A metallic microcantilever electric contact probe array incorporated in an atomic force microscope

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We present the realization and performance of a multiprobe microcontactor made of an array of metallic microcantilevers inserted in an atomic force microscope (AFM). This instrument permits simultaneous AFM imaging and electrical characterization of nanoscale devices. It is therefore well adapted for future generations of molecular devices. The probes are 2-μm-wide metallic cantilevers that are brought in contact with 3 μm×3 μm metallic pads of a nanocircuit using a nanopositioning table. The performance of the instrument, tested on mesoscopic metallic wires and carbon nanotubes, shows that the reproducibility of the electrical contact between the probes and the circuit is better than 99.2%. © 2000 American Institute of Physics.

I. INTRODUCTION

Planar electrical connections with a nanoscale device like a nanotube or a molecular gate actually require four levels of interconnections: (i) a planar nanojunction where the molecular device is adsorbed with both ends to the junction electrodes, (ii) mesoscopic metallic leads which are generally a direct continuation of the nanojunction electrodes, (iii) large metallic pads connecting these leads to facilitate the bonding of a 20-μm-diam metallic wire, and (iv) the bonding between the pads and metallic wires bonded usually by thermosoldering or conducting paste on the pads. Consequently, the number of interconnected nanojunctions per wafer is relatively small, generally less than one nanojunction per 0.01 mm² because of the large surface area required for bonding the pads. Furthermore, these pads are generally fabricated using a photolithography process which introduces difficulties in terms of cleanness and alignment of the pads compared to the e-beam fabricated mesoscopic leads. The large pads also result in an heterogeneous surface which puts constraints on the molecular deposition and the manipulation techniques used to position the molecular device on the nanojunction.

To reduce the number of interconnection levels, to increase the number of nanojunctions that can be tested per wafer, and to suppress the photolithography and the wire bonding steps, we have designed, realized and tested a metallic microcantilever array of electrical probes integrated with a commercial atomic force microscope (AFM) head. This technology enables the systematic electrical characterization of nanodevices mass fabricated on a single wafer without need for the standard free-wiring technique.

The principle of this multiple electrical contact probe is presented in Fig. 1. It is a generalization of the use of a single metallic tip cantilever for local electrical testing and of the standard multiprobe heads used in microelectronics. A dual imaging procedure is required for the coarse alignment of the multiprobe array on the metallic structures with an optical microscope and for imaging the nanojunction and the molecular device with the AFM during its electrical characterization.

In the first part, the extension of a commercial AFM to incorporate the nanojunction testing unit, under the AFM head, is described. Second, we describe the fabrication process of the metallic microcantilever array. Finally, the performance of our microprobes are presented for two and four point resistance measurements of mesoscopic wires and single wall carbon nanotubes.

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II. AFM MODIFICATION

Our multiprobe electrical contact array is inserted under the head of a Dimension 3000 AFM (Digital Instruments). Being a moving probe—stationary sample based scanning instrument, the new multifunctional sample holder can be used without perturbing the image acquisition. The AFM head is also equipped with an optical microscope for positioning of the tip apex on the surface. Furthermore, the geometry of the instrument is such that new tools are easily introduced laterally under the scanning head, like the microcantilever probe array proposed in Fig. 1.

First, the sample holder was modified to incorporate a low noise nanopositioning table NPS3330 (Queensgate Instruments). This “nanotable” was mounted on a D80MM rotation stage (Micro-Controle) to correct for possible angular orientation mismatches of the microcantilever array in the x and y directions relative to the sample surface. The rotation stage precision is 1/60° which is sufficient to correct for a possible misalignment. This new sample holder has four degrees of freedom, with software controlled x, y, and z positions. Its close-loop controller allows reliable displacements of 100 μm x 100 μm x 15 μm in the x, y, and z directions, respectively. Nanometer level reproducibility was demonstrated by the manipulation of a nanofiber of 1 nm in diameter with the AFM tip apex. Instead of using the x and y scans’ ability of the AFM head to perform this manipulation, we activate the x and y degrees of freedom of the nanotable and define the target position of the manipulation with the computer controlled program. The nanotable z degree of freedom was used to bring the tip apex in close contact with the nanofiber, the fiber was then successfully manipulated by moving the nanotable along the x and y axis. No deterioration of the AFM imaging performances was measured with this new sample holder. Notice that we have kept the resident step-by-step motorization stage of the original Dimension 3000 AFM for easy positioning of the nanotable under the AFM head (Fig. 1).

To support and position the microcantilever array under the AFM head, a lateral holder has been designed and fabricated. In addition to hold the silicon chip integrating the metallic microcantilever array [see Fig. 2(a)] it is also used to connect each of these cantilevers to the external measurement units (Keithley 617 electrometer and Keithley 192 multimeter). Part of the microcantilever electric wiring is integrated on the cantilever silicon chip and the other part on a standard printing board. The position of the holder is controlled by three M-UMR3.5 translation stages (Micro-Controle). Note that the holder is fixed on the translation stage with a small vertical tilt (a few degrees) to facilitate the contact of the cantilevers with the surface. The array is positioned within 5 μm of the microcircuits on the wafer [Fig. 2(a)], and the nanotable sample holder is then used to contact the microcantilevers with the microcircuits contact pads. The microcantilever array chip holder fits under the cantilever holder of the AFM head [Fig. 2(b)], enabling imaging of the nanojunction when the metallic cantilever array is in contact with the microcircuit.

An image of the modified AFM is shown in Fig. 2(a). Notice that, to install the nanotable under the AFM head, the AFM stage had to be raised by 3 cm. The influence of this modification was tested on the AFM resolution. With the Queensgate table turned off, no difference in resolution could be detected between the modified and nonmodified AFM (a noise of 0.3 Å was measured in both cases). With the nanotable on, we optimized the parameters of the feedback loop to minimize noise. In this configuration, the noise increases (0.6 Å) but with no noticeable influence on image quality. This configuration gives a slower response when moving the nanotable (42 ms for a 0.5 μm displacement to be compared to 2 ms for the fast response configuration) but
**III. FABRICATION OF THE METALLIC MICROCANTILEVER ARRAY**

The use of an atomic force microscope with a conducting cantilever is the most common way to perform electrical characterizations of nanostructures especially for measuring capacitance. These conducting cantilevers are usually fabricated by coating commercially available cantilevers with thin metal films.9 Nevertheless, this method suffers from wear of the metallic thin film that drastically impacts the quality of the electrical contact after repeated scans. A promising alternative is to use metallic cantilevers that can provide reproducible electrical contacts.7–10 The metallic cantilevers used in our experiment differ from those presented in these works by the fact that they are used in an array with reduced size and used solely for electrical characterization since imaging is performed independently with the AFM.

The metallic microcantilever array chips are fabricated in three steps, as presented schematically in Fig. 3(a). The process starts with a 4 in. double-side polished (100) p-type silicon wafer of 1–5 Ω·cm resistivity. The first step consists in thermal growth of a 1-μm-thick silicon dioxide layer at 950 °C. This layer is selectively etched in buffered HF on the front side and left on the back side to be used as a masking layer. A second oxidation at 1150 °C is then performed to obtain a thinner oxide (0.1 μm) with improved electrical properties. The purpose of this layer is twofold, it acts as an interfacial insulating layer between the substrate and the metallic cantilevers and as an etch stop layer during the final etching. The shape of the cantilevers, as well as the connecting wires and pads, are defined on the front side through a 2.8-μm-thick reversible photoresist pattern (AZ5214E). A 0.1-μm-thick Ti layer and a 1-μm-thick Au layer are then deposited by evaporation. Titanium was selected as a plating layer over several candidates considered, including chromium and nickel. Chromium was not chosen in order to avoid the formation of a highly stressed layer that can drastically affect the mechanical properties of the released structures. Nickel was also avoided even though it leads to a reduction of the residual stress within the layer since the adhesion of nickel on silicon dioxide is poor. Titanium is a good compromise to obtain a good adhesion layer between silicon dioxide and gold while ensuring the formation of a low stressed interfacial layer. A lift-off process which consists in selectively removing the Ti/Au film on the photoresist layer is then performed leaving the cantilevers with the connecting wires and pads.

Since slightly upwardly bent cantilevers are desirable to produce a prominent tip for electrical contact under the AFM, special attention has been paid to the control of the beam stress by tailoring the residual stress within the Ti/Au bilayer before releasing the cantilevers. This has been achieved by carefully choosing the thickness of the Ti layer and by adding an annealing step in the fabrication process. Annealing has been performed at 450 °C for 30 min under an N₂/H₂ environment.

On the opposite side of the substrate, rectangular areas are defined by selectively removing the 1-μm-thick silicon dioxide. During the final step, these exposed areas are etched in a 20% TMAH solution at 90 °C while the front side is mechanically protected against etching with a dedicated chuck made of Teflon. TMAH etching was preferred to KOH etching since the etch rate of silicon dioxide in TMAH is very low. At this temperature: The silicon etch rate is about 0.9 μm/min and the silicon dioxide etch rate is 0.02 μm/min. The 0.1-μm-thick SiO₂ layer can therefore be used as an etch stop layer while the 1-μm-thick SiO₂ layer serves as a masking layer. During the last step, the 0.1-μm-thick silicon dioxide layer is etched in buffered HF leaving arrays of free standing metallic microcantilevers as shown in Fig. 3(b).
The cantilevers are 1.1 μm thick, 2 μm wide, and 60 μm long with a spacing of 4 μm. The intercantilever separation is compatible with the micron scale pads fabricated by e-beam lithography together with the connection pads and the nanojunctions (see below). A complete microcantilever-array chip with cantilevers, integrated wiring networks, and contacting pads is presented in Fig. 3.

IV. ELECTRICAL CONTACT PERFORMANCES

To study the performances of our electric microprobe array, a series of mesoscopic metallic wires 0.2 μm in width were fabricated. Now a classic in transport physics, such structures are very useful to test the ability of each microcantilever in the array by contacting the wires in a two or four probes geometry. For these two types of measurements, the micropads on the wafer are only 3 μm×3 μm in size and are e-beam fabricated at the same time as the mesoscopic wires [Fig. 4(a)].

The outcome of a 4-point resistance measurement is shown in Fig. 4(b). AFM images can be taken in parallel with the recording of the current–voltage (I–V) characteristics [Fig. 4(b)]. The four metallic microcantilevers are first prealigned on the four microscopic pads using the four degrees of freedom coarse displacements of the microcantilever holder under the AFM head optical microscope. Thereafter, the nanotable is used to fine align the lever and establish the electrical contact using the z axis displacement to gently rise the sample wafer until electrical contact is achieved. Notice that the micropads metal thickness is only 10 nm. However, no net change of the corrugation of the pads surface could be recorded by AFM after contact with the microcantilevers. The I–V measurements are performed as in any 4-point measurement, using a current source through levers A–D and a voltmeter contacted on levers B–C [Fig. 4(a)]. The room temperature conductivity of our mesoscopic wire is found to be 0.76 μΩ m. The probe–nanocircuit contact resistances of about 800 Ω were deduced from the measurement of the 2-point and 4-point resistances on the BC nanowire segment between electrodes B and C [Fig. 4(b)].

2-point resistance measurement structures were used to test the stability and reproducibility of the microprobe technique described above. To evaluate the stability of the electric contact, we have first recorded the fluctuation during 12 h of the 2-point resistance measured on a single mesoscopic wire [Fig. 5(a)]. The maximum residual resistance variation is only 0.2% after an initial stabilization time. The reproducivities of freedom coarse displacements of the microcantilever holder under the AFM head optical microscope. Thereafter, the nanotable is used to fine align the lever and establish the electrical contact using the z axis displacement to gently rise the sample wafer until electrical contact is achieved. Notice that the micropads metal thickness is only 10 nm. However, no net change of the corrugation of the pads surface could be recorded by AFM after contact with the microcantilevers. The I–V measurements are performed as in any 4-point measurement, using a current source through levers A–D and a voltmeter contacted on levers B–C [Fig. 4(a)]. The room temperature conductivity of our mesoscopic wire is found to be 0.76 μΩ m. The probe–nanocircuit contact resistances of about 800 Ω were deduced from the measurement of the 2-point and 4-point resistances on the BC nanowire segment between electrodes B and C [Fig. 4(b)].

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ibility of the contact was evaluated by inputting a regular vertical $z$ shift of the nanotable in order to establish and then suppress the contact between the levers and the micropads [Fig. 5(b)]. This experiment also gives an indication of the short time fluctuations, i.e., the time constant of the equilibration of the resistance. We find that after a stabilization time of about 20 s, an equilibrium value is reached. The variation of this value from one contact to the next is less than 0.8%. This shows that the electrical probe–circuit contact is reproducible. Notice that the vertical procedure to suppress the contact is difficult to perform due to adhesive forces which constrain the microlever on the surface. Therefore, a $z$ shift as high as 7 $\mu$m was necessary to reach the noncontact regime. We have also noticed that a current intensity larger than 1 $\mu$A is more effective in providing a fast contact stabilization than current intensity in the nanoampere region.

To evaluate the ability of our instrument to rapidly test different circuits on the same wafer, we selected two nearest-neighbor microcantilevers and, without changing the selected microcantilevers, we successively recorded the $I–V$ characteristics of six mesoscopic wires, 0.4 $\mu$m in width selected each in a family of eight identical structures (Fig. 6). The 3% variation of the resistances measured can be attributed to small fluctuations from wire to wire in their granular structures. Measuring such a series takes less than 10 min with a manual procedure. In this case, the positioning of the microcantilever array is fine tuned by using the computer controlled displacements of the nanotable sample holder, following the displacements with the optical microscope in the AFM head. A fully automated version of this procedure is easy to implement and will reduce the measurement time.

A limitation of our microcontacting procedure is the leaking current observed at large voltage, due to surface states on the surface of the silicon chip where the microlevers are fabricated. Currents as high as 10 pA for a 1 V bias are recorded between the microlevers when the array is not in contact with a metallic structure. The setup is therefore adapted for the measurement of currents in the nanoampere regime or higher. For lower currents, a compensation method can be implemented taking into account the static leaking current. Bias voltage up to a few volts can be applied between the microlever without perturbing the AFM imaging. However, higher voltages may create electrostatic effects that perturb the imaging of nonconducting samples as we observed in a study of the breakdown limit of nanojunctions.

Finally, to prove the advantage of our microprobes array in nanoelectronics, we have fabricated molecular nano-Wheatstone bridges using two carbon nanotubes on four electrodes geometry. First, 1000 nanojunctions with four electrodes were fabricated on a single wafer. Then, nanotubes were combed in parallel on these junctions to form the...
four resistances of a nano-Wheatstone bridge. The one presented on Fig. 7(a) is made of two nanotubes of different diameters (7 nm and 1.4 nm), the larger diameter one being a bundle of 1.4 nm single wall nanotubes. This nanocircuit realizes a detuned Wheatstone bridge. Due to our ability to contact simultaneously at least four micropads, measurements can be performed on any of the nano-Wheatstone bridges found on the wafer using the AFM images. Furthermore, an advantage of our AFM microprobe array is that a given bridge can be imaged permanently during the electrical measurements. As reported in Ref. 1, the electrical resistance of a carbon nanotube can be changed by deforming the tube by AFM manipulations. Deformation of one branch of the bridge thus opens the possibility to fulfill the bridge equilibrium conditions. While detuned, the potential difference $V$ in the center of the bridge as a function of the driving current $I$ is presented in Fig. 7(b) together with the corresponding circuit diagram.

In conclusion, we have assembled an AFM based electric microtester which is very well adapted for molecular devices. It combines the full nanoscale image capability of an AFM with an optical coarse approach, both well adapted to the nanotable which supports the wafer and to the metallic multiprobe array which fits under the AFM tip holder. It permits simultaneous imaging and electrical characterization of the large number of nanoscale devices that can be fabri-

FIG. 6. Optical micrograph of a wafer with rows of mesoscopic wires 0.4 μm wide. Each of the 12 horizontal segments appearing in the image consists of eight wires with their 3 μm×3 μm contact pads as shown in the inset. The $I(V)$ curves correspond to characteristics measured on one wire in every row. The values of the resistances are 11, 10.7, 10.8, 10.7, 10.8, and 11 kΩ.
cated per wafer. Further improvement of the system described in this article includes the fabrication of piezoelectric cantilevers arrays. Such cantilevers require no external detection sensor and will be used as individual active electrodes for intermittent electrical contact.

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FIG. 7. (a) AFM image of a nano-Wheatstone bridge and schematic representation of the electrical circuit. The four electrodes ABCD are bridged by two nanotubes with diameters 7 nm and 1.4 nm for the upper and lower tubes, respectively. The current $I$ is applied between electrodes B and D and the tension $V$ is measured between electrodes A and C. (b) $I(V)$ characteristics measured on the nano-Wheatstone bridge following the electrical diagram schematized in the inset. The values of the resistances determined by 2-point measurements are $R_1 = 670 \, \text{k}\Omega$, $R_2 = 16 \, \text{M}\Omega$, $R_3 = 20 \, \text{M}\Omega$, and $R_4 = 1.1 \, \text{M}\Omega$. This gives a detuned bridge. Note that the $I(V)$ characteristics gives a resistance of 280 k$\Omega$ comparable with the value of 340 k$\Omega$ which is expected for a circuit with the 2-point measured resistances reported above.