Improved Nanoscale Electrical Characterization with Graphene-Coated Atomic Force Microscope Tips

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Abstract

Conductive atomic force microscope (CAFM) has been shown to be a powerful technique to characterize the electrical properties of both conductive and thin insulating materials at areas as small as 10-100nm² [1–3]. One of the main challenges associated with this technique is the poor reliability of the conductive tips. Traditionally, metal-varnished silicon tips are commonly utilized in these kinds of applications [4]. However, due to the low stability of the metallic varnish, these tips can wear out very fast when measuring high currents and/or because of intense tip–sample frictions. In this work, we will show how the intrinsic properties of a commercially available CAFM tip can be modified if it is coated with a sheet of CVD-grown graphene. A graphene single layer (GSL) sheet grown on copper foils by CVD [5] was transferred onto different PtIr varnished CAFM tips. This prototype tip has been used to perform nanoscale electrical characterization of both conductive and thin insulating materials.

The performance of graphene coated tips has been first assessed from the analysis of $HfO_2/SiO_2/Si$ stacks. I-V curves at different positions have been measured with as-received (Figure 1a) and graphene-coated (Figure 1b) tips. As it can be observed, the *I*-V curves measured with the as-received tips show a decrease of the measured current with the number of recorded I-V curves. Since this sample is very homogeneous [6], Figure 1a shows that the as-received tip loses its conductivity by wearing out due to very high driving current densities ($J \approx 10^8$ A/cm² when it reaches the current compliance) [7]. On the other hand, the graphene-coated tip supports several measurements (Figure 1b), which entirely reached the current compliance level approximately at the same onset voltage, without appreciable wearing.

In a second measurement series, the resistance of graphene-coated tips to the high frictions is analyzed from current maps. Figure 2 shows AFM topography and current maps obtained with an asreceived PtIr-coated tip (a-d) and a graphene-coated tip (e-h) on a as-grown GSL on Cu stacks. The current maps are recorded by biasing the samples at -0.1V (e. g., substrate injection of electrons) and keeping them under the high vacuum ambient conditions. From all the topographic images (a, b, e, f) the typical stepped copper surface (covered by GSL) can be observed [8]. On the other hand, the current maps (c, d, g, h) clearly overlap with the topographic images, showing current everywhere except at the step edges, probably due to the loss of contact between the tip and the sample at those locations. Comparing the images obtained with the two kinds of tips, one can easily see that as-received tips allow scanning an area of $6\mu m^2$ (6 scans of $1\mu m \times 1\mu m$) before the tip loses its conduction (using a contact force of 5nN). The tip wearing can be observed from the lowering of the current scale in Figure 2d. However, the maps collected using graphene-coated tips suggest that these tips preserve their intrinsic properties, since no loss of conductivity is observed even after scanning an area of 903µm² and even using contact forces up to 50nN.

Therefore, it is successfully demonstrated that conductive tips for the CAFM, fabricated by coating commercially available metal-varnished tips with a sheet of GSL following already established standard transfer process steps, show ultra-high performance. Graphene-coated tips are much more resistant to both high currents and frictions than commercially available metal-varnished CAFM tips, leading to much longer lifetimes and preventing false imaging due to tip–sample interaction. In total, seven different graphene-coated tips have been fabricated and characterized using the procedures here described, and successful results for all of them were observed. The novel devices can be interesting not only for reducing tip replacement costs, but also for those applications that require high stability and low tip–sample interaction.

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Figures



Figure 1. -V curves measured on the bare surface of an HfO₂/SiO₂ stack using a) a PtIr coated tip, and b) a graphene-coated tip. Each graph shows the 1st, 3rd, 5th, 10th, 15th, and 20th -V curve measured. All -V curves were collected at fresh different locations by applying a Ramped Voltage to the tip (sample substrate grounded).



Figure 2 . Topographic (a,b,e,f) and current (c,d,g,h) maps measured on the surface of GSL/Cu stacks recorded with as-received (a–d) and graphene-coated tips (e–h). All scans are 1 μ m x 1 μ m, and the images of each column have been collected simultaneously. The images clearly show that as-delivered tips experiment a substantial conductivity loss after 6 scans, while in contrast the graphene-coated tips keep measuring current even after have been used to scan an area 150 times larger.