Quantum Confinement of Dirac Quasiparticles in Graphene Patterned with Sub-Nanometer Precision

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Quantum confinement of graphene Dirac-like electrons in artificially crafted nanometer structures is a long sought goal that would provide a strategy to selectively tune the electronic properties of graphene, including bandgap opening or quantization of energy levels. However, creating confining structures with nanometer precision in shape, size, and location remains an experimental challenge, both for top-down and bottom-up approaches. Moreover, Klein tunneling, offering an escape route to graphene electrons, limits the efficiency of electrostatic confinement. Here, a scanning tunneling microscope (STM) is used to create graphene nanopatterns, with sub-nanometer precision, by the collective manipulation of a large number of H atoms. Individual graphene nanostructures are built at selected locations, with predetermined orientations and shapes, and with dimensions going all the way from 2 nm up to 1 µm. The method permits the patterns to be erased and rebuilt at will, and it can be implemented on different graphene substrates. STM experiments demonstrate that such graphene nanostructures confine very efficiently graphene Dirac quasiparticles, both in 0D and 1D structures. In graphene quantum dots, perfectly defined energy bandgaps up to 0.8 eV are found that scale as the inverse of the dot’s linear dimension, as expected for massless Dirac fermions.

Graphene is the thinnest material ever realized. It has one dimension reduced to the ultimate one atom limit and the other two extending over a macroscopic scale. Since its discovery in 2004,[1] scientists ambition the controlled reduction of the remaining two dimensions down to the nanoscale, as a powerful route to modify its properties by quantum confining graphene electrons. Early attempts to nanopattern graphene using standard lithography made it possible to confine graphene electrons and to open energy bandgaps with the creation graphene nanoribbons and quantum dots[2–5] with very poorly defined edges. Even with today’s state-of-the-art technology, it is challenging to improve the patterning resolution below 10 nm sizes.[5,6] Alternative approaches based on growth techniques of islands and ribbons[7–13] do not allow to easily tune the shape of the objects and usually need a subsequent transfer step when metal substrates are used.[11] The use of a scanning tunneling microscope (STM) to nanopattern graphene was also pursued since the early years,[14–17] and faint gaps inside graphene nanopatterned structures have been measured.[14–16] Some outstanding advances in this direction have been recently achieved with the creation of circular graphene quantum dots composed of p–n junction rings.[18–22] The generation of...
Local electrostatic confinement has allowed probing exciting quantum-relativistic graphene properties, such as quasibound states, Berry phase, or the development of a “wedding cake” line shape in the density of electronic states. However, the graphene quasiparticle confinement there was limited due to the existence of Klein tunneling[23] and only circular-shaped dots with diameters of around 100 nm have been studied, the size and shape being imposed by the source of the local potential (a buried charge).

In this work, we have used H atoms as building blocks to define atomically sharp barriers that efficiently confine graphene electrons in all directions. Low-energy electronic transport in graphene takes place in the $\pi$-orbitals, each carbon atom contributing with one electron. Atomic H chemisorbs on top of graphene carbon atoms, forming a strong $s=\pi$ covalent bond that effectively removes the $\pi$-orbital and one electron of graphene from the conduction band.[24] Large-density hydrogenation of graphene is known to open up a very large gap,[25] providing thereby a confinement barrier for massless Dirac fermions. The use of C–H binding to block graphene $\pi$-electrons has been envisioned since already a decade, both by removing H atoms from fully hydrogenated graphene[15,26,27] and by the patterned adsorption of atomic hydrogen onto a moiré superlattice.[28]

Here, we have developed a new methodology that allows the local manipulation of large ensembles of H atoms (see the Supporting Information). First, we perform macroscopic atomic hydrogenation of the graphene surface by the thermal dissociation of H$_2$.[29] Due to the existence of a physisorption channel,[30] H atoms rapidly diffuse on the graphene surface until they chemisorb, mostly forming highly stable dimers.[30,31] H dimers correspond to pairs of H atoms adsorbed on neighboring C sites,[30,31] shown as bright features in STM images of Figure 1A. Next, we stabilize the sample temperature around 220 K. At this temperature, we nanopattern graphene by displacing the STM tip in closed feedback loop, with a sample voltage of $+4$ V and typical tunneling currents of 0.1–1.0 nA, at a constant velocity of 1 nm s$^{-1}$. This creates narrow H patterns along the path followed by the STM tip, as shown in Figure 1A,B, that shows a 22 nm side triangular graphene quantum dot created using this method. The patterning process is schematically illustrated in Figure 1F.

![Graphene nanopatterning with H atoms. A) 100 × 100 nm$^2$ STM topography of a graphene region after macroscopic hydrogenation. Isolated bright features correspond to H dimers formed on the graphene terrace. On the right side of the image, H atoms populating a grain boundary serve as a reference. B) Same region as in (A) after the creation of a 22 nm triangular side graphene dot by H patterning. C–E) Sequence of consecutive STM images where the versatility and reversibility of the patterning method is shown. Nanostructures with different shape and orientation are built–erased–rebuilt in the same 60 × 60 nm$^2$ region. F) Schematic illustration of the H patterning procedure. G) High-resolution STM image of a H pattern edge, highlighting the accuracy below 1 nm of the patterning process. H) 4 nm wide nanoribbon with a length exceeding 100 nm. I) 30 nm wide nanoribbon continuously patterned across a terrace step between ML and BL. All STM images were measured at 220 K. Graphene was grown on SiC(000-1), except in (I), where it was on SiC(0001).](image-url)
The working principle of the patterning method is as follows. After the adsorption of H atoms (and the subsequent dimer formation aforementioned), setting the sample voltage to +4 V seems to modify the energetic landscape at very long distances, favoring the displacement of H toward the tip position. The drift of chemisorbed hydrogen is thermally assisted, as it only works when we rise the sample temperature to ≈220 K (see the Supporting Information). Figure 1A,B shows two consecutive STM images measured before and after the patterning procedure. It is apparent that H atoms, previously adsorbed on graphene (Figure 1A), are attracted from very long distances to accumulate along the path followed by the STM tip while operated under patterning tunneling parameters (Figure 1B). The resulting H patterns are seen by STM as bright protrusions, as in Figure 1B, or as dark depressions depending on the tunneling conditions (see the Supporting Information). From our experimental data, we cannot precisely determine the atomic scale arrangement of H atoms inside the H-trenches.

The method is highly robust and versatile. H patterning can be implemented along any arbitrary direction, which enables us to build graphene nanostructures with diverse shapes (see in Figure 1C), such as circles, squares, and two triangles with different orientation. Structures with sizes ranging from 1 nm up to few micrometers can be built on demand, just limited by the maximum area the STM can scan. The procedure is completely reversible, in the sense that H patterns can be selectively written–erased–rewritten several times with the same or different shapes. This is illustrated in Figure 1C–E, which shows a sequence of consecutive STM images measured in the same 60 × 60 nm² graphene region. First, we patterned four different nanostructures (Figure 1C). Next, we removed the upper right triangle (Figure 1D), and finally we patterned, in this same spot, a new triangle with a different orientation (Figure 1E). Note that the three other nanostructures remained almost intact during the whole procedure, despite the harsh tunneling conditions (see the Supporting Information) used in the writing/erasing procedure. To selectively remove H patterns, we scan the graphene surface with a close feedback loop at high speed (~200 nm s⁻¹), low bias (few mV), and high currents (~1 nA). The H removal procedure is very local, which also enables us to partially modify the created structures and, for example, remove a small fraction of the H pattern providing a tunable gateway to graphene electrons.

We have verified that the H patterning methods work on different graphene systems, from multilayer graphene on SiC(0001) to monolayer (ML) and bilayer (BL) graphenes on SiC(0001). This is illustrated in Figure 1H, showing a 30 nm wide nanoribbon continuously patterned across a step between an ML and a BL graphene grown on SiC(0001). In all these weakly coupled graphene systems, H patterning could be performed by using the same parameters just described. This suggests that it should be equally possible to use our method in graphene over insulating substrates, suitable for electronic gating.

A key point of our method stems in the spatial resolution that we can achieve. As shown in Figure 1G–I, the H patterns that we create are very straight at the nanometer scale. Although we are not yet able to collectively position H atoms down to the last C atom limit, we can comfortably build graphene structures with sub-nanometer precision, as visualized in the zoomed-in images of Figure 1G,H. Figure 1H shows a 4 nm wide nanoribbon with a length exceeding 100 nm and similar length nanoribbons, with 2 nm width, can be equally patterned even across a terrace step (see the Supporting Information). Close to the patterned H-trenches (Figure 1G), graphene shows a (√3 × √3)R30° superstructure associated with the existence of intervalley scattering processes,[32] as expected for strong scattering created by sharp barriers.

In the following, we present evidence for an efficient confinement of graphene electrons in such designed structures to highlight the usefulness of this flexible patterning technique for the studies of controlled graphene-based nanostructures. We do not intend to present here a thorough analysis of confinement effects in graphene, but we want to demonstrate that the patterned H structures actually behave as very efficient quantum dots. In order to prove that the H patterns produce quantum confinement of graphene electrons, we have first spatially mapped the electronic structure of graphene inside the created nanostructures. Acquiring conductance maps dI/dV(x,y) at different sample voltages V, we have directly visualized the quantum-confined electronic states inside 1D and 0D nanostructures. In the upper panel of Figure 2, we show the results for a 23 nm wide nanoribbon fabricated on BL graphene on SiC(0001). We have chosen here BL graphene since, due to pseudospin, the quasiparticle interferences associated with some scattering processes, in particular those related to intervalley backscattering, are hardly detectable in ML graphene, but are clearly observed in BL graphene[32] (see the Supporting Information). This enables the comparison of our experimental results with a simple hard wall model with no free parameters. The model only includes the width of the ribbon, determined with our STM measurements, and the electronic dispersion of graphene electrons in BL graphene on SiC(0001),[32] and it is assumed that H patterns act as impenetrable hard walls for graphene electrons. As it can be seen in Figure 2A, we clearly visualize the first four electronic bound states above the Dirac point inside the ribbon. In the lower panel of Figure 2, we show a 27 nm side square graphene quantum dot. Again, our scanning tunneling spectroscopy (STS) experiments clearly show the existence of several electronic interference patterns, identified as individual quantum-confined states by our simple particle in a box model. Despite the relative high temperatures (and hence high thermal broadening) imposed by experimental constraints (170 K), and the simplicity of the model, the close resemblance between experiments and model points to an efficient confinement of graphene electrons inside the created H patterns.

Ideally, if graphene electrons are perfectly trapped inside a graphene quantum dot, a well-defined energy gap develops and the lifetime of the confined quantum states should be infinite, just limited by thermal effects. To confirm the efficiency of the confinement inside our H-patterned graphene quantum dots, we acquired dI/dV conductance curves at the lowest achievable sample temperature. We were able to decrease the sample temperature, while maintaining the same scanning region under the tip position, to values as low as 130 K, which is a major experimental challenge, considering that the nanostructures have to be created at T ≃ 220 K. Figure 3A shows a 5 nm side triangular graphene quantum dot, patterned on the surface of...
multilayer graphene grown on SiC(000-1). In this system, the rotational disorder of the graphene layers electronically decouples the π bands, leading to a stacking of essentially isolated graphene sheets,[33–35] with the surface layer equivalent to neutral ML graphene. Reference STS spectra, measured in the clean graphene terrace outside the dot, red curves in Figure 3B, show the characteristic featureless V shape of neutral graphene. STS curves, measured with the same tip on the center of the dot, black curves in Figure 3B, show a well-defined electronic energy bandgap developing inside the triangular dot, proving the strong confinement inside the dot. The dI/dV curve to show the width of the first empty bound state. The experimental FWHM of 40 meV corresponds to the expected thermal broadening at 130 K, which implies a very narrow deconvoluted dI/dV peak, associated with a long lifetime of the graphene electron in the bound state.

Figure 2. Visualization of quantum-confined electronic states inside 1D and 0D graphene nanostructures fabricated on BL graphene on SiC(0001). A) STS experiments on a H-patterned graphene nanoribbon. Left: STM image of the 23 nm wide ribbon. Right: comparison between experimental conductance images at different sample biases and a simple hard wall model showing the first four quantum-confined states. Energies in the model refer to the onset of each confined state. The line profiles show the vertical average of each of the corresponding conductance and LDOS images. B) STS experiments on a patterned graphene nanosquare. Left: STM image of the 27 nm wide nanosquare. Right: comparison between experimental conductance images and the simple hard wall model of a particle in a box. nx/ny refers to the x/y energy levels.

Figure 3. Strong confinement of graphene electrons inside H-patterned graphene quantum dots. A) STM image showing a 5 nm side triangular graphene dot fabricated on an electronically decoupled graphene layer on SiC(000-1). The graphene surface layer is twisted 15° with respect to the next graphene layer, which ensures electronic decoupling. B) STS curves measured, with the same tip at 130 K, in the center of the dot (black curves) and in the outer clean terrace (red curves). A well-defined electronic energy bandgap develops inside the triangular dot, proving the strong confinement inside the dot. C) dI/dV curve to show the width of the first empty bound state. The experimental FWHM of 40 meV corresponds to the expected thermal broadening at 130 K, which implies a very narrow deconvoluted dI/dV peak, associated with a long lifetime of the graphene electron in the bound state.
triangular dot of Figure 3A, show the development of a perfectly defined energy gap delimited by two peaks in the conductance, which correspond to the first occupied and empty bound states (see the black curves in Figure 3B).

We would like to stress here that, within experimental resolution, the conductance goes to zero inside the gap. To our knowledge, this is the first STM observation of a patterned graphene quantum dot showing a well-defined gap, which demonstrates that, in our case, graphene electrons are strongly confined in all three directions. An estimation of the lifetime $\tau$ of the confined states inside the dot can also be obtained from STS data. Our experimental data at 130 K show an full width at half maximum (FWHM) of 40 meV, which can be essentially ascribed to the thermal broadening of STS spectra at such temperature, which is $\approx 3.5k_B T$ at FWHM. From here, we infer an upper bound for the nonthermal broadening, $\Gamma < 5$ meV for the deconvoluted peaks width. Using $\tau = \hbar/\Gamma$, we obtain a lower limit for the lifetime of the bound states $\tau > 100$ fs. This implies a very long lifetime, and thus a very efficient confinement, for the bound states inside the created dot.

Early measurements have revealed a size-dependent bandgap (scaling approximately as the inverse of the mean sample length) in graphene nanoflakes with uncontrolled shape. Thus, one of the most appealing possibilities offered by the controlled creation of graphene quantum dots is the opportunity to open tunable energy bandgaps in the graphene electronic structure. For equally shaped 2D quantum dots, the gap induced by quantum confinement should scale with the inverse of $\sqrt{N}$, where $N$ is the number of C atoms inside the dot, on account of the linear dispersion of massless Dirac electrons. This scaling law has been verified using ab initio calculations in triangular graphene nanodots with regular shapes. Figure 4 shows a terrace of neutral decoupled graphene on SiC(000-1), where three graphene triangular dots with sides of 6, 11, and 23 nm have been created. They all have the same orientation with respect to the graphene layer; their sides form 11° with respect to the graphene zigzag direction (see the Supporting Information). Our STS spectra, measured on the center of the triangles, show the existence of well-defined energy gaps in all three cases (Figure 4B,C). In Figure 4D, we show the evolution

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**Figure 4.** Tunable graphene energy bandgap. A) 85 × 60 nm² STM image showing three triangular graphene dots patterned, on the same graphene terrace, with different sizes. The sides of the triangles are rotated 11° with respect to the graphene lattice. Triangles are fabricated on an electronically decoupled graphene layer on SiC(000-1), twisted 17° with respect to the next graphene layer. B,C) $I/V$ and $dI/dV$ curves, respectively, measured, at 140 K, in the center of each of the triangles shown in panel (A). D) Size dependence of the energy gap $\Delta$, plotted as a function of the number of C atoms inside the dot. Circles correspond to experimental data, crosses to tight-binding simulations, and a dotted line is a fit to the experimental data.
of the energy gap $\Delta$ with quantum dot size. We plot the value of $\Delta$, obtained from the energy difference between the maxima of the conductance peaks corresponding to the first occupied and empty bound states, as a function of $N$. A fit to our experimental data, dotted line in Figure 4D, shows that $\Delta$ is proportional to the inverse of $\sqrt{N}$, as expected for massless Dirac quasiparticles.

We have compared our experimental results with an atomistic tight-binding model (see the Supporting Information) that permits to compute the local density of states (LDOS) in triangular graphene quantum dots of different sizes, maintaining the same 11° orientation with respect to the graphene lattice. We have used a value of $t = 3.4$ eV for the nearest-neighbor hopping energy to account for the $v_F = 1.1 \times 10^6$ m s$^{-1}$ experimentally found.\footnote{In line with the experimental approach, we estimate the tight binding value of $\Delta$ from the energy difference between the peak maxima of the first occupied and empty bound states in the LDOS (see the Supporting Information). As shown in Figure 4D, there is an excellent agreement between calculated (black crosses) and experimental (open circles) values of $\Delta$, confirming that the patterned nanostructures definitely behave as strongly confining graphene quantum dots (see the Supporting Information).}

To summarize, we have developed a novel nanopatterning technique that enables us to reproducibly build, with sub-nanometer precision, H trenches acting as hard walls for graphene electrons. As we have demonstrated, this enables us to efficiently confine graphene electrons in 1D and 2D graphene nanostructures of complex shapes and the selective opening of energy gaps in the graphene electronic band structure. The versatile tunability of the presented quantum dots and their selective opening will open a plethora of exciting new possibilities, based on the engineering of artificial Hamiltonians in a graphene platform. In particular, selectively coupled quantum dots can be exploited to engineer artificial topological crystalline phases, higher-order topological insulators, and frustrated lattices, the latter providing potential playgrounds for exotic correlated states such as quantum spin liquids.

**Experimental Section**

The experimental data here reported were acquired at 220–130 K by using a home-made low-temperature scanning tunneling microscope in ultrahigh vacuum conditions. Conductance spectra and conductance maps were obtained using a lock-in technique, with an ac voltage (frequency $= 830$ Hz; amplitude $= 5–10$ mV rms) added to the dc sample bias. The data were acquired and processed using the WSxM software.\footnote{Supporting Information is available from the Wiley Online Library or from the author.}

**Supporting Information**

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**Conflict of Interest**

The authors declare no conflict of interest.

**Keywords**

atomic manipulation, graphene, graphene quantum dots, nanopatterning, scanning tunneling microscopy

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