Oxidation sharpening of silicon tips in 'air' environment

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Sharp tips are essential for high resolution AFM imaging and high performance electron emitters in vacuum microelectronic devices. Marcus et al.[1-3] reported tip sharpening method using thermal oxidation of silicon followed by oxide removal. This method relies on the fact that oxide grows slower on areas with smaller curvature of radius when the oxidation rate is limited by chemical reaction (rather than by oxygen diffusion to the Si/SiO2 interface). For tips having a cone or pyramid shape, this means that oxide film is thicker (thus more silicon is consumed) at places farther away from tip apex, leaving to sharper silicon tips upon oxide removal by HF etching. So far oxidation is carried out in dedicated oxidation furnace that is costly and the tips or wafer of tips must be cleaned thoroughly using RCA cleaning.

Here we report that oxidation sharpening can also be attained using very low cost box furnace in air environment (no pumping, purging, venting of the furnace) that doesn't require the tips to go through a RCA cleaning process. As is apparent, such cleaning is not convenient for millimeter-scale AFM probes. The obvious application is the re-generation of sharp tips out of worn out thus blunt AFM probes at very low cost.

We prepared two types of samples for oxidation sharpening study. The first type is truncated cone-shaped silicon nanostructures fabricated by electron beam lithography, metal liftoff and ICP-RIE using SF6/C4F8 gas[4,5]. The second type is random pyramid shaped structures prepared by maskless anisotropic wet etching of silicon using KOH. We subsequently annealed all samples at 1000 °C in 'air' environment for two hours using a generic box furnace. Finally, the oxide was etched away by HF. This oxidation sharpening may be repeated for several times to further sharpen the apex radius. Note that each time the oxide growth should be less than ~100 nm in order to avoid going to the regime where the growth rate is limited by oxygen diffusion through the already grown oxide layer.

It was found that the oxidation rate in air is very close to that of pure O2 dry oxidation[6] which is much slower than H2O wet oxidation. And our oxide film has an etching rate very close to that of oxide grown using costly dedicated oxidation furnace (22 nm/min for our oxide vs. 24 nm/min, etched in 1:10 diluted HF). Since silicon nitride is etched far slower than oxide in HF, our film should have negligible nitride in it even though the air consists of mostly nitrogen, which is also confirmed by EDX elemental mapping (Figure 1).

Figure 2 shows the fabricated blunt cone structure that was transformed into sharp cone (radius of curvature 7 nm) after single oxidation process in air followed by HF etching. Figure 3 demonstrates truncated cone and random pyramid structures that were sharpened after repeated oxidation sharpening process.

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[6] http://www.cleanroom.byu.edu/OxideTimeCalc.phtml

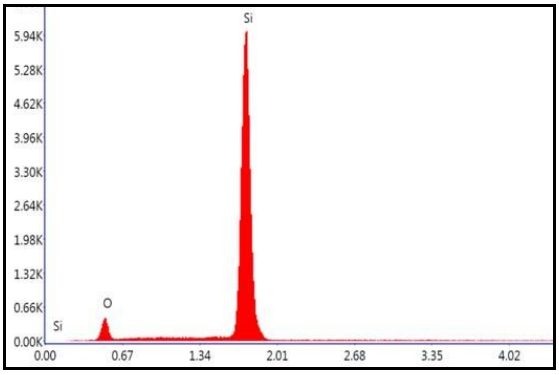

Figure 1 EDX plot of 100 nm oxide film thermally grown in air on silicon, showing Si K and O K peaks. The high intensity for the Si peak is mainly caused by the bulk silicon substrate. No nitrogen is detected in the film.

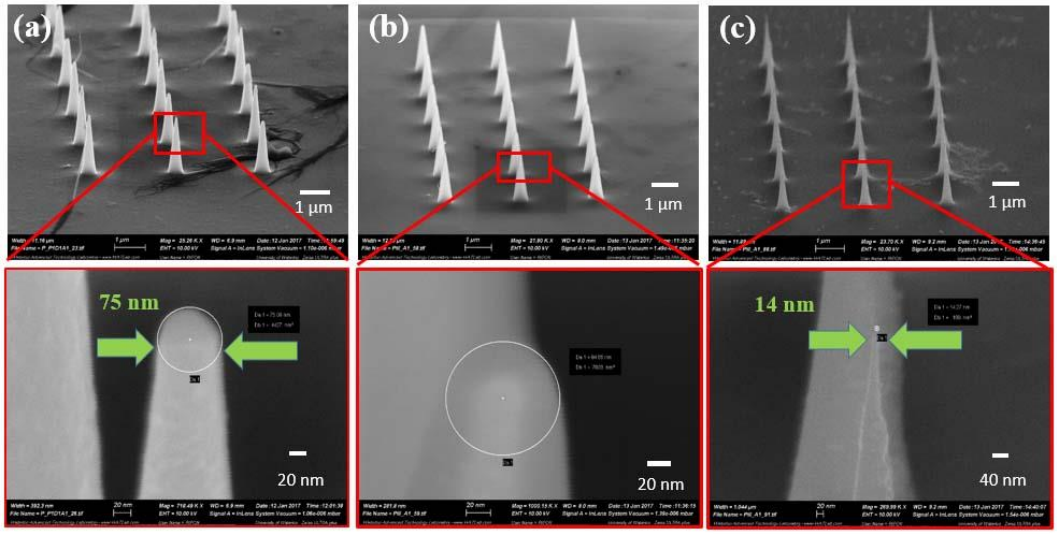

Figure 2 SEM images of cone structures fabricated on Si wafer (<100>). (a) Before oxidation in air; (b) right after oxidation; (c) after HF etching away of the oxide.

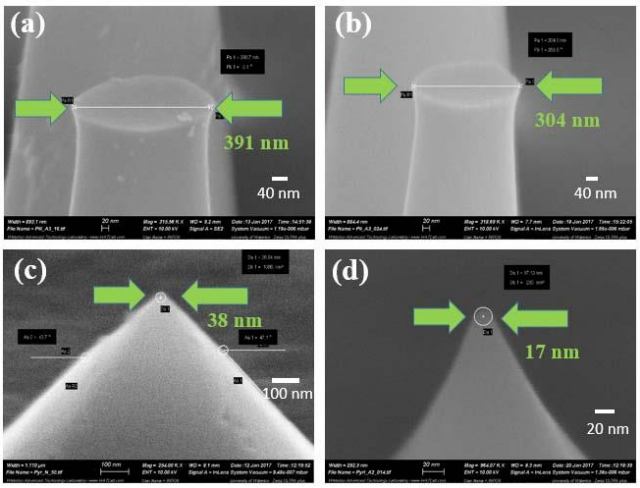

Figure 3 (a-b) SEM images of truncated cone structures fabricated on Si. (a) After first oxidation and HF etching in air; (b) after second oxidation and HF etching in air; (c-d) SEM images of pyramid structures fabricated on Si. (c) Before first oxidation, (d) after third oxidation and HF etching in air.