Removal of FIB-induced amorphization and gallium contamination by focused-electron-beam-induced-etching

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Recently focused electron beam induced etching (FEBIE) of silicon using molecular chlorine (Cl_2-FEBIE) has been developed as reliable and reproducible process capable of damage-free, maskless and resistless removal of silicon [1]. As any electron beam induced processing is considered non-destructive and implantation-free due to the absence of ion bombardment this approach is also a potential method for removing focused-ion-beam (FIB) inflicted crystal damage and ion implantation.
In a FIB system using gallium ions (Ga+) the gallium concentration after sample treatment close beneath the surface has been determined by secondary ion mass spectroscopy (SIMS) to exceed 40 at% for silicon depending on the gallium dose [2]. It has been shown [3] that amorphization at the bottom wall using a 30 keV focused ion beam amorphizes silicon 56 nm deep. On the other hand the side wall amorphization (important in TEM sample preparation) of silicon has been determined to 28 nm.
FEBIE has the advantage of being a localized, purely chemical process which is not accompanied by physical sputtering. It is therefore generally assumed that FEBIE does not cause surface amorphization or implantation of alien species into the substrate at all.
We show that Cl_2-FEBIE is capable of removing FIB-induced amorphization and gallium ion implantation entirely present after processing of surfaces with a focused ion beam (fig. 3).
Also, using this method it has become possible for the first time to directly investigate damage caused by FIB exposure in top-down view utilizing this localized chemical reaction, i.e. without the need of TEM sample preparation. We show that gallium fluences above 4×10^{15} cm^{-2} result in altered material caused by FIB-induced processing down to a depth of ~250 nm (see figure 2). This altered material is expected to be amorphous due to the high Cl_2-FEBIE etch rates observed. We show that the observed inhomogeneous sub-surface structure (see figure 2) is solely caused by FIB processing rather than the Cl_2-FEBIE post-processing.
With increasing gallium fluences, due to a significant gallium concentration close beneath the surface, removal of the topmost layer by Cl_2-FEBIE becomes difficult, indicating that gallium serves as an etch stop for Cl_2-FEBIE.
Finally, Cl_2-FEBIE on a silicon TEM lamella (with no post-treatment applied) has been carried out with subsequent TEM analysis (figure 4). We show that the crystalline signal could be improved after the Cl_2-FEBIE treatment. This implies that the method Cl_2-FEBIE is non-destructive and therefore retains crystallinity. From a technological point of view, the non-destructive nature of this method makes it an interesting alternative to existing post-processing techniques of TEM lamellae which currently all involve ion bombardment.

References

[1] P. Roediger, H. D. Wanzenboeck, G. Hochleitner, E. Bertagnolli and W. Buehler *Nanotechnology* **21** 285306 (2010)
[2] H. Gnaser, C. Kallmayer, H. Oechsner *J. Vac. Sci. Technol. B*, **13**, pp. 19-27 (1995)
[3] S. Rubanov, P. R. Munroe *J. Microscopy*, **214**, pp. 213-221 (2004)

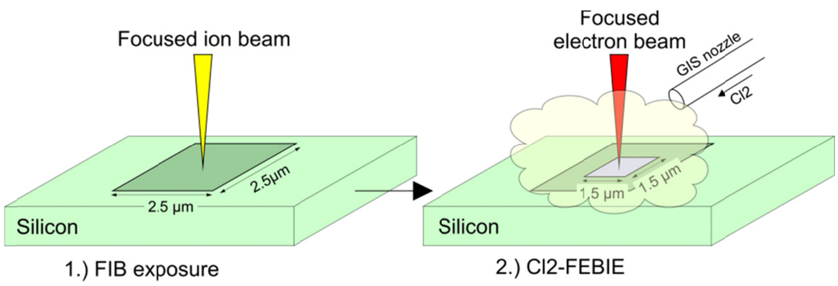


Fig. 1: Sample preparation: 1.) Square-shaped areas (2.5 × 2.5 µm²) have been exposed to 16 different Gallium ion fluences ranging from 1×10^{15} cm^{-2} up to 164×10^{15} cm^{-2}. Exposure was performed at a constant FIB current of 10 pA using different exposure times. 2) The FIB-exposed areas have been processed by Cl_2-FEBIE for 20 minutes each. The Cl_2-FEBIE processing area amounted to 1.5 × 1.5 µm².

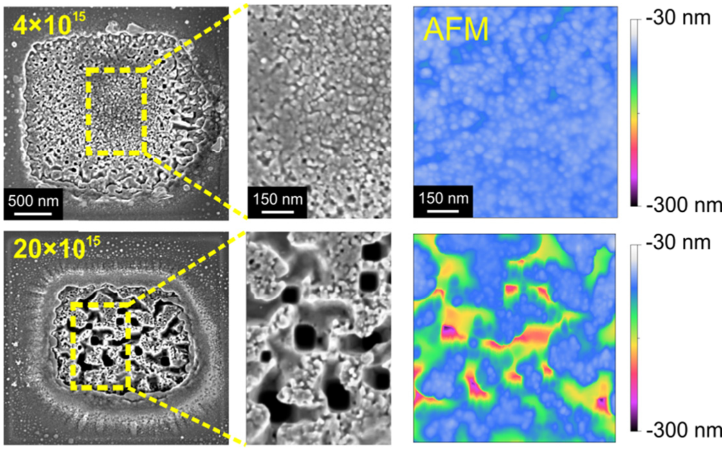
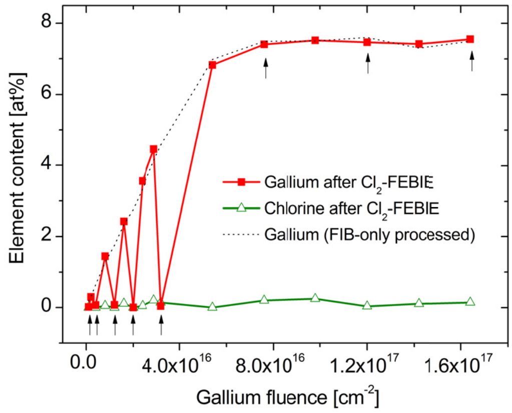


Fig. 2: SEM images and AFM analysis after Cl_2-FEBIE treatment for gallium ion fluences of 4×10^{15} cm^{-2} and 20×10^{15} cm^{-2}. For a fluence of 4×10^{15} cm^{-2} the final surface texture is homogeneous with an average depth of ~80 nm.
For the higher fluence of 20×10^{15} cm^{-2} the surface texture becomes very inhomogeneous featuring deep trenches and holes. Note that there are regions (blue areas in the AFM image) that show a similar surface texture as for the case of 4×10^{15} cm^{-2} at the same etched depth (~80 nm).

Fig. 3: EDX analysis after Cl_2-FEBIE for gallium ("Gallium after Cl_2-FEBIE") and chlorine ("Chlorine after Cl_2-FEBIE"). For comparison purposes, the gallium concentration before the treatment is shown ("Gallium (FIB-only processed)"). Note that Cl_2-FEBIE treatment was only performed for gallium fluences indicated by black arrows. The data show that the FIB-implanted gallium could be removed entirely. For Ga^+ fluences above of 70×10^{15} cm^{-2} it was not possible to penetrate the topmost Ga layer anymore, resulting in an unchanged Ga concentration after Cl_2-FEBIE.



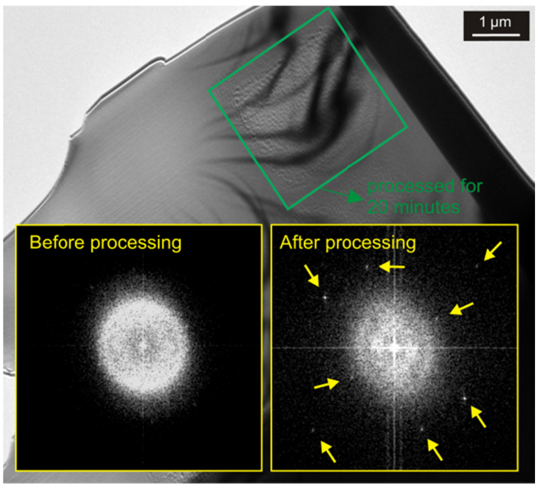


Fig. 4: TEM image of a FIB-prepared TEM lamella post-processed solely by Cl_2-FEBIE. The green rectangle (3 × 3 µm²) shows the Cl_2-FEBIE processed area. The two diffraction patterns by TEM analysis illustrate the crystallinity of the processed area before and after Cl_2-FEBIE. The yellow arrows indicate the reflections which originate from the crystalline silicon structure of the TEM lamella.
The TEM analysis proves that the crystalline signal could be significantly improved by applying Cl_2-FEBIE to a FIB-prepared TEM lamella that has not be post-treated otherwise. The results verify the assumption that focused electron beam processing is a non-destructive method that retains crystallinity.