Electron beam induced etching of silicon using chlorine gas

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Resistless etching in the nanometer scale has a wide range of applications including chip or mask repair. While precise and fast etching can be performed by focused ion beam technique this has the disadvantage of significant gallium implantation into the substrate and amorphization of the substrate. It is therefore desirable to use electrons instead of ions which will not exhibit these disadvantages. While ions are capable of physically removing almost any material, an etching process involving only electrons will remove material chemically and hence requires a specific etching agent for each substance that shall be removed.
A well-known silicon etching agent for focused ion beam systems and electron beam systems alike is xenon diflouride (XeF_2)[1]. Although XeF_2 allows for a very efficient etching process, its property to etch silicon spontaneously (once the topmost silicon oxide layer is removed by electron beam induced etching), makes its handling difficult (spontaneous etching is often already triggered by simply scanning a certain area for imaging purposes).
Using chlorine as etching agent it is possible to convert solid silicon into volatile silicon tetrachloride (vapor pressure: 259 mbar at 20 °C):

$$Si + 2Cl_2 \rightarrow SiCl_4$$

Electron beam induced etching in a reaction cell of chromium using chlorine was first published by Sun et al in 2005[2,3]. We present a detailed experimental analysis of an electron beam induced etching process of silicon using chlorine gas as precursor that was carried out using a conventional gas injection system (GIS) setup in a scanning electron microscope (SEM). This GIS, which is equipped with a mass flow controller, has been adapted to work reliably and safely with highly corrosive and toxic substances.
The etching of an n-doped silicon substrate by using chlorine gas introduced into the SEM chamber by means of a conventional GIS is demonstrated. The efficiency of etching was observed to be up to 3 nm per minute for an area of 1.5x1.5 µm². No spontaneous etching could be observed. The influence of various etching parameters such as electron beam current, acceleration voltage, dwell time, defocusing and chlorine gas flow on the etching efficiency and the etch pits' shape have been investigated. After finishing the fabrication of the etch pits, all structures did undergo extensive AFM and EDX analysis.
Finally, the etch-inhibiting effect of an increased level of contamination in a SEM chamber will also be discussed.

[1] S. Matsui, K. Mori, Appl. Phys. Lett. 51 (19), pp. 1498-1499 (1987)
[2] Y.-M. Sun, S. Wang, J.M. White, A. Stivers, T. Liang, Appl. Surf. Sci. 252 (2), pp. 311-320 (2005)
[3] S. Wang, Y.-M. Sun, J.M. White, A. Stivers, T. Liang, JVST B, Vol. 23, Issue 1, 2005, pp. 206-209 (2005)

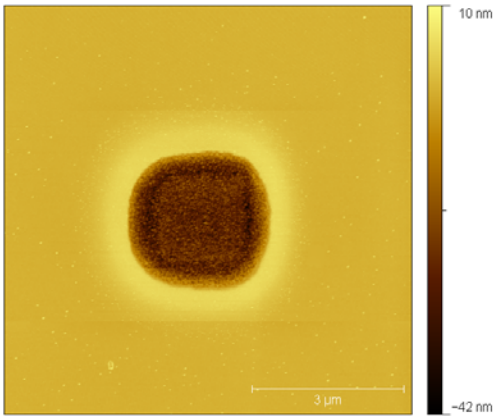

Fig. 1: 2D AFM image of an etched pit with an area of 1.5x1.5 µm². The experiment was carried out at 6 nA electron beam current using an electron beam energy of 10 keV.

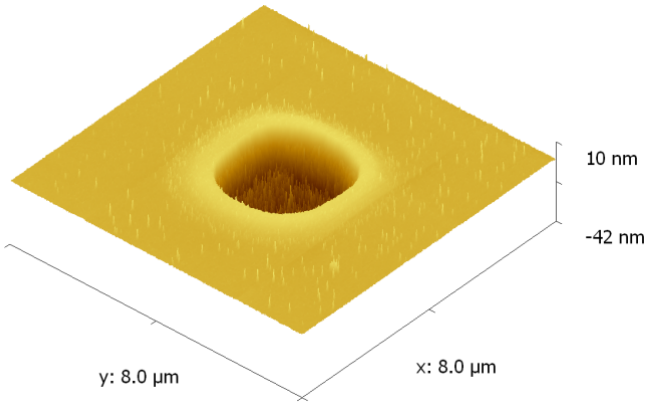

Fig. 2: 3D AFM image of the above structure. The slightly increased rim around the etch pit can be attributed to residual gas deposition and contains mostly carbon.

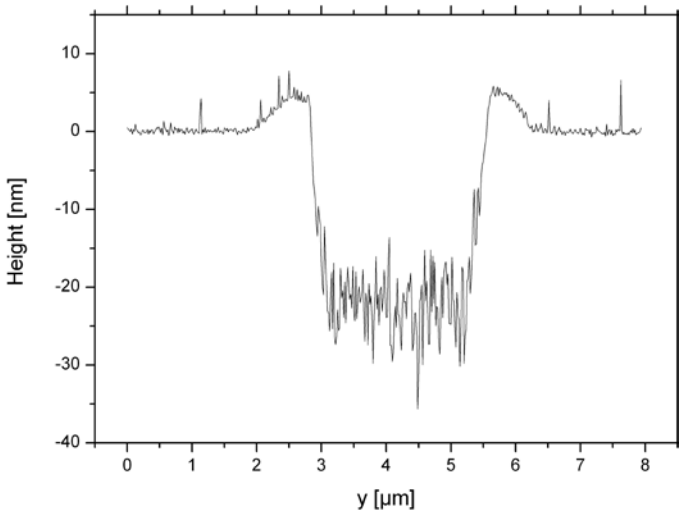

Fig. 3: Cross-section obtained from AFM analysis along the y-axis through the center of the etch pit. The rough bottom of the etch pit indicates an inhomogeneous etching process possibly caused by the doping of the substrate which might inhibit the etching