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The manipulation of C₆₀ in molecular arrays with an STM tip in regimes below the decomposition threshold

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Received 27 July 2012, in final form 18 December 2012

Published 11 January 2013

Online at stacks.iop.org/Nano/24/055302

Abstract

The ability of scanning tunneling microscopy to manipulate selected C₆₀ molecules within close packed C₆₀ arrays on a (Au,In)/Si(111) surface has been examined for mild conditions below the decomposition threshold. It has been found that knockout of the chosen C₆₀ molecule (i.e., vacancy formation) and shifting of the C₆₀ molecule to the neighboring vacant site (if available) can be conducted for wide ranges of bias voltages (from -1.5 to $+0.5$ V), characteristic manipulation currents (from 0.02 to 100 nA) and powers (from 2×10^{-8} to 0.1 μ W). This result implies that the manipulation is not associated with the electrical effects but rather has a purely mechanical origin. The main requirement for successful C₆₀ knockout has been found to be to ensure a proper ‘impact parameter’ (deviation from central impact on the C₆₀ sphere by the tip apex), which should be less than ~ 1.5 Å. A certain difference has been detected for the manipulation of C₆₀ in extended molecular arrays and molecular islands of a limited size. While it is possible to manipulate a single C₆₀ molecule in an array, in the case of a C₆₀ island it appears difficult to manipulate a given fullerene without affecting the other ones constituting the island.

(Some figures may appear in colour only in the online journal)

1. Introduction

The paradigm of molecular electronics is based on the idea of using single molecules or their groups in nanoelectronics [1]. Fullerenes are considered to be especially attractive for future device applications due to their ability to change their electronic properties by adopting electrons from other molecules or substrate surfaces [2]. The manipulation of molecules is a promising technique for the fabrication of molecular nanostructures with desired geometries and properties. Scanning tunneling microscopy (STM) has been shown to be a powerful tool for achieving the goal of controlled manipulation of atoms and molecules on surfaces [3–9]. Various types of molecule manipulations can

be performed with an STM tip, including pushing, pulling, sliding and even molecule decomposition. The manipulation mechanisms can be very complicated since the effects of the tip on the manipulated molecule can be manifold. High electrostatic fields, high-density electron currents and interatomic forces occurring in the tip–molecule gap can affect the molecule. The ability to control the tip conditions (bias voltage, tunneling current, tip height) in a desired way opens an opportunity to clarify the nature of manipulations. An additional merit of STM is its capability of visualizing the result of manipulations on the atomic scale by using milder tunneling conditions.

For C₆₀ fullerenes on solid surfaces [10, 11], most work has so far been devoted to manipulation of isolated

single fullerenes. For example, rolling of C_{60} along the troughs between Si dimer rows on a Si(100) surface has been explored in detail both experimentally [12, 13] and theoretically [14–16]. Pushing of C_{60} via the repulsive interaction with the tip has been demonstrated both along a plane Cu(111) surface [17, 18] and along an atomic step on it [19]. Manipulation of C_{60} forming a continuous array means removal of selected fullerenes to form a desired vacancy pattern. STM-induced decomposition of selected C_{60} molecules within an array on a metal surface as a result of heating the molecule by an electron current passing through the junction has been demonstrated in [20, 21]. The average power for thermal decomposition appears to depend on the substrate material, being $21 \mu\text{W}$ for C_{60} on Cu(110), $2.9 \mu\text{W}$ on Pb(111) and $1.0 \mu\text{W}$ on Au(111) [21]. It is worth noting that the products of C_{60} decomposition can be left at the surface leading to undesirable contamination. Thus, the goal of this study was to look for regimes where a chosen C_{60} molecule can be removed from a molecular array without destruction. The complementary goal was to check this possibility on metal-reconstructed Si surfaces, which present a type of advanced surface due to the possibility to vary their structure and properties. In this paper, we report on the results of STM-induced manipulation of selected C_{60} molecules within close packed hexagonal molecular arrays adsorbed onto an In-modified $\text{Si}(111)\sqrt{3} \times \sqrt{3}$ -Au surface. In our experiments, we have used a ‘z-pulse’ technique similar to that reported in [20, 21]. Our observations demonstrate that by using relatively mild tunneling conditions it is possible to remove C_{60} without its decomposition. The results appear to be quite similar for sample voltages in the range from -1.5 to $+0.5$ V, for various currents ranging from 0.02 to 100 nA and powers in the 2×10^{-8} to $0.1 \mu\text{W}$ range. This implies that the manipulations are associated with the purely mechanical impact of the STM tip on C_{60} molecule.

2. Experimental details

Our experiments were performed with an Omicron STM operating in an ultrahigh vacuum ($\sim 2.0 \times 10^{-10}$ Torr). Atomically clean $\text{Si}(111)7 \times 7$ surfaces were prepared *in situ* by flashing to 1280°C after the samples had first been outgassed at 600°C for several hours. Gold was deposited from an Au-wrapped tungsten filament, indium from a Ta crucible and C_{60} fullerenes from a resistively heated Mo crucible. To prepare the In-modified $\text{Si}(111)\sqrt{3} \times \sqrt{3}$ -Au surface, a two-step procedure was employed. First, a $\text{Si}(111)\alpha\text{-}\sqrt{3} \times \sqrt{3}$ -Au surface [22] was formed by Au deposition onto a $\text{Si}(111)7 \times 7$ surface held at 700°C . Then, ~ 0.5 ML of In was deposited onto this surface held at room temperature followed by brief (~ 15 s) annealing at 600°C . The resultant surface was highly ordered and homogeneous, being free of domain walls, characteristic of the pristine $\text{Si}(111)\sqrt{3} \times \sqrt{3}$ -Au surface. It preserves the atomic arrangement of the $\sqrt{3} \times \sqrt{3}$ -Au phase described by the conjugate honeycomb chained trimer (CHCT) model [23, 24] and contains ~ 0.14 ML of In left after the high-temperature treatment and forming a 2D gas of adatoms [25, 26]. These

In adatoms are highly mobile at room temperature, but their mobility slows down with cooling until the adatoms become almost fixed in their positions at 120 K. Although this surface possesses a number of peculiar properties (e.g., it shows up as a perfect isotropic two-dimensional electron-gas system [26]), its main merit for the present study resides in the ability of reproducible and easy preparation of a homogeneous highly ordered substrate surface with a defect density much lower the level typical for most metal/silicon reconstructions.

Room-temperature deposition of C_{60} onto this surface results in the formation of close packed hexagonal C_{60} molecular arrays whose periodicity coincides with the bulk fullerite nearest-neighbor distance of 10.02 \AA [27]. Two types of C_{60} arrays with different orientations have been detected. In the arrays of the first type (0° -rotated arrays), the molecular rows are aligned along the principal crystallographic directions of the $\text{Si}(111)$ surface, i.e., $\langle 10\bar{1} \rangle$. These arrays display a characteristic moiré pattern of dim and bright C_{60} fullerenes which indicates the difference in their adsorption geometries (hence, the possible difference in their adsorption energies). In the arrays of the second type (19.1° -rotated arrays), the molecular rows make angles of $\pm 19.1^\circ$ with the $\langle 10\bar{1} \rangle$ directions. In the 19.1° -rotated arrays, all fullerenes exhibit the same STM contrast, which can be treated as a sign of a similar bonding state. Therefore, we have conducted our experiments with C_{60} manipulations at the 19.1° -rotated arrays to simplify analysis of the results by the suggestion that all the C_{60} molecules are in similar bonding states. It is worth noting that high-resolution STM observations of the adsorbed fullerenes have not revealed the presence of any intramolecular features, which plausibly means that C_{60} molecules reside in continuous rotation even at low temperatures. Therefore, effect of C_{60} orientation on the manipulation efficiency has not been considered in the present study.

For STM observations and C_{60} manipulation, electrochemically etched tungsten tips cleaned by *in situ* heating were employed. The STM observations and C_{60} manipulation were conducted on samples cooled to 120 K.

3. Results and discussion

Each of the experiments was as follows. First, an STM image of the C_{60} array was acquired using normal tunneling conditions. Then, an STM tip was placed above a chosen C_{60} within an array, the feedback was switched off, the desired bias voltage V_s and current were preset, and the tip approached a distance Z towards a C_{60} and retreated back holding V_s constant. During tip approach and retreat, the current flowing through the molecular junction was recorded as a function of tip displacement, $I(Z)$. The acquisition time was ~ 300 ms for each $I(Z)$ branch. The result of manipulation was checked with subsequent STM observations at normal conditions. However, the shape of the $I(Z)$ dependence can already provide an indication on the C_{60} manipulation that has occurred. As an example, figure 1 shows typical $I(Z)$ plots for the cases when the C_{60} is displaced and when it is not. The most essential sign of C_{60} displacement is an abrupt drop

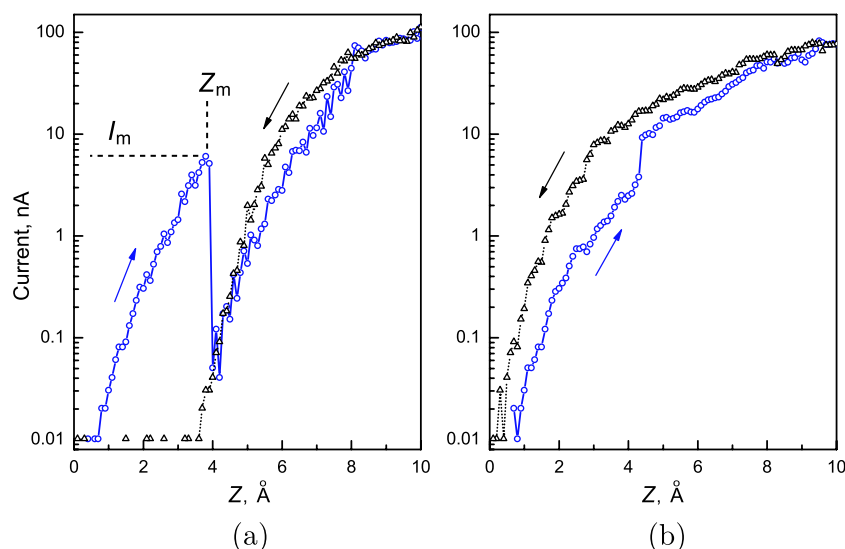


Figure 1. Typical $I(Z)$ plots in which the approach branch is shown by blue solid lines and open circles and the retreat branch by black dashed lines and open triangles for the cases (a) when C_{60} manipulation takes place and (b) when it does not. The sample bias voltage is +0.25 V and the preset current is 0.01 nA.

at the approach branch of the $I(Z)$, the next is the difference between the approach and retreat branches of the $I(Z)$ curve at $Z \leq 4$ Å. Both features are naturally explained by removal of C_{60} from under the tip apex. The current drop corresponds to the exact moment of C_{60} removal. In the absence of C_{60} in the junction, the current is lower than that when C_{60} is present, hence the difference between approach and retreat $I(Z)$ branches. When complete removal of C_{60} does not take place (say, when the C_{60} molecule is shifted from its original position but still remains under the tip), there are no abrupt features in the $I(Z)$ plot and its approach and retreat branches are very similar to each other.

Figures 2 and 3 present typical manipulation examples, where each panel shows STM images of the C_{60} array before and after manipulation and the corresponding $I(Z)$ plot. In particular, figure 2(a) shows an example where a pre-selected C_{60} molecule disappears as a result of the interaction with the tip (the sample bias being +0.5 V) and a vacancy is formed in the C_{60} molecular array. A similar result but with nominally zero sample bias voltage is shown in figure 2(b). One can see that in this case the $I(Z)$ plot can be recorded only after moving the tip ~ 7 Å towards the surface, when a tiny residual voltage (which is ~ 1 mV according to our estimation) causes a detectable value of current (the detection limit was 0.01 nA). Figures 3(a) and (b) show another type of manipulation, namely repositioning of a C_{60} molecule within the array by tip impact on a fullerene neighboring an existing vacancy. The manipulation shown in figure 3(a) is conducted at a sample bias of +0.5 V and that in figure 3(b) at nominally zero voltage. In the $I(Z)$ plot in figure 3(b), one can notice a small current peak in the approach branch at $Z \approx 4.5$ Å, which is reminiscent of the abrupt current drop seen in figures 2(a) and 3(a). A general conclusion that can be derived is that the shapes of the $I(Z)$ plots are very similar for complete removal of C_{60} and shifting it to a neighboring vacant site.

Consider now the parameters at which C_{60} manipulation events have been detected in the present experiments. Figure 4(a) shows the characteristic displacement Z_m (see figure 1(a)) at which an abrupt drop in the $I(Z)$ plot (the signature of the C_{60} manipulation event) is observed as a function of the sample bias voltage V_s . One can hardly notice any well-defined correlation between Z_m and V_s . The Z_m deviates randomly from one experiment to another and averaging yields a value of 4.7 ± 0.6 Å. Figure 4(b) illustrates how the sample bias affects the peak current value I_m when a manipulation event takes place (for the definition of I_m see figure 1(a)). One can see that the lower V_s is the lower I_m is, which is a natural consequence of the fact that manipulations take place at almost the same Z_m , hence the plot in figure 4(b) just reflects the I - V curve for this tip-sample separation. In particular, the dependence asymmetry with respect to zero voltage is reminiscent of the C_{60} HOMO and LUMO band structure: the current growth is faster at positive than at negative sample voltage, since the LUMO energy for C_{60} on a metallic surface is typically between +0.5 and +1.0 V, while the HOMO energy is between -1.5 and -2.0 V [28, 29]. What is more essential is that C_{60} manipulation can occur at any I_m value in a very wide range of 0.02 to 100 nA, i.e., covering four orders of magnitude. A similar regularity is valid for the power P_m to be dissipated in the junction during the manipulation event. The range of well-defined P_m values is from 3×10^{-5} to $0.2 \mu\text{W}$, but it can be extended downward if one takes into account the power dissipated at nominally zero bias voltage. In this case the actual voltage is not more than ~ 1 mV, $I_m \approx 0.02$ nA, hence the upper estimation for P_m is $2 \times 10^{-8} \mu\text{W}$.

Let us address now the possible mechanisms underlying C_{60} manipulation in the regimes used in the present study. By analogy with the reported results [20, 21], one could tentatively suggest thermal decomposition of C_{60} , at least for

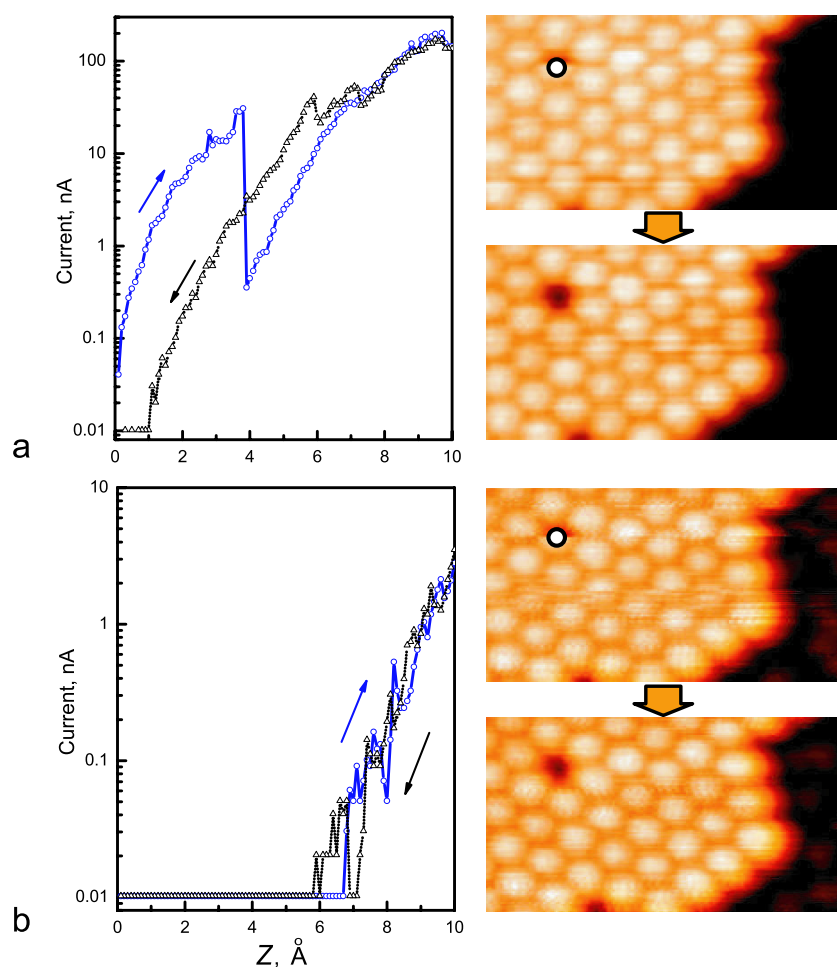


Figure 2. Knockout of the pre-selected fullerene (vacancy formation). $I(Z)$ plots and successive STM images before and after knockout of the chosen C_{60} (marked by a white dot) (a) at a sample bias of +0.5 V and (b) at nominally zero bias voltage.

the cases where the C_{60} molecule disappears after STM-tip impact. However, there are several arguments against this suggestion. First, the fact that C_{60} manipulation occurs in a similar way at any I_m and V_s , including very small currents and voltages, implies that effects associated with current flow or the presence of an electric field in a junction could hardly be responsible for C_{60} removal. Second, the similarity of the $I(Z)$ plots for C_{60} removal and shifting suggests a similar manipulation mechanism, which in the latter case is definitely pushing of the fullerene without any sign of its decomposition or even distortion of its original structure. Third, after C_{60} disappearance no traces of decomposed C_{60} fragments can be found. In some experiments, we observed that a neighboring C_{60} molecule can move to the formed vacancy. After filling it, the C_{60} preserves its STM appearance, which is possible only when nothing else is left at this region of the surface. Fourth, the range of powers at which C_{60} manipulation proceeds in the present study ($0.2 \mu\text{W}$ at maximum) is far below the reported threshold powers for C_{60} thermal decomposition (1.0 to $21 \mu\text{W}$ [20, 21]).

Thus, one can conclude that the observed manipulation has plausibly a purely mechanical origin (in the sense that it is controlled by the repulsive interatomic forces between

the tip apex and the C_{60}) and the manipulation mechanism is thought to be as follows. The approaching tip presses down the fullerene and induces a stress in it. The accumulated strain energy at some moment could be abruptly released, causing a shift of the fullerene (if a neighboring vacant site is available) or its knockout (if all neighboring sites are occupied). The knocked-out C_{60} can freely migrate a long distance over the C_{60} array until it is trapped at some point at its periphery. In some experiments, we were able to detect the appearance of a new fullerene at the edge of an array within a scanned area.

The main requirement for successful C_{60} manipulation has been found to be associated with achieving a proper ‘impact parameter’ (see figure 5). The ‘impact parameter’ is defined as the deviation from central impact on the C_{60} sphere by the tip apex, as illustrated by the schematics in the inset in figure 5. This illustration reflects our conception of what the STM tip looks like. It is known that an etched tungsten tip has an apex with a typical radius of curvature of 100 nm, which means that originally it shows up as almost flat on the atomic scale. However, *in situ* training of a tip by current and voltage pulses eventually results in the development of a ‘nanotip’ having an atomic-scale pyramidal shape, which ensures acquisition of high-resolution STM images. It is

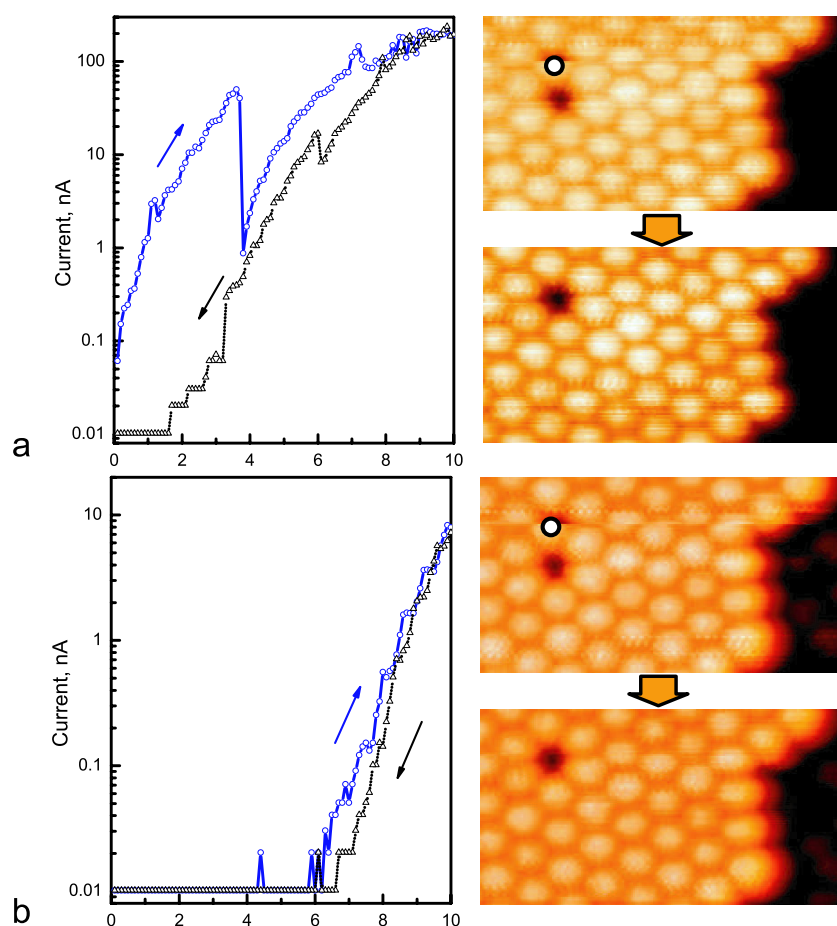


Figure 3. Shifting of the pre-selected fullerene to a neighboring vacant site (a) at a sample bias voltage of +0.5 V and (b) at nominally zero bias voltage.

reasonable to suggest that the same ‘nanotip’ is responsible both for acquisition of STM images and for manipulating surface species, adatoms or molecules, since this tip feature is the closest one to the sample surface.

To control the ‘impact parameter’ accurately, the following experimental procedure was used. A selected surface area with a C_{60} array was recorded several times to minimize the drift, so that two subsequent STM images practically coincided. In that case, the drift rate was typically less than $\sim 0.3 \text{ \AA/s}$. When approaching the target C_{60} during scanning of the final image, the point for manipulation was selected manually and a manipulation macro was executed. After executing the macro (which took $\sim 0.6 \text{ s}$, hence the drift was less than $\sim 0.2 \text{ \AA}$), scanning was continued automatically. After complete recording of the frame, the distance from the manipulation point to the center of the target C_{60} molecule (i.e., the ‘impact parameter’) was determined.

One can see that knockout of fullerene is possible only with an ‘impact parameter’ of less than $\sim 1.5 \text{ \AA}$. Otherwise, the tip-induced stress is plausibly dissipated among neighboring fullerenes and the fullerene to be manipulated does not accumulate sufficient energy for the knockout. The shifting manipulation of C_{60} to the neighboring vacant site is naturally not so critical on this requirement and

can occur at greater ‘impact parameters’ (provided that the hit is applied to a proper C_{60} side).

In addition, we examined the possibility of C_{60} manipulation within molecular arrays of limited size, namely hexagonal islands consisting of 37 fullerenes, like the one shown in figure 6(b). This type of C_{60} island is the most abundant species in a C_{60} island array formed by depositing 0.1 C_{60} ML at 110 K followed by prolonged annealing at room temperature. It has been established [27] that the bright fullerene in the island center resides directly atop a Au-trimer of the underlying $\text{Au/Si(111)}\sqrt{3} \times \sqrt{3}$ CHCT substrate surface [23, 24], while the other molecules occupy various less regular sites. In the experiment, the bright fullerene was the target for manipulation. Figure 6(a) presents a typical approach branch of the $I(Z)$ plot for manipulation of a C_{60} molecule in the island. If we compare it with those for C_{60} manipulation within extended molecular arrays (figures 2(a) and 3(a)), we can find apparent differences. Namely, the characteristic current drop is less pronounced and is shifted towards greater displacements Z_m . Sometimes, some additional features looking like even smaller current drops can be noticed in the $I(Z)$ plots (e.g., see the one at $Z \approx 4.0 \text{ \AA}$ in figure 6(a)). These features indicate that manipulation can proceed in a rather complicated multi-step way. The subsequent STM observations indeed confirm that

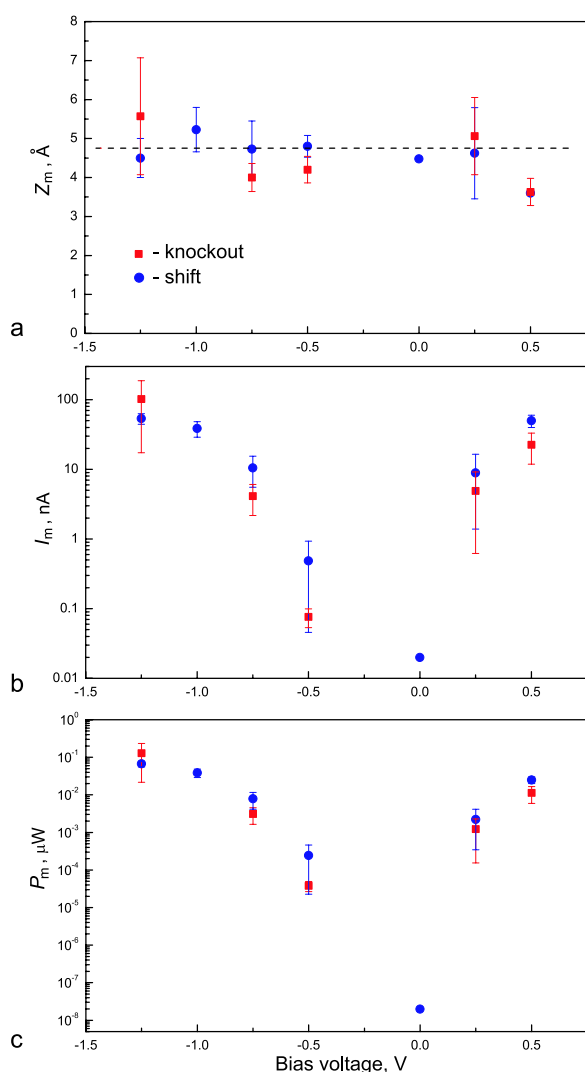


Figure 4. The parameters at which C_{60} manipulation events (red squares for C_{60} knockout and blue circles for C_{60} shifting) take place, including (a) the characteristic manipulation displacement Z_m , (b) the manipulation current I_m and (c) the manipulation power P_m , as a function of the sample bias voltage. The meanings of Z_m and I_m are illustrated in figure 1(a).

several C_{60} molecules (if not all) have been involved in the reordering process. Various results can be obtained in such a manipulation, some of which are presented in figures 6(c)–(e). In figure 6(c), one can see that the island preserves its shape but is shifted as a whole (as indicated by the appearance of the bright C_{60} in the non-central position). In figure 6(d), the result of manipulation is just the formation of a vacancy in the island center by knocking out the central C_{60} or moving a C_{60} row towards the island edge. In figure 6(e), already three fullerenes have been displaced from the island center to its edge, which also causes reordering among the inner C_{60} molecules. These observations seem to emphasize the enhanced flexibility of the fullerenes in an island, hence it is difficult to manipulate a single fullerene without affecting other ones.

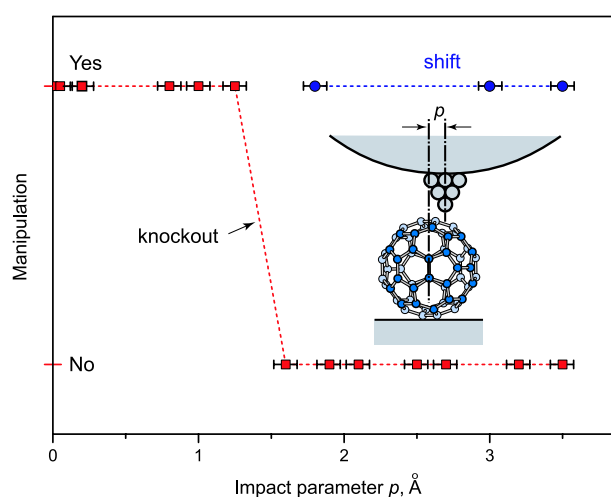


Figure 5. A schematic graph showing how the ‘impact parameter’ p affects whether a manipulation event (red squares for C_{60} knockout and blue circles for C_{60} shifting) takes place or not. A sketch is given to illustrate the meaning of the ‘impact parameter’ p as the deviation from central impact on the C_{60} sphere by the nanotip apex.

4. Conclusions

In conclusion, we have examined the possibility of manipulating C_{60} molecules within close packed molecular arrays with an STM tip in regimes below the decomposition threshold. Two types of manipulation have been considered, knockout of the chosen C_{60} molecule (i.e., vacancy formation) and shifting of the C_{60} molecule to a neighboring vacant site. It has been found that this manipulation can be conducted in wide ranges of bias voltages (from -1.5 to $+0.5$ V), characteristic manipulation currents (from 0.02 to 100 nA) and powers (from 2×10^{-8} to 0.1 μW). This implies that the manipulation is not associated with the electrical effects but rather has a purely mechanical origin. The approaching tip presses down the fullerene and induces a stress in it. The accumulated strain energy at some moment is abruptly released causing a shift of the fullerene (if a neighboring vacant site is available) or its knockout (if all neighboring sites are occupied). The main requirement for successful C_{60} knockout has been found to be associated with achieving a proper impact parameter, which should be less than ~ 1.5 Å. A certain difference has been detected for manipulating C_{60} in extended molecular arrays and molecular islands of limited size (e.g., hexagonal C_{60} islands containing 37 fullerenes). While it is possible to manipulate a single C_{60} molecule in an array, in the case of a C_{60} island it appears difficult to manipulate a given fullerene without affecting other ones.

As a final remark, we would like to outline that our findings have shed some light on the mechanisms involved in the ‘mechanical’ interaction between the STM tip and the C_{60} molecule adsorbed at the surface. We believe that these findings could be understood more deeply if it were possible to conduct appropriate simulations of how the atomically sharp tip presses the fullerene at an off-centered point under the condition that the given fullerene is gripped by neighboring fullerenes. This could help us to learn more about

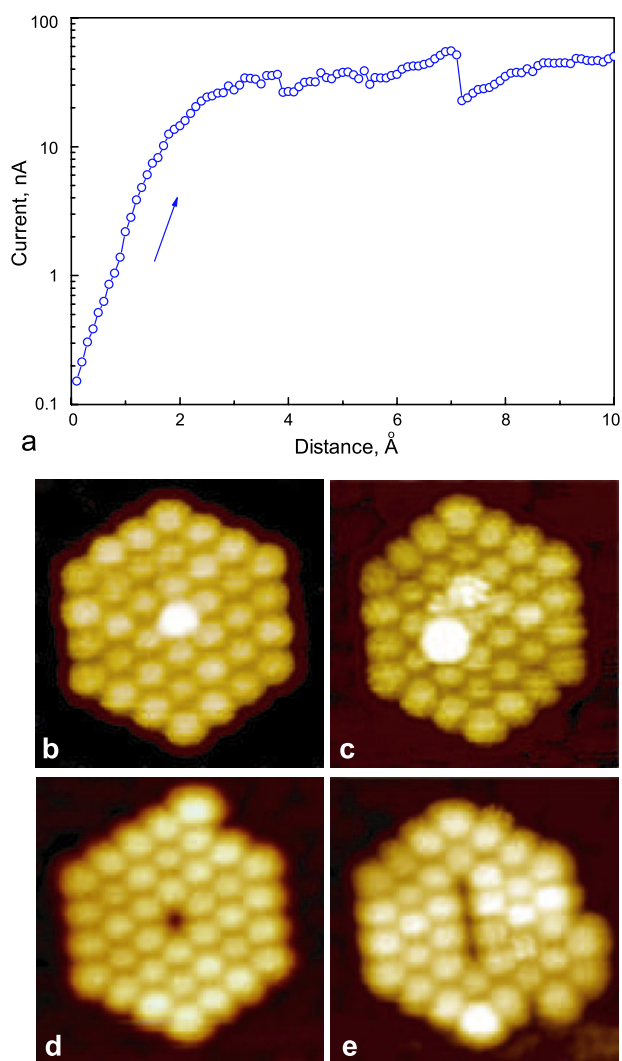


Figure 6. Manipulation of C_{60} within a C_{60} hexagonal island built of 37 C_{60} molecules. Typical $I(Z)$ plot (approach branch) (a), and STM images of the island before (b) and after (c)–(e) the manipulation event.

the elasticity and stiffness of the C_{60} molecule. Regarding experimental studies, it seems interesting to elucidate how the C_{60} adsorption energy affects the manipulation efficiency. 0° -rotated C_{60} arrays on an In-modified $Si(111)\sqrt{3} \times \sqrt{3}$ -Au surface are promising candidates for such a study, since these arrays display a characteristic moiré pattern of dim and bright C_{60} fullerenes [27], which indicates the difference in their adsorption geometries (hence, the possible difference in their adsorption energies).

Acknowledgments

Part of this work was supported by the Russian Foundation for Basic Research (Grant Nos 11-02-98515r, 11-02-98516r, 12-02-00416 and 12-02-00430), and the Ministry of Education and Science of the RF (Grant Nos 8022, 8581 and 2.1004.2011) and NSh-774.2012.2.

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