A combined FEG-SEM and TEM study of silicon nanodot deposit

P. Donnadieu [a,*], F. Roussel [b], V. Cocheteau [c], B. Caussat [c], P. Mur [d], E. Scheid [e]

[a] SIMAP, INPGrenoble-CNRS-UJF, BP 75, 38402 Saint Martin d'Hères
[b] CMTC INPG, Domaine Universitaire, BP 75, 38402 Saint Martin d'Hères
[c] LGC/ENSIACET/INPT, 5 rue Paulin Talabot, BP1301, 31106 Toulouse Cedex 1
[d] CEA/LETI–MINATEC, 17 Avenue des Martyrs, 38054 Grenoble Cedex 09
[e] LAAS, Avenue du Colonel Roche, 31077 Toulouse Cedex

Résumé – Silicon nanodots deposited on a SiO_2 substrate have been studied by several electron microscopy techniques to estimate size and density, both quantities being of high interest for functional device application. The present results point out the efficiency of image treatment to improve the FEG-SEM image quality and electron diffraction to get quantitative information on the dot assembly.

Silicon nanodots are are increasingly attracting attention because of their unique physical properties that allow to develop new silicon based functional devices. Various elaboration methods have been proposed such as chemical vapor deposition, ion implantation, aerosol …techniques. Since, for floating gate memory applications, densities should be in the range 10^{11} to 10^{12} dots/cm^2 and the nanodot mean diameter between 2 and 5 nm [1], Low Pressure Chemical Vapor Deposition (LPCVD) seems to be the most appropriate methods [2]. However, industrial applications require to produce silicon controlled size, size distribution and density nanodots. This leads to a large effort in modelling the LPCVD process and developping characterization tools and procedures to validate the results of the performed calculation. Nevertheless, high density deposited nano-objects are difficult to characterize due to the nanometer range of the separating distances between dots. Therefore we are left mainly with electron microscopy techniques. The present communication is focussed on the electron microscopy characterization of a dense assembly of silicon nanodots using the combination of Field Emission Gun - Scanning Electron Microscopy (FEG-SEM) and Transmission Electron Microscopy (TEM).

1. FEG-SEM imaging

A Zeiss ultra FEG-SEM was used to carry out plane view images of the nanodot deposits. To reach better resolution, the in lens dectector is used. Besides, to improve the dot contrast, a low voltage is chosen. Because the nanodots are deposited on an insulating silica layer, imaging with a too low voltage involves a charge effect of the sample. A good compromise in terms of dot contrast is obtained with 8 keV voltage. Because of the weakness of the dot signal with respect to the background, the obtained SEM images still have a quite poor contrast as illustrated by Figure 1 a. An obvious way to improve the image quality is to use a pass band filter to cut the non significant information : i.e. the signal from objects larger than 20 nm and below 2 nm is removed.

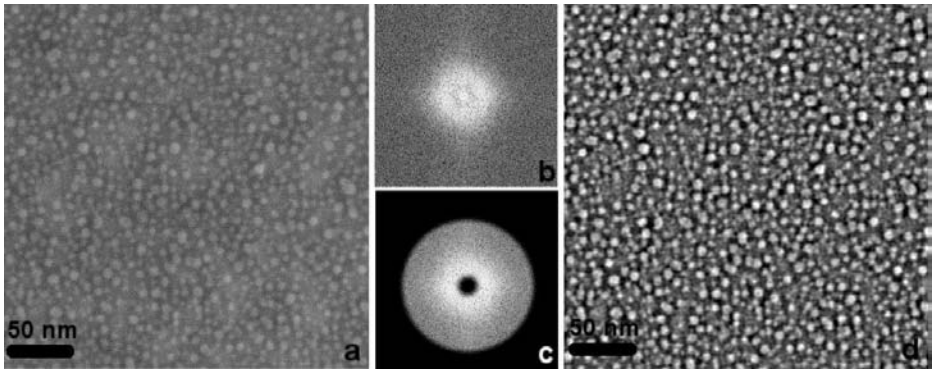


Figure 1 *– (a) FEG-SEM image of the nanodot assembly obtained with the in lens detector at 8 keV (b) FFT of image (a); (c) mask applied to (b); (d) filtered image derived from image in Fig. 1a*

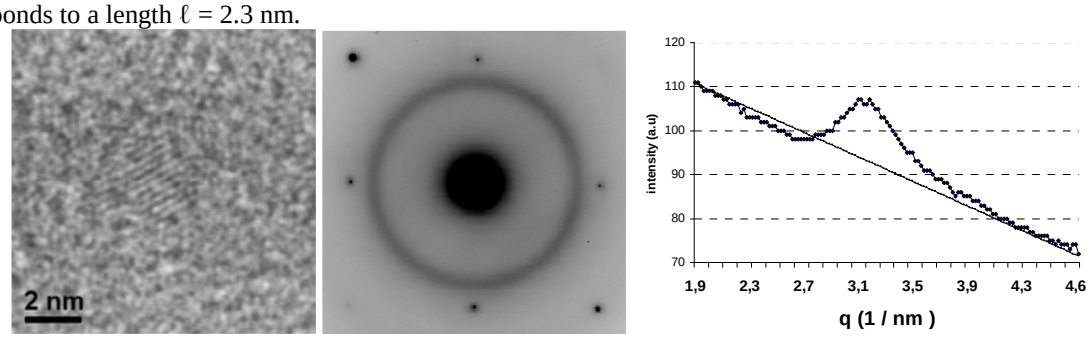
Figure 1 shows the original image and the filtered one, obtained using FFT transforms. The filtered SEM images gives a dot density of 1.6 10^{12} /cm^2 for a dot diameter of about 7 ± 2 nm However the resolution does not seem high enough to separate dot aggregates and the density is underestimated. To check the dot size using a higher resolution technique, is then imperative..

* Auteur à contacter : patricia.donnadieu@ltpcm.inpg.fr – Tel : 04 76 82 67 46

2. HRTEM and electron diffraction information

The most appropriate way to study on substrate deposited nano-objets is to prepare a plane view sample.. First, the sample is mechanically side up polished from 750 μm to about 50 μm. and further thinned to reach the electron transparency by chemical etching or by ion milling.

As shown in figure 2a, nanodots are imaged by HRTEM essentially as a stacking of fringes. The distance between fringes is about 0.310 nm which is consistent with the (111) plane spacing in Silicon (0.313 nm). According to the HRTEM images the dot size is about 3 nm which appears quite smaller in comparison with the dot size determined by FEG-SEM images. As illustrated by figure 2a, a simple approach consists in using electron diffraction.., The nanodot assembly gives a quite intense ring centred on a position close to 0.32 nm which roughly correspond to the (111) silicon plane spacing (0.313 nm). The homogeneous intensity of the diffuse rings indicates that the nanodots deposit is isotropic. The graph (Figure 2c) displays the intensity profile of that diffuse ring. After background subtraction, the ring at mid height is equal to $\Delta q = 1.1$ nm^{-1} which corresponds to a length $\ell = 2.3$ nm.



(a) (b) (c)

Figure 2 *– (a) HRTEM image of a nanodot, its size is about 3 nm (b) electron diffraction pattern of the plane view sample. The nanodots correspond to the intense diffuse ring (c) Intensity profile across the rings allowing to measure (after background subtraction) the ring width at mid height*

To intrepret the ring width the shape factor has been calculated for several shape function like a crenel, a traingle or a flake shape. The latter one gives the best agreement between the HRTEM image since the experimental ring width $1/\ell$ ($\ell = 2.3$ nm) would correspond to a size L = 3.6 nm for dot with a flake shape. This results points out the efficiency of electron diffraction for characterizing an assembly of nanoobjets. However our intrepretation of the ring width consider only the shape effect while deformation and defects can also contribute to the width.

3. Conclusion

According to the FEG-SEM, the nanodots size is about 7 nm while electron diffraction and HRTEM indicates rather smaller size. THis means that still more TEM analysis are necessary to precise the actual silicon dot size and density. The phase retrieval method, used earlier, appears to be the most appropriate to fill this gap in terms of resolution and dot assembly images [3]. It is worth insisting that nano-objects of 5 nm size, separated by distance below 10 nm, still represent a challenge for electron microscopy technique since at that scale objects may present strain or ill defined crystalline states. In that respect electron diffraction has a major interest since it is able to test a large amount of particles and is sensitive to size as well as strain. Of course, as most of the nanocharacterization techniques, combining all information is essential.

4. Références

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