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Large-area in plane molecular junctions by electrografting in 10 nm metallic nanotrenches

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ABSTRACT
A key issue to push molecular devices toward a new range of applications is the ability to master large scale integration while preserving the device’s functionality. Furthermore, providing extra tunability of the device by external parameters, such as gating in a transistor-like configuration, is highly suited for molecular electronics. Large area molecular junctions in crossbar geometry have demonstrated high yields and compatible fabrication with Complementary Metal Oxide Semiconductor (CMOS) technology. However, such a device’s geometry favors diffusion of metallic atoms in the molecular layer and gives a very limited access to perform electrical or optical gating on molecules. In this work, we propose a new molecular junction architecture going behind these limits. We report a robust approach for the fabrication of molecular junctions based on the electrografting of a nanometer-thick molecular layer in high aspect ratio metallic nanotrenches. Nanotrenches are obtained by edge-mediated shadow deposition, resulting in laterally aligned electrodes with a 10.3 nm ± 3.3 nm average spacing along a 20 μm length. An in-solution electroreduction of diazonium salts is subsequently performed to fill the nanotrenches by a thin oligomeric layer of anthraquinone molecules. Electronic transport measurements performed at room temperature reveal the ability to produce stable molecular devices. Such a new junction’s engineering offers the key advantages of high fabrication yield, great amenability for compact assembly, and reduced leakage current. The proposed architecture opens interesting perspectives to investigate fundamental and applied questions in molecular electronics, in which coupling of the molecules with external stimuli is required.

I. INTRODUCTION
Over the past 40 years, charge transport in molecular systems has tremendously caught the interest of the scientific community. Single-molecule and self-assembled monolayer (SAM) based junctions have opened the way to investigate fundamental questions on charge transport through molecules, such as transport mechanisms, coherent effects, and electron–phonon interaction. Device’s functionalities related to the molecular structure have also been recently demonstrated. In the quest of applications, large area molecular junctions, consisting of a molecular layer embedded between two metallic electrodes in a crossbar geometry, have become of major interest in the molecular electronic community because of their scalable fabrication processes, improved stability, and high yield. The introduction of the method of electroreduction of diazonium salts to grow molecular thin films in molecular electronics has provided an important step forward the integration of large area molecular devices in compatible complementary metal–oxide–semiconductor (CMOS) electronic chips. This procedure has been proved to form robust and compact molecular layers covalently attached to carbon and gold surfaces, with thicknesses varying from a few nanometers up to ∼30 nm. However, the large area molecular junction approach usually implies the exposition of the molecular layer to standard chemical products linked to techniques used in micro- and nano-fabrication technologies. Thus, it strongly limits the nature of molecules and metals that can be
used due to their reactivity. Besides that, this junction configuration suffers from different intrinsic limitations. It is not suitable for electrostatic or optical gating and metallic filaments can easily penetrate within the molecular thin layer. In this context, research on designing device architectures offering the advantages of a robust and reproducible fabrication process, massive production, and opening toward an easy coupling to external tools is of high technological and fundamental interest.

In this work, we present a new molecular junction fabrication method. The method combines the electrode mediated shadow edge deposition process producing laterally aligned electrodes with high aspect ratio and the electrografting procedure realizing robust covalently bonded molecular layers. We have systematically realized 10 nm spaced nanotrenches over 20 μm distances and proved successfully the electrografting of an anthraquinone (AQ) molecular layer inside the nanotrenches. The obtained molecular junctions reveal symmetric and stable electronic transport characteristics, with an 80% yield. Laterally aligned metallic electrodes spaced by nanometric distances over micrometric lengths represent a viable solution for the fabrication of molecular junctions. This particular junction geometry offers the advantages of large scale integration, reduced leakage currents, and easier access to the molecular layer with external tools. Arrays of high aspect ratio nanotrenches have been already fabricated by different approaches, including adhesion lithography,24 high resolved sketch and peel lithography,25 nanomasking,26,27 self-aligned lithography,28 and electrode mediated shadow edge deposition.28–31 They constitute a platform for a large panel of devices, ranging from electronic to photonic purposes, but they have not found yet direct applications for molecular electronics.

II. METHODS

Devices are fabricated on doped Si substrates covered by a 500 nm thick SiO$_2$ oxide layer. Nanotrenches are realized by means of the shadow edge evaporation method. The first junction electrode is used as a shadow mask for the deposition of the second lateral electrode.29–31 The successive steps of the fabrication process are depicted in Fig. 1. The mask for the first electrode is patterned by e-beam lithography and it has the shape of a 20 μm wide, 15 mm long wire connected to a large contact pad (4 mm × 3 mm) necessary for the electrografting procedure [Fig. 1(a)]. E-beam lithography has been used to realize well-defined edges of the resist mask and as a consequence a better definition of the electrode edges after lift-off. This is a crucial point as the first electrode edge is used as a shadow mask for the laterally aligned second electrode evaporation. In principle, this lithography step can also be performed by using a well-cared optical lithography process. The first electrode is then realized by e-beam evaporation (P ~ 10$^{-8}$ mbar) of a Ti/Au bilayer (3 nm/20 nm), whose total thickness must be lower than the first electrode one. When depositing the second electrode, the layer growth produces a disconnected zone, whose width reduces as the thickness increases, as shown in Fig. 1(d). Note that in this simplified picture we are neglecting atom diffusion on the substrate. The typical evaporation rates are of the order of 0.05–0.1 nm/s to minimize heat and atomic diffusion. The width of the nanospaced electrodes is evaluated using scanning electron microscopy (SEM), by imaging different areas located along the trench (Fig. S1 in the supplementary material). The described procedure allows the simultaneous fabrication of 24 nanotrenches over a SiO$_2$/Si chip of 5 mm × 15 mm area. The total number can be clearly increased for a massive production.

Nanotrenches are electrically characterized by measuring their current–voltage characteristics I(V) at room temperature. We use a two-probe measurement setup, in which a DC voltage is applied between the two electrodes and a low noise current amplifier is used to measure the current passing through the device. In order to be suitable for molecular grafting, nanotrenches have to be completely disconnected to show an open circuit behavior in the explored voltage range, −2 V < V < 2 V. After these first characterisation steps, we realize the electrochemical grafting by the method of electroreduction of diazonium salts, using the anthraquinone (AQ) molecule as a test system. The electroreduction of 9,10-dioxyo-1-anthracenediazonium salt in C$_2$H$_5$N−NBu$_4$BF$_4$ solution is controlled by cyclic voltammetry (Figs. S2 and S3 in the supplementary material). Note that only the first evaporated electrode is connected as the working electrode in the voltammetry cell, and, as a
consequence, the molecular grafting occurs mainly at its surface. Contact with the second electrode occurs likely by physiosorption. Redox grafting parameters, such as scan rate (1 V/s), switching potentials (−1.5 V < V < 0.5 V), and number of cycles (N = 4), are optimized to obtain a molecular layer with a thickness at least corresponding to the nanotrench width. This method ensures the formation of a robust and thin layer of AQ molecules bonded covalently to the electrode. The grafted AQ molecular layer and its total thickness are characterized by atomic force microscopy (AFM). After the grafting process, a second electronic transport characterization of the obtained molecular junctions is performed at room temperature. The electric responses of the samples before and after grafting are crucial steps to validate the correct realization of the molecular junctions.

III. RESULTS AND DISCUSSION

The optimization of the fabrication process results in an average nanotrench width of w′ ≈ 10 nm for θ = 40°. Figure 1(e) shows the top nanotrench width w′ averaged over ∼200 measurements for different deposition angles (points). For θ < 40°, electrical tests reveal that the number of short-circuited electrodes exceeds statistically the number of disconnected ones. For θ > 40°, too wide (>20 nm) nanotrenches are realized implying, if successfully grafted, to measure low current intensity levels. The measured average nanotrench width is always lower than what expected by simple geometrical considerations. Neglecting atom diffusion, the bottom nanotrench width w is given by w = h t gθ, where h is the first electrode total thickness, and the top nanotrench width w′ is given by w′ = h t gθ − h′ sin θ, where h′ is the second electrode total thickness. For an evaporation angle of 40°, w is expected to be of the order of 40 nm, while w′ is of the order of 30 nm. Metal atom diffusion can only partially explain such a discrepancy since the diffusion coefficient of Au atoms on SiO2 is quite small at room temperature (10−26 cm²/s). Most probably, a systematic error in the angle displayed by the sample holder motor controller in the evaporation chamber can explain this unexpected result. The continuous line in Fig. 1(e) is the expected theoretical trend of w′(θ) corrected by a systematic error. The experimental data are in good agreement with the expected behavior based on geometrical consideration. Figure 2(a) shows a SEM image of a fabricated nanotrench. The nanotrench is clearly visible as a discontinuity of the second evaporated electrode and it appears regular along the width. A statistical analysis of the nanotrench width performed over 800 measurements on 75 different devices with identical deposition parameters is shown in Fig. 2(b). The histogram is fitted well with a Gaussian distribution with an average width of 10.3 nm ± 3.3 nm. The statistical analysis performed over each single nanotrench reveals the same result, confirming the reproducibility of the fabrication process at the single device level (Fig. S4 in the supplementary material).

Current–voltage I(V) characteristics have been measured at room temperature on 120 nanotrenches in a typical maximal voltage range of −2 V < V < 2 V. Out of 120 I(V) curves measured, 75% shows open circuit behavior, 14% results in linear ohmic behavior (short circuit), while the remaining shows an unstable trend. In the latter case, continuous sweeping of the applied voltage through the nano-spaced electrodes from negative to positive values and vice versa results in a gradual change toward an open circuit (6%) or a short circuit (5%). Clean nanotrenches with open circuit I(V) characteristics are selected for the electrochemical grafting by the method of electroreduction of diazonium salts, using the AQ molecule as a test system. The total thickness and the average roughness of the grafted molecular layer are evaluated by atomic force microscopy (AFM). We found for 4 cycles a molecular layer thickness equal to 20 nm ± 5 nm (Fig. S5 in the supplementary material) and an average molecular surface roughness of 10 nm ± 2 nm (Fig. S6 in the supplementary material). We have tested that for a lower number of cycles no electrical connection is obtained between the lateral contacts (Fig. S7 in the supplementary material).

Figure 3(a) shows an artistic view of the molecular junction architecture. A typical SEM image of the electrografted nanotrench based molecular junction is shown in Fig. 3(b). Molecular bridges connecting the two lateral aligned electrodes are clearly visible, distributed non-uniformly along the nanotrenches. Figure 3(c) shows an example of the I(V) characteristics of the same nanotrench before
(black) and after (red) grafting in the voltage range of $-0.6 \, V < V < 0.6 \, V$. After grafting, a clear nonlinear behavior of the $I(V)$ characteristics emerges with a low bias junction resistance higher than 1 GΩ. This is a typical signature of electronic transport occurring through the molecular layer.$^{15,22,23}$ The inset of Fig. 3(c) shows the $I(V)$ curve of the bare nanotrench in a higher voltage range ($-2 \, V < V < 2 \, V$). The current level is of the order of a few pA, which corresponds to the noise level of the measurement setup, confirming the absence of electrical connection between the electrodes. Electrical tests of the grafted nanotrenches show that 80% of the nanotrenches having initially an open circuit behavior shows signature of molecular transport after grafting. Note that as electrografting occurs in an electrochemical cell, the outcome of the procedure depends on the uniformity of the wetting of the electrode in the electrochemical solution. Capillary effects could hinder a homogeneous penetration of the solution inside the nanotrenches, preventing a uniform molecular layer grafting in the nanotrench. We cannot be sure if the molecular layer is formed between the edges of the inner electrodes or it is mostly bridging the upper part of the electrode surfaces. As a test, we have realized 50 nanotrenches, where the first electrode has been capped by a thin (a few nanometers) Al₂O₃ layer, in order to prevent electrografting on the top electrode surface. The Al₂O₃ layer makes the electrode surface inert to electron transfer and it is not reduced by the electrochemical process. In this case, 82% of the fabricated nanotrenches shows electrical open circuit behavior and only 29% of the grafted nanotrenches shows molecular signatures in the electronic transport (Fig. S8 in the supplementary material). Such a decreased yield with respect to the previous case indicates that the molecular junction is likely to be realized by molecular chains connecting the topmost surfaces of the two lateral metallic electrodes.

The good stability of the junction charge transport properties is investigated by repeated measurements over the same device. Figure 4(a) shows $10 \, I(V)$ curves measured continuously on the same molecular junction at room temperature, showing a narrow dispersion of the data. Figure 4(b) displays a set of $14 \, I(V)$ curves in a semi-log scale at room temperature measured over different nanotrench based molecular junctions, each resulting from averaging over 30 repeated measurements. A good reproducibility of the electronic transport characteristics can be observed, with most of the $I(V)$ curves located in the $0.1–10 \, \mu A$ current range and showing a symmetric behavior. All these observations validate the efficiency of the grafting procedure. The observed behavior is qualitatively similar to what has been already reported in AQ based molecular junctions in a crossbar geometry.$^{26,37}$ However, by evaluating the current density, we find out values bigger than what typically observed in that case for a similar layer thickness.$^{26,37}$ By considering the junction area as the one given by the lateral surfaces of the parallel electrodes ($\sim0.4 \, \mu m^2$), $J$ can be estimated to be $10^4$ to $10^5 \, A/cm^2$ at 0.5 V, while being typically equal to 0.1–1 A/cm² in the crossbar geometry case. In a simplified picture for both junction geometries, a linear potential profile can be assumed in the junction. However, the effective potential profile depends on many factors, such as internal or interfacial dipoles, coupling between the contacts and the molecular layer, and the presence of charged or neutral impurities. All these factors can be dependent on the particular junction configuration (lateral or vertical), impacting finally the charge transport mechanism. Additionally, the current density value found in the nanotrench based molecular junction is likely to be underestimated, since the effective junction area is probably lower than the estimated one. Controlling the effective surface of a large area molecular junction is generally a difficult task. In particular, in a crossbar geometry, surface roughness and metal atom penetration in the molecular layer prevent the formation of a uniform and well-controlled molecular layer thickness. It has been demonstrated in vertical SAM based junctions that the presence of defects induces a variability of the effective thickness of the molecular layer.$^{4}$ This results in an important uncertainty over the current density. The current is strongly dependent on the local molecular layer thickness, since in this particular case electronic transport occurs in the tunneling regime. In the nanotrench based geometry, the molecular layer thickness is mainly dictated by the distance between the electrodes. Edge roughness also generates a finite nanotrench width dispersion and higher current densities are likely to develop in the region with reduced width. For an average width of $\sim10 \, nm$ anyway, the electronic transport is beyond the tunneling limit and a less significant impact of the molecular layer thickness variability should occur. Current densities similar to what we observe have been reported for molecular nanowires$^{9,40}$ embedded in similar lateral geometries but of larger widths (from 8 to 50 times higher than in our case). Anyway, a direct comparison with our experimental observation is not actually possible, being charge transport through molecular structures highly dependent on the molecular nature, length, and on the method used to insert the structure in the nanotrenches. Iron oxide nanoparticles$^{31}$ and nanometer-sized metallic clusters$^{32}$ have been embedded in nanotrenches with similar dimensions. These lateral devices result instead in very different charge transport characteristics, showing linear $I(V)$ curves and single electron transistor signatures, respectively, with currents in the nA range. Molecular transistors based on 2–3 nm lateral nanotrenches functionalized by chemical self-assembly have also been realized,$^{33}$ showing nonlinear $I(V)$ curves with reduced current levels with respect to what we observe.

The proposed approach has clearly the key advantage of a highly reduced probability of realizing diffusion of metallic atoms within the molecular layer and of a reduced contamination of the grafted molecular layer, being the functionalization process the last step of the molecular junction fabrication. It is robust enough to be exploited in molecular electronics. Furthermore, a high electric field
can be attained in the nanotrench based molecular junction configuration (~50 MV/m for 0.5 V on a ~10 nm gap). At such a high field, injection barrier can be highly deformed, inducing a more efficient charge injection, by potentially improving the metal/molecule contact transparency. The robustness of the lateral geometry should play a major role in the reproducibility of the transport characteristics at a high field. Anyway, to gain a better insight into the charge injection and transport mechanism in nanotrench based large area molecular junctions, the electrical response of the junction should be investigated as a function of the temperature, the thickness of the molecular layer (width of the nanotrenches), and the nature of the metallic electrodes. This is part of future work.

IV. CONCLUSIONS

We have demonstrated a viable lateral architecture of molecular junctions based on the fabrication of laterally aligned metallic electrodes spaced by a distance of the order of 10 nm over lengths of 20 μm obtained by the edge mediated shadow mask technique. The molecular deposition realized by the method of the electro-reduction of diazonium salts has allowed us to successfully bridge the gap between the two electrodes. The whole procedure results in molecular junctions with a very good stability and reproducibility. Surprisingly high current densities are measured in the fabricated devices. Such an approach allows for a better control of the molecular layer thickness inside the junction and a reduced contamination of the molecular layer. Moreover, a lateral junction architecture is particularly suited to investigate electronic transport as a function of external parameters, such as local electrical and optical gates. The improved control over the molecular layer thickness allows us to better investigate the fundamental questions about the nature of the electronic transport in molecular systems.

SUPPLEMENTARY MATERIAL

See the supplementary material for details on the nanotrenches and molecular layer SEM and AFM analysis, the electrografting of the molecular layer in the nanotrench, the nanotrench width statistical analysis, and electrografting tests with a reduced number of grafting cycles and with Al₂O₃ capped electrodes.

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