doi:10.1017/S1431927611004156
Microsc. Microanal. 17 (Suppl 2), 2011

Direct Imaging of Microstructural Changes in Si Induced by FIB-Patterning with Si^{++} and Ga^{+} Ions

See Wee Chee*, Martin Kammler**, Jeremy Graham***, Frances M. Ross**** and Robert Hull*

* Department of Materials Science and Engineering, Rensselaer Polytechnic Institute, 110 8th Street, Troy, NY 12180
** Institut für Experimentelle Physik, Universität Duisburg-Essen, 47048 Duisburg, Germany
*** Department of Materials Science and Engineering, University of Virginia, Charlottesville, VA 22906
**** IBM Research Division, T.J. Watson Research Center, Yorktown Heights, New York 10598,

Focused ion beam (FIB) lithography and micromachining is widely used for the "top-down" fabrication of nanostructures [1]. Lately, new fabrication schemes have emerged that use FIB pre-patterning of a surface to direct subsequent self-assembly of nanostructures [2]. For example, self-assembled epitaxial Ge quantum dots can be templated on a Si surface by first patterning using Ga^{+} ions [3]. The resolution of FIB pre-patterning techniques depends on the specific interaction that directs the growth and the spatial range over which it operates. We have therefore examined the microstructural changes induced by FIB-patterning a Si membrane and the ensuing annealing behavior *in situ* in a transmission electron microscope (TEM) to understand the changes that take place prior to the growth of Ge quantum dots. Here, we compare Si^{++} with Ga^{+} ions, since it is highly desirable for nanoelectronic applications to avoid introducing a shallow dopant like Ga.

FIB patterning was performed by writing arrays of single-pixel spots on single crystal Si membrane samples with the dose varied through the dwell time per pixel. 30keV Ga^{+} arrays were written using a Carl Zeiss Ultra 1540 commercial dual-beam FIB system while 60keV Si^{++} arrays were written using a mass-selecting Orsay Physics Canion 31+ column. The samples were 200nm thick Si membranes made by etching the substrate and buried oxide from silicon-on-insulator wafers (Figure 1). After pattern writing, the samples were transferred to a 300kV Hitachi H-9000 TEM for imaging and subsequent annealing by resistive heating *in situ*.

Figure 2 shows a typical implanted array, in this case with $5x10^{4}$ Si^{++} ions per spot. For both Ga^{+} and Si^{++} the as-implanted spots appear similar: amorphous regions ranging in diameter from ~80 nm to a few hundred nm depending on dose. These spot sizes are larger than the lateral straggle predicted by SRIM calculations [4]. We will discuss possible origins for this discrepancy and compare our results with the wealth of literature on FIB implantations, especially for Ga^{+}.

On annealing, Ga^{+} and Si^{++} show quite different and interesting behavior. For both species, above 300°C recrystallization occurs and the spots shrink. For Ga^{+}, the only contrast remaining by ~450°C consists of small features that we believe [3] to be Ga precipitates (Figure 3). But for Si^{++} with $>5x10^{4}$ ions per spot, each spot forms an individual dislocation loop (Figure 4). For higher Si doses, these loops follow the edges of the implanted spots (Figure 5a). We will discuss this behavior in terms of the differences between Si and Ga diffusion in Si, and consider its possible uses in terms of dislocation engineering. We will also describe the use of these Si-implanted arrays for position control during subsequent growth of Ge quantum dots (Figure 5b).

References
[1] R.M. Langford et al., *MRS Bull.*, 32 (2007) 417-423.
[2] J Gierak, *Semicond. Sci. Technol.*, 24 (2009) 043001.
[3] R. Hull et al., *Mat. Sci. Semicon. Proc.*, 11 (2008) 160-168; *J. Phys.: Conf. Ser.*, (2010) 209 012003, and references therein.
[4] J. Ziegler, The Stopping and Range of Ions in Matter (http://srim.org/).
[5] The authors are grateful for the assistance of P. Balasubramanian with the operation of the Zeiss FIB, Y.-C. Chou with the Hitachi TEM, S. Ji, A. W. Ellis and M. C. Reuter with the Orsay FIB and K. B. Reuter with sample preparation.

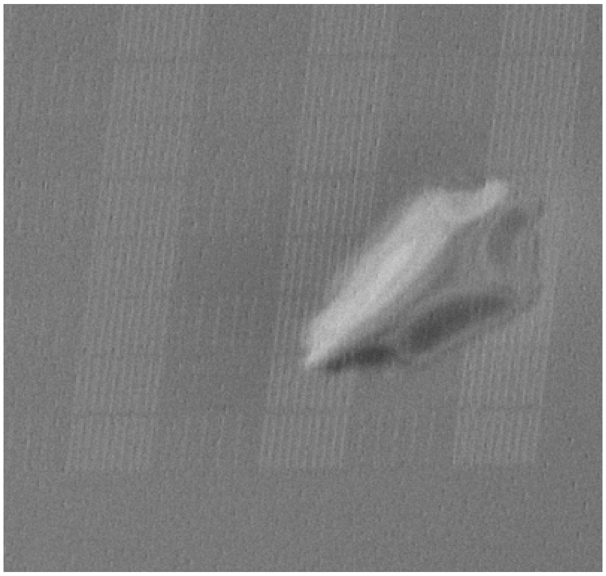
Figure 1. SEM image of a Si membrane (buckled region) after etching and patterning.

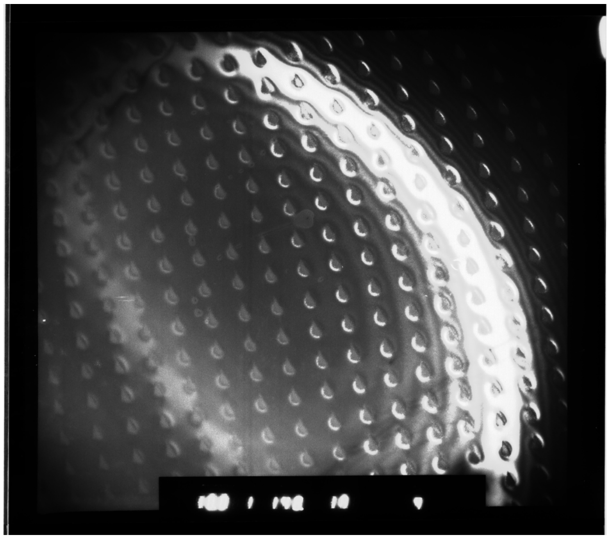
Figure 2 Dark field TEM image of a Si^{++} implanted array with $5x10^4$ ions per spot (0.5 ms dwell time.

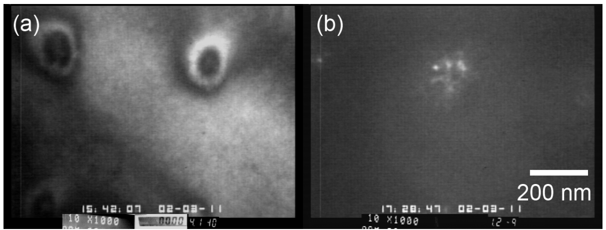

Figure 3. Dark field TEM images of Ga^+ implanted spots with $1x10^4$ ions per spot, (a) as-implanted and (b) showing Ga nuclei after annealing at 450°C.

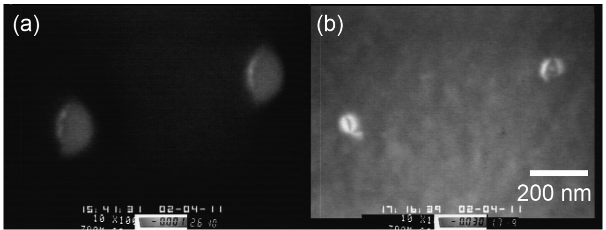

Figure 4. Dark field TEM images of Si^{++} implanted spots with $5x10^4$ ions per spot, (a) as-implanted and (b) showing dislocation loops after annealing at 500°C.

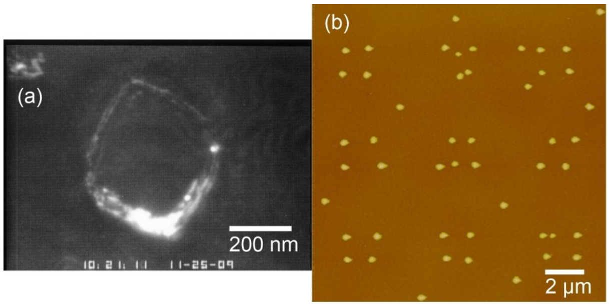

Figure 5. (a) Dark field TEM image of a spot implanted with $2.8x10^7$ Si^{++} ions and annealed to 600^oC for 5 min. (b) AFM image showing alignment of Ge quantum dots above patterns consisting of groups of four spots implanted with $3.3x10^7$ Si^{++} ions.