Cleaning silicon and gold-coated substrates for SPM measurements

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Short abstract

This recipe describes the cleaning procedure of solid silicon based and gold-coated substrates for thin film deposition and SPM measurement in ambient conditions. The same procedure may be applied to quartz glass. Care should be taken to limit the duration of exposure to piranha solution in order to avoid extensive etching of the surface.

Step-by-step description of procedures

Cleaning Si-based surfaces

1. If the substrates have been exposed to any kind of organic contamination, clean them with the detergent that doesn't leave residue (2% HELLMANEX solution or similar)*.
2. Thoroughly rinse with distilled water.
3. Dip the substrates in hot (50-60 ^{o}C) acetone and keep them in ultrasound bath for 15 minutes.
4. Rinse well with DI water.
5. Dip the substrates in absolute ethanol and keep them in ultrasound bath for another 15 minutes.
6. Rinse well with copious amount of DI water.
7. Dry with clean stream of N_2 (necessary if performing measurements on dry samples or in vacuum).

Cleaning Au-plated surfaces

1. If the gold layer is thick (> 50 nm), take a small amount of freshly prepared piranha solution (30 ml of the 1:3-4 mixture of 30% H_2O_2 and 100% H_2SO_4) and keep the substrate in for a couple of minutes. The good sign of the reaction going on is the total coverage of the surface with small bubbles.
2. Rinse the substrates with absolute ethanol.
3. Dry with clean nitrogen (drying in the oven is **not** recommended).
4. For thinner gold layers it is safer to use ozone cleaner* instead of piranha solution.

(* for very light organic contamination a short (5-15 min) UV ozone plasma cleaning can be applied on bare Si wafers or gold-plated substrates after they have been cleaned in acetone or alcohol and rinsed with DI water.)

Special comments

Piranha solution is very effective in removing the top layer on surfaces due to its reactivity. Therefore, etching of the surface resulting from this procedure may introduce undesired roughness, which could, on the other hand, interfere with the formation of very thin well ordered biopolymers. However, for thicker samples, like multilayers or cells, this is usually not a problem, so the only special care should be taken that the substrates are well rinsed with DI water after they are treated with piranha solution.

Ozone will form a hydrophilic layer on silicon and gold alike, but the one on gold is only a few nm thick and degrades in the matter of minutes. Therefore, gold substrates should be used for hydrophilic molecules deposition (DNA, for example) immediately after cleaning.

Materials/chemicals/devices required

- Conductive silicone wafers (p- or n-doped).
- Annealed gold-plated silicon wafers.
- Absolute ethanol, > 99%
- Acetone, >99%
- HELLMANEX or similar solution
- Distilled and DI water (R>18.2 MOhm)
- 30% hydrogen peroxide (H_2O_2)
- Concentrated sulphuric acid (H_2SO_4)
- Ultrasound bath with the heater.
- UV lamp and the well ventilated area with the opaque cover or a commercial ozone cleaner.
- Source of clean and dry nitrogen gas

References

[1] http://www.hellma-analytics.com/text/200/en/cleaning-and-dilution.html

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[3] Ryugo Tero, Hidekazu Watanabe and Tsuneo Urisu, *Supported phospholipid bilayer formation on hydrophilicity-controlled silicon dioxide surfaces*, *Phys. Chem. Chem. Phys.* **8** (2006) 3885–3894.

[4] Clemens Barth , Adam S. Foster , Claude R. Henry , and Alexander L. Shluger, *Recent Trends in Surface Characterization and Chemistry with High-Resolution Scanning Force Methods*, *Adv. Mater.* **23** (2011) 477–501.