Supporting Information of

Oxide Nanocrystals based Nanocomposite for Fabrication of photoplastic AFM Probes

Chiara Ingrosso^{a,b,§,}, Cristina Martin-Olmos^{c,d,} [†], Andreu Llobera^c, Claudia Innocenti^e, Claudio Sangregorio^{e,f}, Marinella Striccoli^a, Angela Agostiano^{a,b}, Anja Voigt^g, Gabi Gruetzner^g, Jürgen Brugger^d, Francesc Perez-Murano^c, and Maria Lucia Curri^a

Figure S1 shows a TEM image of a spin-coated film made of the N1 nanocomposite. The image shows the NC dispersion in the nanocomposite film along with the absence of relevant aggregates.



Fig. S1. TEM micrograph of spin-coated film made of N1.

Finger-like microstructures (sizes and pitches of such microstructures ranged from 1 up to 50 microns) made of the bare epoxy photoresist and N1 and N2 nanocomposites were fabricated by the following procedure.

First of all, bare silicon wafers were dehydrated for 30 min at 150 °C. The samples was spin-coated for 30 seconds at 1000 rpm for preparing 7.5 µm thick films and at 2000 rpm for 5 µm thick films, using 400 rpm sec⁻¹ ramping speeds. Right after the deposition, a soft bake was performed on a hotplate. The samples were slowly (2 °C min⁻¹) ramped from 65 °C to 95 °C and after 15 min at 95 °C they were cooled down to environmental temperature (20 °C) with a speed of approximately 4 °C min⁻¹. The samples were exposed using 400 nm light in a Karl-Süss MA6. The exposure dose was varied in order to find the highest resolution. The doses applied ranged from 32 to 144 mJ cm⁻² in steps of 4 mJ cm⁻². Then, a post exposure bake was performed on a hotplate heating the samples slowly (2 °C min⁻¹) from 65 °C to 95 °C and, after 20 min at 95 °C, they were cooled down to environmental temperature (20 °C) with a speed of approximately 4 °C min⁻¹. The samples were developed by soaking them in Propylene glycol monomethyl ether acetate (Aldrich \geq 99.5 %, DOWANOL PMA) upside-down, for up to two hours in order to fully remove the non exposed resist. Afterwards, they were rinsed with isopropyl alcohol (Sigma-Aldrich, \geq 99.5 %). The last step was a hard bake process in which the samples were slowly (2 °C min⁻¹) ramped from 65

°C to up to 120 °C and, after two hours, they were cooled down to room temperature (4 °C min⁻¹). This hard bake process was performed in a N_2 rich environment.

The fabricated finger-like structures were imaged by SEM (Fig. S2). In order to avoid charging effects, the samples were coated with 30 nm of gold using an e-beam evaporator working at room temperature and 2×10^{-6} mbar. Fig. S2 shows typical SEM images of the fabricated finger-like structures made of the bare epoxy based photoresist and the N1 and N2 samples.



Fig. S2. 30° tilted SEM micrographs of finger-like structures formed of bare epoxy photoresist (a) and N1 (b) and N2 (c) nanocomposites.

Fig. S3 reports the fabrication process sequence of AFM probes. A p-type silicon wafer (100 mm in diameter) was used during the process as a support for the multiple epoxy based resist layers. First, a layer of 400 nm of silicon dioxide was thermally grown using a wet oxidation at 1100 °C. The silicon oxide layer was patterned using a dry Reactive Ion Etching with CF₄, and used as a mask to etch the silicon by an anisotropic wet etching with Tetra-Methyl-Ammonium Hydroxide (aqueous solution at 25vol, Aldrich), defining an inverted pyramid as mould for the tip (Fig. S3(i)). The initial silicon oxide was removed and another 400 nm thick layer of silicon oxide is grown (using a wet oxidation at 950 °C) in order to sharpen the inverted pyramid and to be used as a sacrificial layer (Fig. S3(ii)). A layer of N1 with a thickness of 7.5 µm was spin-coated on the wafer, filling the inverted pyramids (Fig. S3(iii)). After the soft bake (process described in the previous section), it was exposed in order to define the cantilevers shapes using the optimum dosage found in the exposure tests described before: 96 mJ cm⁻² (Fig. S3(iv)). After the post-exposure bake (process described in the previous section), a second layer of anisole based negative tone epoxy resist (mr-L 5025 XP, Microresist Technology) with a thickness of 250 µm was deposited and soft-baked (Fig. S3(v)). It was exposed in order to define the body of the AFM probe chips using a dose of 1456 mJ cm⁻², in 8 steps of 182 mJ cm⁻² to avoid overheating the resist (Fig. S3(vi)). After post-bake, both resist layers were developed using PGMEA for more than 3 hours (Fig. S3(vii)). Finally, the silicon oxide sacrificial layer was removed in a buffered HF solution (SiOetch, 6% HF + 16% NH₄F, Aldrich) and the AFM probes were released (Fig. S3(viii)).



Fig. S3. Scheme of the fabrication process sequence of AFM probes.

Fig S4 compares the profiles of some gold nanoparticles deposited on a silicon substrate after being imaged with AFM using a commercial silicon probe (Fig. S4(a)) and using a N1 AFM probe (Fig S4(b)). From the information shown in the profiles, it is clear that the effect of tip convolution is much higher in the case of the commercial Si tip than for the fabricated one: assuming that the nanoparticles are nearly

perfect spheres, the measured height in the profiles provides an accurate value for the diameter of each nanoparticle. Therefore, the comparison of the height with the width of each peak in the profiles evidences the effect of tip convolution to be larger for the commercial Si AFM probes.



Fig. S4. Topography AFM images and related profiles of gold particles randomly deposited on a silicon substrate imaged by using a (a) commercial Si probe and (b) N1 AFM probe.