THERMAL ANALYSIS AT THE NANOSCALE

Yonathan Anahory, Rachid Karmouch, Jean-François Mercure and François Schiettekatte
Laboratoire René-J.-A.-Lévesque, regroupement québecois
sur les matériaux de pointe (RQMP), Université de Montréal,
C.P. 6128 succ. centre-ville
H3C 3J7,Montréal, Canada
y.anahory@umontreal.caT

Nanocalorimetry is a technique that makes possible the characterization of nanostructures from the thermal point of view. It is based on similar principles as DSC but, by reducing the calorimeter cell size to a 25-nm-thick Pt strip supported by a 100-nm-thick SiN_x membrane (Fig. 1) we are able to obtain a scanning rate reaching 10^6 K/s and to reduce significantly the heat losses. Therefore, it is possible to reach a sensitivity up to 0.2 nJ/k on the processes occurring in the deposited materials. This procedure has already been used to measure the melting point depression of nanostructures down to 2nm. [1]

A detailed description of nanocalorimeter fabrication can be found in Ref. 2. A 100 nm low-stress SiN_x layer is deposited on both sides of a silicon wafer by low-pressure chemical vapor deposition. On one side we first deposit a 3 nm Ti adhesion layer by evaporation followed by a 25 nm Pt patterned layer deposited by sputtering. On the other side, a rectangular opening is performed in the SiN_x by reactive ion etching followed by silicon etching in tetra methyl ammonium hydroxide. We thus obtain a SiN_x rectangular membrane supporting a Pt strip.

During nanocalorimetry experiments, the power required to increase the temperature of each calorimeter is calculated from the measured voltage drop across the Pt heater and the current flowing through it. The electrical resistance of the Pt strip is used to calculate the temperature of the heater as a function of time.

We used this technique to study the thermal processes involved in the annealing of damage induced by ion implantation in silicon. To achieve this, we deposited a 140 nm layer of Si (Fig.1b) on the SiN_x membrane by plasma sputtering. After ion implantations of 30 keV Si in the layer, we performed nanocalorimetry scans *in situ*. One scan lasts typically 20 ms to reach about 600 K and released a relatively uniform amount of heat over the temperature range, independent of the implantation fluence. From these results we conclude that the heat release is mostly due to the annealing of highly damaged zone rather than interstitial-vacancy recombination. [3]

We now develop an isothermal nanocalorimetry system. Using the same nanocalorimeters with a two-level current supply, the temperature is quickly increased to a desired value and then maintained constant by providing to the nanocalorimeter the power to compensate thermal losses. The temperature evolution measured during our first experiments on unmodified nanocalorimeters is shown in Fig. 2, showing the rising and stabilization time. First released heat experiments on implanted nanocalorimeters will be presented. This new system will allow us to better understand the kinetics of the occurring processes.

In conclusion we explain the principles of nanocalorimetry and how it can be used to study the thermal properties of materials that can be deposited in small quantities over a thin SiN_x membrane. We summarize the results obtained with low-energy self implanted poly-Si.

References:

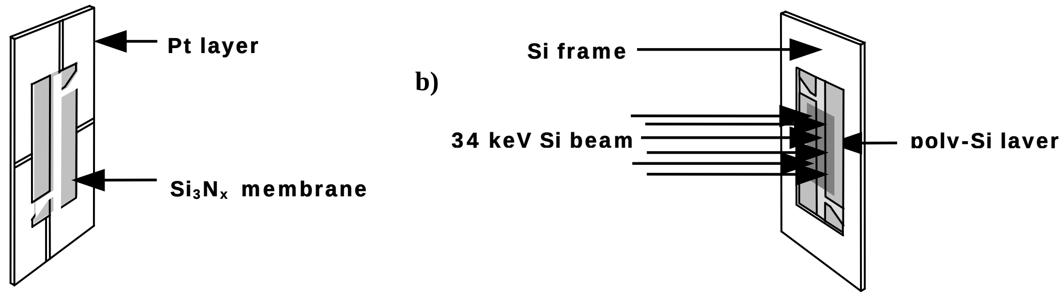
[1] M. Zhang, Yu. Efremov, F. Schiettekatte, E.A. Olson, A. T. Kwan, S.L. Lai, T. Wisleder, J. E. Greene, L.H. Allen. “Size dependent melting point depression of nanostructures: Nanocalorimetric measurements” , Phys. Rev. **B 62** (2000)10548

[2] R. Karmouch, J.-F. Mercure, F. Schiettekatte. “Nanocalorimeter fabrication procedure and data analysis for investigations on implantation damage annealing”,Thermochimica acta,*In press* (2005) .

[3] J.F. Mercure, R. Karmouch, S. Roorda, F. Schiettekatte, Y. Anahory. “Radiation damage in silicon studied in situ by nanocalorimetry”, Physica **B 340-342** (2003) 622

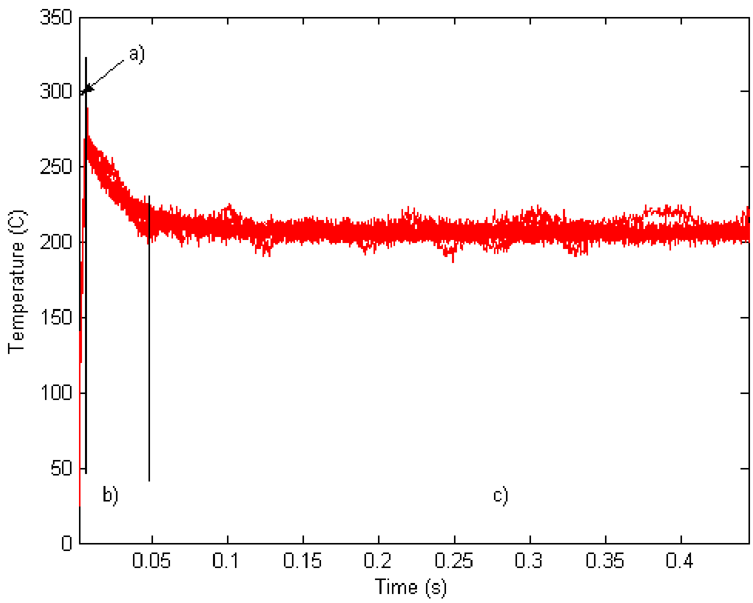
Figure 1

a)



a)Front side and **b)** back side of a nanocalorimeter with a poly-Si layer during ion implantation.

Figure 2



a) Initial heating regime (5 ms) **b)** stabilisation regime (40 ms) **c)** The isotherm plateau (400 ms) for 12 scans