Production of adhesive-free borosilicate glass colloidal probes

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Short abstract

This recipe describes how to produce monolithic adhesive-free borosilicate glass colloidal probes. Capillary adhesion between sphere and cantilever is exploited to attach temporarily a micro-sphere to a tipless cantilever; curing the sphere-loaded cantilever in an oven at 780-820°C ensures a firm covalent attachment of the silica sphere and the production of a monolithic probe that can be aggressively cleaned before and after use. See Ref. [1].

Step-by-step description of procedures

1. Prepare a diluted solution of microspheres in water. To this purpose:
   a. in a 100-200 ml vial, disperse ~3 mg of dry sphere powder in a droplet (~100-500 µl) of alcohol;
   b. add ~100 ml of de-ionized or (better) milliQ or HPLC grade water;
   c. sonicate or vortex the solution for a few minutes.
2. Clean an optical microscope glass slide by standard procedures (wash in ethanol or acetone – sonicate if possible - then rinse with DI water; repeat twice).
3. Treat the glass slide surface to reduce its wettability. Depositing a thin (50- 200 nm) film of gold by evaporation or sputtering is very effective, but other strategies can be adopted.
4. Spot a droplet of ~10-20 µl of the diluted microsphere solution onto the treated glass slide and let it dry. This procedure typically leads to well dispersed microspheres on the glass slide surface.
5. Fit and secure the slide and the tipless cantilever in the XYZ micro-translation stage, so that the free end of the cantilever can be positioned very close to a single isolated sphere and brought in contact with it in order to pick the sphere up (Fig. 1b; we mount the slide on the XY stage of our bio-AFM and the cantilever on the AFM head, and exploit the optical inverted microscope to guide the fishing operations).
6. Upon withdrawal of the cantilever, the sphere remains attached to the cantilever and is detached from the substrate that has a lower adhesion (Fig. 1c); transfer the chip into the oven and keep it for 2 h at 780-820°C (use a quick temperature ramp and put the chip into the oven already at relatively high temperature).

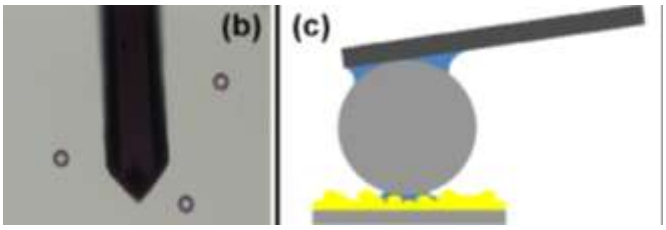

Figure 1. (b) Typical optical microscope view of spheres and cantilever. Microspheres are dispersed on the slide at low density. (c) The cantilever/sphere/substrate contact geometry during the picking-up process; meniscii of different strength form between the cantilever and the sphere (strong adhesion), and between the sphere and the treated glass slide (weak adhesion).

7. Let the oven cool down, extract the probe(s). Probes are typically clean when they emerge from the oven. Probes support aggressive cleaning (piranha, acqua regia,…).

Special comments

1. This recipe allows fabricating BOROSILICATE GLASS probes monolithically mounted on SILICON cantilevers; application of the protocol to other probe/cantilever combinations is still to be experimented.
2. It is possible to functionalize the glass slide surface, alternatively to the deposition of a thin Au film, with some suitable molecule or chemical group that makes it less adhesive for microspheres (i.e. that makes it more hydrophobic, increasing the contact angle). Suggestions are welcome. Exposure of the slide to APTES vapors could be worth to be tried.
3. The monolithic character of these probes allows aggressive cleaning processes to be employed in order to remove contaminant from probe surface. Probes can therefore be used many times.
4. The characterization procedures of probe radius and roughness are described in details in Ref. [1].

Materials/chemicals/devices required

1. Borosilicate glass microspheres with diameters 1-20 um (spheres of smaller size are very difficult to handle; larger spheres can be tried out) in form of dry powders (as provided by, among others, SPI Supplies, USA).
2. Standard glass slides for optical microscopy.
3. Tipless SILICON cantilevers with desired force constant (no metal coating must be present!).
4. Ethanol and DI (or better HPLC- or milliQ-grade) water for microsphere suspension.

You will also need (non-standard devices):

1. An XYZ micro-translation stage with optical access (bio-oriented AFMs provide this).
2. An oven capable of heating up to 800-1000°C.
3. A sputter coater or an evaporator to deposit a thin Au film on the glass slide to render its surface less adhesive. We use a Pabisch Top Autocoater SC-20 apparatus for sample metallization. But look at Special Comment #2.
4. An ultrasonic bath.

References

[1] Adhesive-free colloidal probes for nanoscale force measurements: production and characterization. M. Indrieri, A. Podestà, G. Bongiorno, D. Marchesi, P. Milani, Review of Scientific Instruments 82, 023708-10 (2011). doi:10.1063/1.3553499