicon layers is limited by its porous nature and the blocking of transport in the silicon skeleton what supports its use in the thermal sensors.

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