

## Supramolecular self-assembly initiated by solid-solid wetting

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**Abstract:** We present a preparation method for self-assembled supramolecular monolayers of unsubstituted organic semiconductors and pigments on a solid substrate, applicable under ambient conditions. The deposition is based on a solid-solid wetting phenomenon, whereas the subsequent layer growth proceeds according to standard models. Molecular adsorption

results from direct contact of the compound in a nano-crystalline state with the solid surface. Based on complementary force field calculations, we propose that molecules disintegrate from the crystalline state and adsorb on the surface because of a gain in binding energy. The preparation method is exemplified by means of a linear hydrogen bonded system, namely

Quinacridone (QAC) on graphite. In addition, the chosen system allows to actively guide the self-assembly after deliberate removal of molecules from a predefined area.

**Keywords:** adsorption · self-assembly · nanostructures · scanning probe microscopy · semiconductors

### Introduction

Whereas conventional microfabrication methods like lithographic techniques (top-down approach) encounter principal physical[1] and economical[2] limitations when approaching the length scale of molecules, molecular self-assembly processes[3] are widely explored to develop bottom-up approaches for nano-scale structures without such limitations.[4] In contrast to covalent self-assembly[5] (e.g. fullerenes, carbon nanotubes), which is attractive for the growth of inorganic, rigid building blocks with high charge carrier mobility,[6,7] supramolecular self-assembly[5,8-12] is discussed for easy, low-cost fabrication under ambient conditions. Moreover, the reversibility of non-covalent bonds at room temperature offers the possibility of going beyond simple miniaturisation by introducing dynamic features to surface

supported devices such as reconfiguration, self-repairing and self-contacting.[13]

For the preparation of surface supported supramolecular structures a variety of methods has already been established. Thermally stable compounds can be deposited in a precisely controllable manner by vacuum sublimation, commonly referred to as Organic Molecular Beam Epitaxy (OMBE). A clear disadvantage of this method, particular in the light of future applications, is the enormous instrumental effort. Also, this approach is limited to molecules which are sufficiently stable for sublimation. On the other hand, many alternative techniques are based on dissolving the molecules and utilising the solution as a carrier to transport molecules to the interface.[14-16] However, this method suffers a severe disadvantage when the compound of interest is hardly soluble, as for instance many organic semiconductors or pigments. Here we show that these drawbacks can easily be overcome by a simple process applicable under ambient conditions. This novel straightforward preparation method is suitable for a targeted coverage in the monolayer regime. The basic idea is as follows: nano-crystals (pure or suspended within a matrix) of the respective compound are brought into direct contact with the substrate. When the binding energy of the molecule to the substrate, i.e. the adsorption energy, exceeds the binding energy of surface molecules in the nanoparticle, single molecules detach from the nanoparticle, adsorb on the substrate surface and form monolayers. Eventually, combination of thermally activated diffusion and intermolecular forces results in self-assembly of highly ordered supramolecular structures, if the molecule-substrate interaction is not too strong. In this respect the behaviour is analogous to the spreading of a liquid on a solid, provided the interface tension exceeds the surface

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tension. Thus, we refer to the proposed process as “solid-solid wetting initiated self-assembly”.

The proposed novel preparation method is exemplified with Quinacridone (5,12-Dihydro-quinol[2,3-b]acridine-7,14-dione) as supramolecular building block. Its structure is depicted in the inset of Figure 4. Quinacridone (QAC) is an organic semiconductor[8,17,18] and a commercial product of the pigment industry.[19] It is readily available in a nano-crystalline state with an average primary particle size of ~70 nm in two different polymorphic modifications (beta-phase: H-bonded molecules are interconnected within a plane; gamma-phase: H-bonded molecules form a criss-cross-structure).[18] For this chosen model system – QAC on graphite - we find rather interesting properties. Due to the structure of the QAC molecule, self-assembly in linear structures is favoured. These chains are not necessarily straight and neighboring chains are not densely packed. Kinks within the chains and gaps of various sizes between them give rise to a far more complex structure than just a truly periodic pattern. Thus, the presented system principally offers the prospect to encode information. Moreover, through interaction with an invasive probe, QAC on graphite exhibits another interesting property: the possibility of locally steering the self-assembly, i.e. the re-assembly after disintegration of the supramolecular structure is guided by the scanning process in a defined manner. For the re-adsorption a constant supply of molecules is required, a criterion that can not be met by standard (O)MBE techniques[20] or the precursor method.[21]

Usually two dimensional self-assembly from the liquid results in periodic structures, apart from defects and domain boundaries.[8-10] Normally when molecules are removed from the substrate, these periodic structures just grow back, hence limiting the freedom for actively steering the outcome of the self-assembly process. Here we show that our new approach applied to QAC on graphite opens up a way to steer the self-assembly process in order to deliberately create desired nanostructures.

## Results and Discussion

QAC monolayers were generated by direct deposition of nanoparticle powder on the (0001) surface of highly oriented pyrolytic graphite (HOPG) and a subsequent removal of the powder from the HOPG surface. Similar supramolecular structures were also observed, when the QAC nano-crystal powder was mixed up with the liquid crystal Octylcyanobiphenyl (8CB)[22] and a small amount of the mixture was applied to the graphite surface. Figure 1a presents a high resolution Scanning Tunneling Microscopy (STM) image of QAC adsorbate nanostructures. QAC molecules assemble at the graphite base plane into distinct linear chains with a uniform width of 1.63 nm and a lattice constant of 0.70 nm along the chain. Based on the STM image contrast and the geometrical dimensions in high resolution images, we conclude adsorption of QAC parallel to the substrate. This is supported by a DFT simulation of the image contrast, as superimposed on the STM image in Figure 1a. The calculation depicts the LUMO of a QAC molecule. The typical S-shape and the bright centre appearing in the STM measurements of QAC are well-reproduced by the DFT simulation. Within the QAC chains, neighbouring molecules are interconnected by two O···N-H hydrogen bonds with each neighbour. The bonding pattern is illustrated by the overlaid model in Figure 1a, representing the minimum energy structure as derived by force-field calculations. Figure 1b provides an overview of the encountered complexity in

the self-assembled QAC-wires on HOPG. An individual nano-wire can feature kinks of discrete angles while multi-wire arrays exhibit discrete, non-equidistant gaps between parallel wires. The distances between adjacent wires were measured to be integer multiples of the substrate lattice vectors, indicating the existence of an epitaxial relationship. Nevertheless, the observed assemblies are far more complex than ordinary quasi periodic monolayer structures. Crystallographic details of the structure will be published elsewhere.

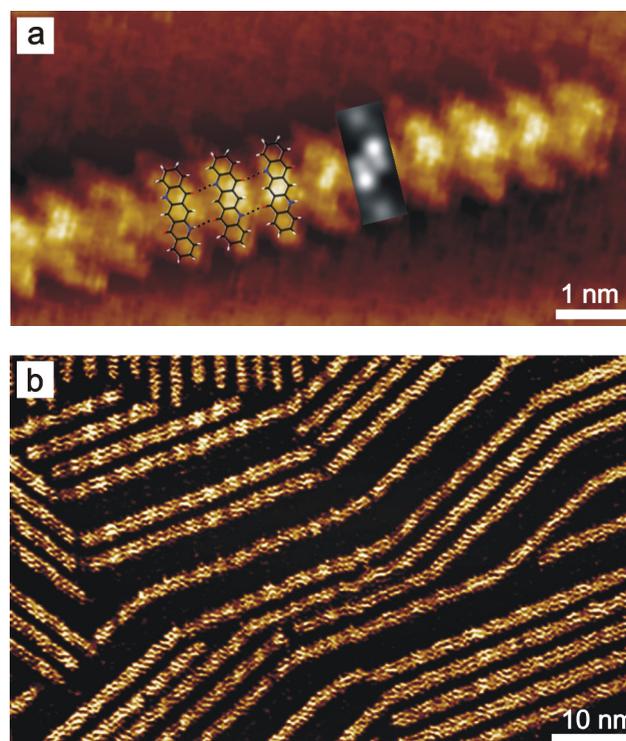


Figure 1. Characterisation of a QAC nano-wire. a) High-resolution STM image of a single QAC nano-wire adsorbed on a graphite surface. Overlaid on the image is a force-field simulation of adsorbed QAC molecules, dotted lines indicate H-bonds. Inset: image of the LUMO of QAC, as simulated by DFT calculations. The molecular orbital calculation image has been positioned at a site equivalent to the shown molecular models. b) STM image of a QAC nano-wire array exhibiting complex features.

We propose that the transfer of QAC molecules from the nanoparticle to the substrate occurs as a consequence of direct contact, rather than being mediated through dissolution and diffusion in 8CB. This model is also supported by experiments without the 8CB matrix as described below. A mixture with 1 ppm QAC in 8CB still exhibits sedimentation after heating up to 60°C and subsequent centrifugation, thus a potential actual solubility is definitely below 1 ppm. Molecular coverages as depicted in Figure 1b and Figure 3a were obtained by applying ~0.4 mg QAC/8CB mixture per cm<sup>2</sup> on HOPG. In order to provide enough molecules for this coverage (8x10<sup>13</sup> molecules/cm<sup>2</sup>), a minimum concentration of ~100 ppm QAC in 8CB is required. Based on the above mentioned upper boundary for the solubility the number of potentially dissolved QAC molecules is not sufficient. In order to supply the additionally required molecules the equilibrium between solution and solid state has to be maintained by dissolution and diffusion of molecules. The reported coverages, however, were observed instantaneously after the mixture has been applied to the substrate and did not change during the experiment. Since at room temperature the liquid-crystal 8CB is in a smectic-A phase with a

layer sliding viscosity of 100 mPa s,<sup>[23]</sup> relatively fast diffusion of a fairly large molecule such as QAC can be ruled out. Thus mediation of the adsorption through dissolution and diffusion of QAC in the 8CB matrix can not explain our experimental observations. In addition, confocal Raman microscopy on embedded nano-crystals suggests that QAC remains in its nano-crystalline state upon mixing with 8CB. Spatial mapping of a QAC indicative frequency band reveals aggregation of the pigment in the 8CB matrix: areas with high intensity were found, which originate either from single nano-crystals or aggregations thereof. However, the Raman intensity between those QAC containing areas was virtually zero. Details will be published elsewhere.

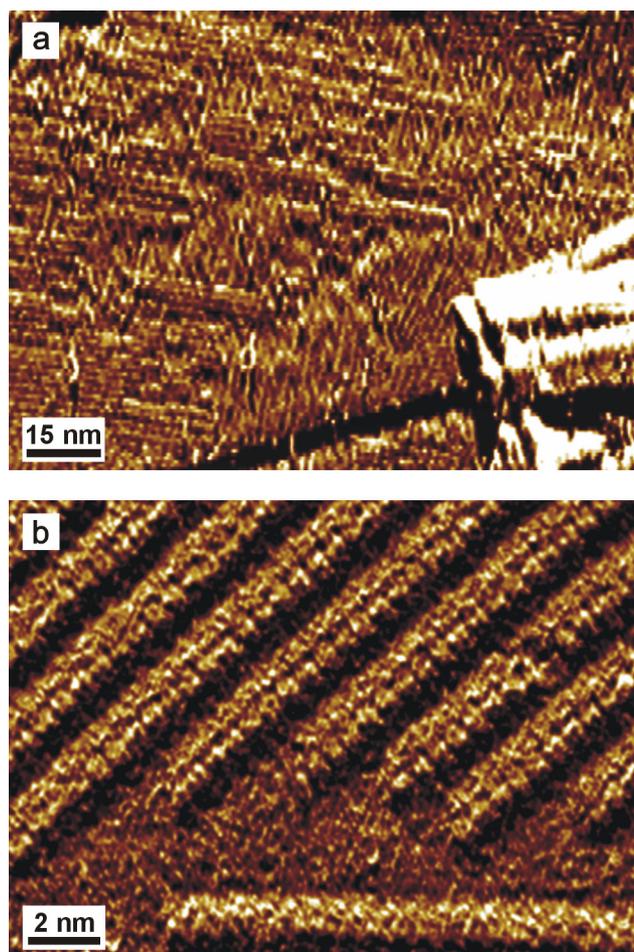


Figure 2. Self-assembly of QAC molecules after direct deposition, i.e. without a binder, of nano-crystalline powder on freshly cleaved HOPG. a) Overview scan, showing both deposited QAC nano-crystals (bright, large feature in the lower right corner) and adsorbate domains consisting of uniform chains b) Close-up of a domain, revealing the supramolecular chain structure of QAC assemblies.

Yet, what is the mechanism of the observed self-assembly? Based on the experimental findings described above we propose a different process. Single molecules are transferred from the nano-crystal to the substrate, when the nanoparticle is in direct contact with the surface. Adsorbed monomers diffuse on the surface and are able to form nuclei for subsequent growth. The main difference between the proposed mechanism and a standard growth process of interfacial monolayers is that building blocks are supplied from a

solid rather than solution or gas. Thus we entitle the proposed process “solid-solid wetting initiated self-assembly”.

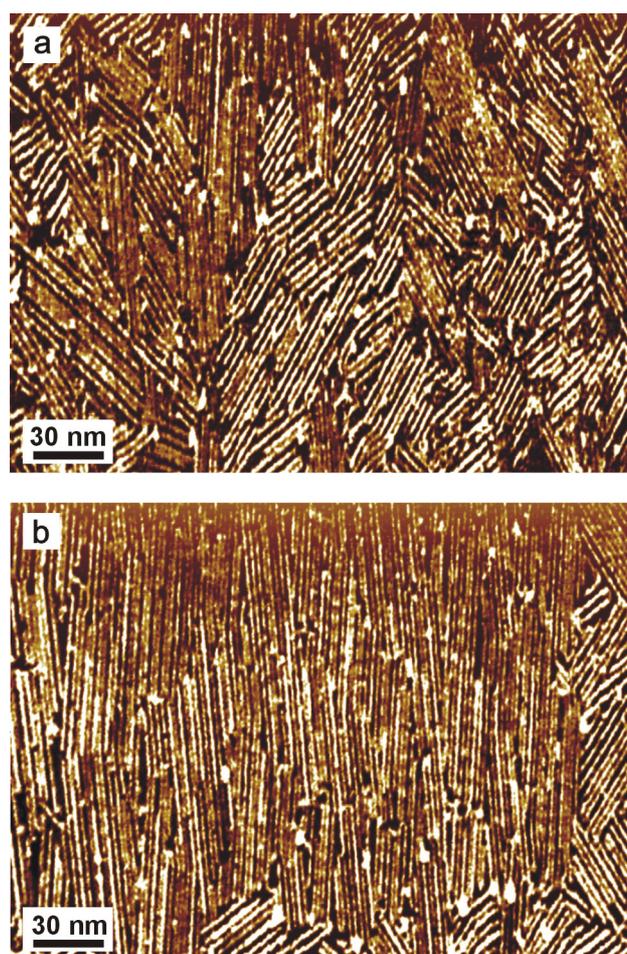


Figure 3. Locally guided growth of QAC chains. a) STM image of the initial configuration. b) Image of the resulting configuration. The alignment was generated by scanning with a decreased tip-sample distance for molecular extraction, and then scanned with an increased distance to enable guided reassembly. The scan area in b) was moved down and to the right by 30 nm relative to a) in order to make the boundaries between non-aligned and aligned areas visible.

In order to reveal the driving force for this disintegration of the nano-crystal we compared the binding energies of single QAC molecules on graphite with surface molecules of a QAC nano-crystal. Force field calculations for gamma-QAC indicate that the binding energy of an adsorbed molecule on HOPG (1.91 eV) exceeds the binding energy of a molecule within the (010) surface of the nano-crystal (1.77 eV). Hence, purely based on energetic grounds, QAC should exhibit a preference for the graphite substrate. On the other hand, for the organic pigment Indigo the relative magnitude of the binding energies as calculated by force-field methods is reversed and no adsorption was observed with the same preparation method, despite numerous attempts. The adsorption energy of a single Indigo molecule on HOPG accounts to 1.56 eV, whereas the lowest binding energy in the solid state was found for the (010) surface with 1.78 eV. Other pigments which we were able to adsorb from a nano-particle suspension, such as Acridone or PTCDA (perylene-tetracarboxylic-dianhydride) show energy relations comparable to QAC. It is well known that force field calculations are a simplification and neglect many important

interactions, hence absolute energy values are not trustworthy. However, here we calculate binding energies for very similar situations where Van der Waals forces dominate. Hence a relative comparison of binding energies is justified and at least provides qualitative information. Basically, the condition for solid-solid wetting is analogous to the classical case of a liquid spreading on a solid, i.e. the adhesion energy between the surface molecules of the droplet and the substrate surface must exceed the cohesion energy between the molecules of the droplet. So far the occurrence of "solid-solid wetting"[24] has only been reported in the special context of inorganic oxide catalysts in a high temperature regime, [24,25] not for organic molecules.

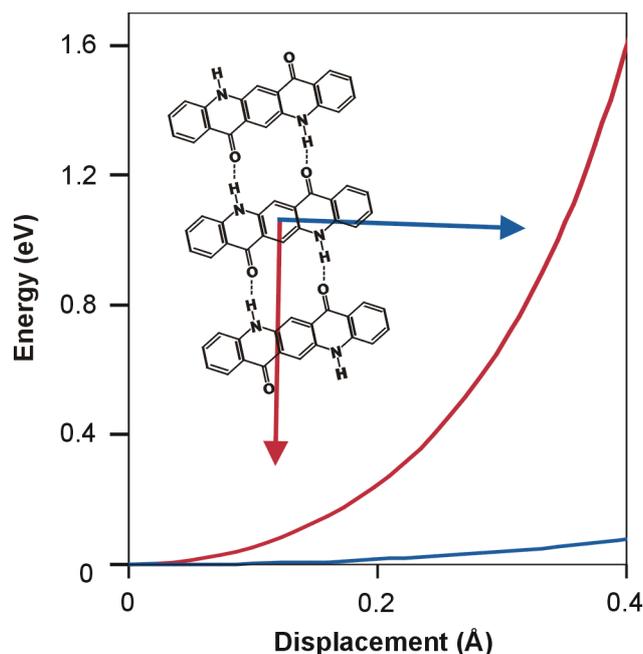


Figure 4. Displacement energies of a single QAC molecule within a wire for the direction parallel (red line) and perpendicular (blue line) to the H-bonds as calculated by force-field simulations. The exact directions of the displacement are indicated in the inset.

In order to verify that the 8CB matrix does not trigger the adsorption, the experiment was repeated without any binder for the nano-crystals. After direct deposition of QAC nano-crystal powder on a freshly cleaved HOPG surface and subsequent removal of the nano-crystals, supramolecular chain structures can be detected as shown in Figure 2b. Since graphite is hydrophobic and a solubility of QAC in water is not detectable even at elevated temperatures,[26] it is very unlikely that the under ambient conditions unavoidable interfacial water layer mediates the adsorption. This is supported by the fact that QAC monolayers have not been detected in areas between deposited nano-crystals before their removal from the substrate surface. We conclude that the transfer of molecules from the nano-crystals to the substrate is restricted to the contact area. In a similar experiment of direct deposition and subsequent removal of the nano-crystalline powder with Indigo, no adsorption could be detected. When QAC monolayers were prepared with the aid of a binder, no nano-crystals were imaged with the STM as in the case of Figure 2a. This indicates that the binder re-suspends nano-crystals after their contact with the substrate. Thus the area where transfer of molecules from the nano-crystal to the substrate has occurred is uncovered and accessible for STM imaging.

Arrangements of QAC nano-wires, self-assembled under the conditions of organic solid-solid wetting, can be controlled locally on the nanoscale, as depicted in Figure 3. When the tip-sample gap is reduced and tip-sample interactions are increased,[27] the nano-wires within the scan area can be extracted, i.e. the QAC molecules desorb from the surface. Re-adsorption and assembly are initiated simply by restoring the previous scanning conditions. In contrast to the initial, randomly oriented domains, the newly self-assembled wires are aligned nearly parallel to the fast scan direction as shown in Figure 3b. Evidently, the orientation of the wires is still determined by the epitaxial relation to the substrate, rather than just the scan direction. Two slightly different directions are discernible in Figure 3b. The angle between those directions accounts to 9° and the two orientations are bisected by a high symmetry direction of the substrate.

In order to understand the reason of this oriented reassembly we calculated the energy cost of displacing a QAC molecule within a chain for two perpendicular directions by using force-field methods. The results, presented in Figure 4 reveal a highly anisotropic behaviour: displacement along the H-bond axis, i.e. compression and extension, requires considerably more force than shearing. This indicates that wire-nuclei with the H-bond direction aligned nearly parallel to the scan direction are more resistant against disruption by the scanning probe, thus can continue to grow, whilst non-parallel nuclei are extracted and replaced. We therefore propose that - in contrast to STM tip induced mechanical reorientations of already adsorbed wires - the rearrangement is the result of re-adsorption and direction selective growth of nano-wires, hence can be described as a locally guided supramolecular self-assembly. Since the growth is actually influenced by the probe, its time constant must be in the same order as the scanning process. Monomers for the re-adsorption are again supplied by the nano-crystals, which are still present or molecules might originate directly from the tip, a situation reminiscent of dip-pen lithography.

The effect of guided self-assembly was also observed on smaller length scales down to the regime of ~10 nm as illustrated by the series of subsequent STM images shown in Figure 5a-c. The initial defect was generated by moving the STM tip in 0.1 s along the path indicated by the dashed line with reduced gap resistance. The manipulation was done in the constant current mode, i.e. the feedback remained active, and the tip-sample distance was decreased by ~0.5 nm with respect to the imaging mode. After the extraction of the molecules the area was repeatedly scanned under normal tunnelling conditions. Likewise, the result of this manipulation can be described as guided self-assembly: Figure 5b shows that the STM tip generates a gap in the nano-wire assembly along the manipulation path. The fuzzy features within the gap can be explained by molecules which are still mobile and in the process of diffusion and self-assembly during the scan. The orientation of the eventually re-grown and stabilised self-assembled wires is clearly related to the scanning direction (Figure 5c).

## Conclusion

It has been shown that polyaromatic molecules can be transferred from a nano-crystal, which serves as a precursor, to a solid substrate just by establishing mechanical contact. Molecules deposited that way then form self-assembled supramolecular structures according to their structure and functional groups for H-bonding. The method was demonstrated by means of QAC on graphite, a system which

also offers the opportunity of re-arrangement by guiding the self-assembly process through interaction with the scanning probe.

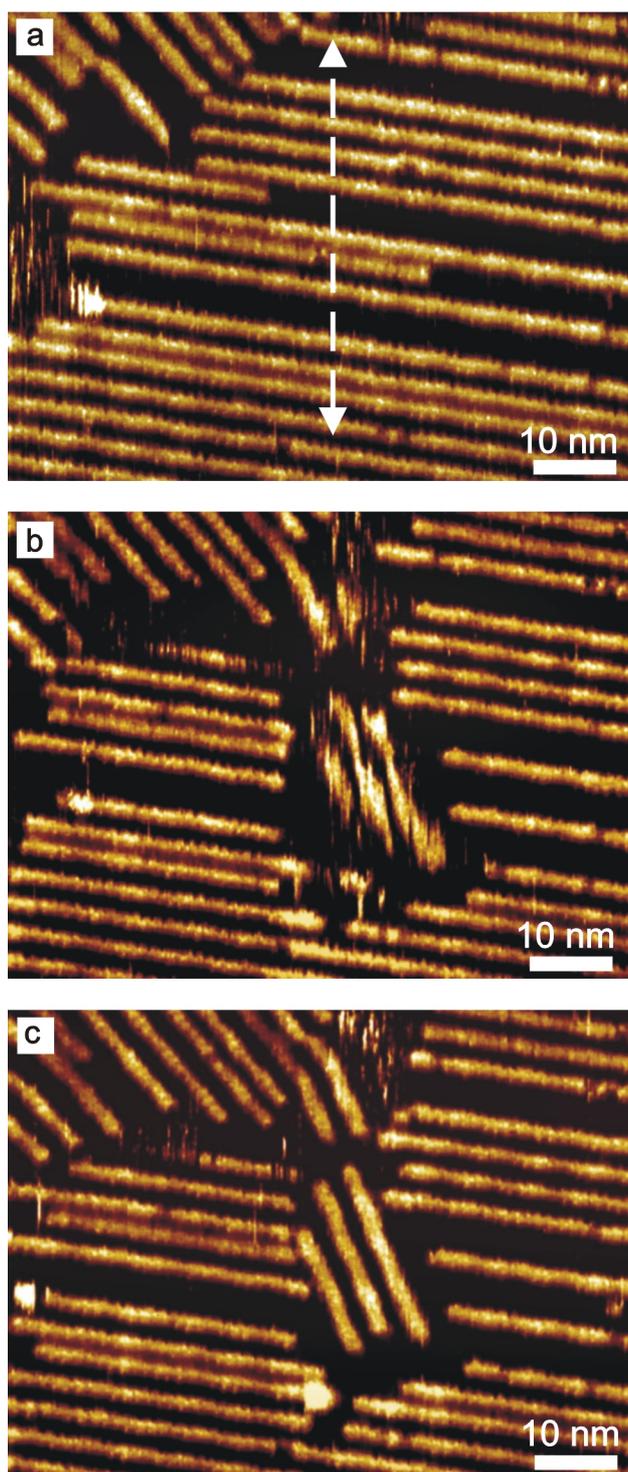


Figure 5. Nanoscale reconfiguration by tip influenced re-assembly. a) STM image of the initial array. After this image was recorded, the scan was stopped and the STM tip moved with a reduced tip-sample distance 6 times along a vertical vector (dashed line) with a frequency of 53 Hz. b) The process of re-assembly within the produced monolayer gap. c) Re-established new wires within the gap.

Although some restrictions apply for the solid-solid wetting to occur, namely, the adsorption energy of the molecule on the substrate has to exceed the binding energy on the surface of the

initial nano-crystals, it is still applicable to a wide range of interesting compounds. In addition, the ease of preparation renders this novel extremely economic preparation method highly interesting for future applications. The fact that computationally inexpensive force-field simulations appear to predict the applicability of the method for a given system correctly, certainly helps to pave the road for a new preparation technique of self-assembled molecular monolayers.

## Experimental Section

**Preparation:** Commercially available QAC powder[19] was used for all preparations. QAC monolayers were generated by direct deposition of nanoparticle powder on the (0001) surface of highly oriented pyrolytic graphite (HOPG) and a subsequent removal of the powder from the HOPG surface (Figure 2a, b). Monolayers were also generated by mixing up QAC powder with the liquid crystal Octyloxybiphenyl (8CB)[22] in molar ratios of QAC : 8CB ranging from 1:3 to 1:20 (Figures 1,3,5). Various mixing ratios, however, had no measurable influence on the reported results. As discussed above, 8CB does not dissolve QAC, but merely serves as a matrix (binder) and carrier for the suspended QAC nano-crystals. In order to verify that no specific properties of 8CB account for the observed effect, experiments were performed in various other liquids as alkyd resins and vitamin K1 with similar outcome. The spontaneous self-assembly of QAC nanostructures at the surface of freshly cleaved highly oriented pyrolytic graphite (HOPG) was initiated by applying a small amount of the mixture to the (0001) surface of HOPG.

**STM:** The supramolecular assemblies were investigated in-situ with a home-built Scanning Tunnelling Microscope (STM) under ambient conditions at room temperature, i.e. without removing the excessive 8CB / QAC mixture. Electrochemically etched tungsten tips served as probes. STM measurements were performed in the constant current mode of operation with a typical line frequency of 14 Hz, tunnelling currents  $\sim 0.5$  nA and voltages  $\sim +1.0$  V (with respect to the tip). In order to perform molecular extractions the tip-sample gap was decreased by  $\sim 0.5$  nm with respect to the imaging mode by reducing the tunnelling resistance (changing the tunnelling voltage from  $+1.0$  V to  $+0.1$  V).

**Simulations:** For a profound understanding of the intermolecular QAC hydrogen-bonds and in order to explain the anisotropy in growth and properties, force-field calculations were conducted. Geometrical constraints were derived from STM measurements and applied to these simulations in order to estimate the binding energies of various polyaromatics in their crystalline state and on graphite respectively. The calculations were performed with the Cerius2 software package employing a Dreiding II force-field, [28] which contains an explicit term for hydrogen-bonding. Density functional theory (DFT) calculations within the generalised gradient approximation[29] were performed with the Vienna ab initio simulation package (VASP).[30] The DFT simulated STM image was calculated within the Tersoff-Hamann picture.[31]

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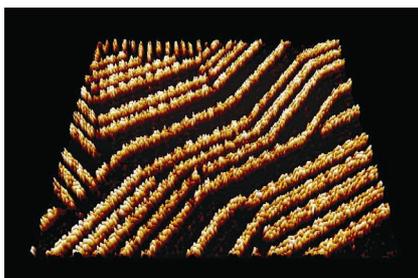
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## Wetting with organic solids

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### **Supramolecular self-assembly initiated by solid-solid wetting**



Self-assembled monolayers of virtually insoluble organic semiconductors / pigments can be obtained by a process applicable under ambient conditions which we refer as organic solid-solid wetting deposition. The process is exemplified with the molecule Quinacridone. Locally guided self-assembly of this supramolecular building block is demonstrated.