Removal of residues from reactive ion etched silicon surfaces characterized with XPS and Raman spectroscopy

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As an example of a RIE process we used a SF₆/O₂ RIE process to fabricate silicon nanopillars, cf. Schmidt Raman Spectroscopy (SERS) processes. Here, in this work, the effect of plasma ashing and thermal treatment in an effort to remove reactant residues from the surface of RIE processed silicon was studied by XPS and Surface Enhanced Raman Spectroscopy (SERS).

Previously, Thomas et al. [2] characterized a silicon surface exposed to SF₆/O₂ RIE with X-ray Photoelectron Spectroscopy (XPS) and found that fluorine was left on the surface as a residue from RIE processes. Here, in this work, the effect of plasma ashing and thermal treatment in an effort to remove reactant residues from the surface of RIE processed silicon was studied by XPS and Surface Enhanced Raman Spectroscopy (SERS).

As an example of a RIE process we used a SF₆/O₂ RIE process to fabricate silicon nanopillars, cf. Schmidt et al. [3]. To test the hypothesis that the etchant residues can be removed from the surface by chemical reaction/spattering and/or thermal desorption, the nanopillars were subsequently treated either/both with a standard O₂ plasma ashing for 1 min and/or placed in a furnace at 800 °C for 3 hours in a N₂ atmosphere. The surface of the nanopillars was characterized by XPS and the result is shown in Figure 1 and Table 1. XPS reveals that the surface contains a mixture of mostly silicon, carbon and oxygen and as expected, also fluorine as a residue from the RIE process. The surface is contaminated with approximately 5 atomic percent fluorine after the RIE process. This does not change upon O₂ plasma ashing. However, the fluorine is practically removed after the thermal treatment.

The silicon surface is also characterized using SERS and the results are shown in Figure 2. In order to perform SERS we create plasmonic structures by evaporating 225nm of silver onto the nanopillars after plasma/thermal treatment. SEM images of silver coated nanopillars without and with thermal treatment is shown in Figure 3 and Figure 4, respectively. It can be seen that the pillars do not change shape or form after the thermal treatment, and that the coverage of the silver coating is similar in both cases, making it suitable to compare the SERS measurements. The SERS substrate now created usually performs by trapping an analyte between the nanopillars enhancing the Raman signal of the analyte. A significant part of the Raman signal also stems from the silver cavity formed at the lower part of the silver cap on the silicon pillar. This plasmonic cavity mode gives a strong Raman response from the surface of the silicon as well. This makes it excellent for analysis of the cleaning procedures. A 1µl drop of H₂O is positioned on the SERS substrate. H₂O does not give rise to a significant Raman shift with our measurement conditions. As the drop evaporates, the surface forces pull the pillars together forming clusters creating electromagnetic "hot spots" which greatly enhance the SERS signal. The signal is measured at 780nm with 0.5mW and 50X for 3x1sec. These results indicate that significant levels of etchant residues are removed from the silicon surface at 800 °C.

In conclusion, the standard plasma ashing procedure has no effect on the cleanliness of the silicon substrate after the RIE process. On the other hand, the thermal treatment of 800 °C seems to remove the unwanted fluorine from the surface. These findings can be utilized in applications where the surface of the silicon is important, e.g. in the solar cell and chemical sensing industries.

Keywords: RIE, SF₆, Raman spectroscopy, SERS, XPS, silicon nanopillars, SEM