Ultra-high Yield On-surface Synthesis and Assembly of Circumcoronene into Chiral Electronic Kagome-honeycomb Lattice

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On-surface synthesis has revealed remarkable potential in the fabrication of a plethora of elusive nanographenes with tailored structural, electronic and magnetic properties unattainable by conventional wet-chemistry synthesis. Unfortunately, surface-assisted synthesis often involves multiple-step cascade reactions with competing pathways, leading to the formation of a diversity of products with limited yield, which reduces its feasibility towards the large-scale production for future technological applications. Here, we devise a new on-surface synthetic strategy for the ultra-high yield synthesis of a hexagonal nanographene with six zigzag edges, namely circumcoronene on Cu(111) via surface-assisted intramolecular dehydrogenation of the rationally-designed precursor molecule, followed by methyl radical-radical coupling and aromatization. An elegant electrostatic interaction between circumcoronene and Cu(111) drives their self-organization into an extended superlattice, as revealed by bond-resolved low-temperature scanning probe microscopy and spectroscopy measurements. Density functional theory and tight-binding calculations reveal that unique hexagonal zigzag topology of circumcoronenes, along with their periodic electrostatic landscape confines two-dimensional (2D) electron gas in Cu(111) surface into chiral electronic Kagome-honeycomb lattice with two emergent electronic flat bands. Our findings open up a new route for the high-yield fabrication of elusive nanographenes with zigzag topologies and their novel 2D superlattices with possible non-trivial electronic properties towards their future technological applications.
On-surface synthesis has emerged as a powerful strategy for the bottom-up fabrication of low-dimensional nanographenes (NGs) with atomic precision\[1-5\]. The core concept of the surface-assisted synthetic strategy relies on the rational design of the elegant molecular precursors, which subsequently undergo chemical transformation along certain reaction pathway towards desired NG product. The versatility of this synthetic strategy has been exemplified by the seminal fabrication of a wealth of open-shell and closed-shell NGs. In addition, the electronic, magnetic and optical properties of NGs can be precisely tuned \textit{via} engineering their chemical structure and/or concomitant $\pi$-electron topology of the NG\[6-14\]. Therefore, on-surface fabricated NGs with tailored physicochemical properties represent highly prosperous functional materials for next-generation quantum device applications\[15-18\].

However, on-surface synthetic strategies have long been constrained by a low scalability due to a rather limited yield of target NGs. Conventional surface-assisted synthetic routes often involve a series of cascade reactions with competing reaction pathways, which inevitably lead to the formation of numerous undesired products. The limited yield of target products imposes a formidable challenge towards the practical applications of the NGs synthesized on the surface, since feasibility of the large-scale (i.e. high-yield) fabrication is vital for their technological relevance. To overcome this issue, it is required to devise new synthetic route with new conceptual design of precursor molecules, which allow for their robust transformation towards target NGs \textit{via} suppressing the side reactions, including undesired intermolecular coupling and intramolecular transformations.

To this end, we design a novel surface-assisted synthetic route for the ultra-high yield fabrication of a hexagonal zigzag-edged NG, namely circumcoronene, on Cu(111) surface. Our surface-assisted cyclodehydrogenation strategy relies on the robust dehydrogenative coupling of
the methyl groups at the adjacent sites of rationally-designed precursor molecule II, followed by dehydrogenative aromatization on metallic substrate (Fig. 1a). Due to a remarkably high yield synthesis and elegant molecule-substrate interaction, circumcoronene molecules self-assemble into extended superlattices (Fig. 1d). The structural and electronic properties of both individual molecules and their superlattices are characterized via bond-resolved non-contact atomic force microscopy (ncAFM) and scanning tunneling microscopy (STM) / spectroscopy (STS), respectively. We demonstrate that circumcoronene superlattice confines the 2D electron gas on Cu(111) into extended Kagome-honeycomb electronic lattice (Fig. 1d) with two flat-band electronic structure, in contrast to conventional electronic Kagome lattice with a single dispersionless band. Our findings open a new route for both, ultra-high yield synthesis of elusive NGs and precise fabrication of artificial 2D lattices with possible non-trivial electronic properties towards the future technological applications.

**Surface-assisted synthesis of circumcoronene.** To fabricate a circumcoronene, we adopt a strategy of a surface-assisted cyclodehydrogenation of the rationally-designed precursor molecule II (C_{54}H_{42}), which was obtained via multi-step wet-chemistry synthesis (Fig. 1a). Oxidative cyclodehydrogenation of the hexakis(3,5-dimethylphenyl)benzene precursor molecule I by iron(III) chloride gave the hexa-*peri*-hexabenzocoronene (HBC) precursor molecule II carrying twelve methyl groups at the bay regions in nearly quantitative yield. This additional step reduces the rotational degree of freedom of six pairs of methyl groups and locks their positions ready for the subsequent dehydrogenative coupling into circumcoronene with zigzag edged topology on the catalytically-active Cu(111) surface. Such a precursor design prevents the undesired intramolecular and intermolecular side reactions, allowing for high-yield synthesis of
zigzag-edged NGs. Furthermore, the presence of methyl groups in precursor II reduces intermolecular \(\pi-\pi\) interactions within compound and thus facilitates \textit{in situ} sublimation for on-surface synthesis. A complete synthetic route of II along with standard structural characterization is presented in Supplementary Materials (Supplementary note 1).

Precursor II was then deposited onto Cu(111) surface held at room temperature \textit{via} thermal sublimation from Knudsen cell under ultra-high vacuum (UHV) conditions. Upon deposition onto Cu(111) surface, precursor molecules self-assemble into close-packed ordered structures (Fig. 1b). Precursor II adopts a non-planar saddle-shape conformation geometry\cite{19}, which results in the triangular-shaped topography in STM image (inset in Fig. 1b). Subsequently, the transformation of precursor II into target circumcoronene was achieved by thermal annealing at \(\sim 140{^\circ}C\) for one hour, whereby the intramolecular cyclodehydrogenation of the methyl groups can be triggered. The chemical transformation of precursor II into final product involves two major steps, namely the dehydrogenative coupling of the adjacent methyl groups followed by dehydrogenative aromatization on Cu(111) (Supplementary Fig. S5), leading to a remarkably high product yield (> 98%) (Fig. 1c), in stark contrast to the case of precursor I (unsuccessful, Supplementary Fig. S7). Note that the product yield is determined by a statistic analysis of multiple large-area STM images (\(\sim 2000 \text{ nm}^2\)) (refer to Supplementary Fig. S6 for details).

\textbf{Characterization of single circumcoronenes and their superlattice.} A high-yield synthesis allows individual circumcoronene molecules to self-organize into extended superlattices characterized by a \(\sqrt{3} \times \sqrt{3}\) elementary unit cell with a lattice constant of 18.4 Å (Fig. 2a). To determine an accurate intramolecular structure of individual molecules and their superlattices, we conducted a constant-height ncAFM measurement with a carbon-monoxide (CO) terminated
tip\textsuperscript{[20-24]}. The frequency shift ($\Delta f$) contrast revealed by constant-height ncAFM images reflects the magnitude of short-range forces acting between CO-terminated tip in a Pauli repulsion regime. Therefore, bright areas with higher $\Delta f$ value represent regions with an enhanced electron density. A set of ncAFM images collected at various tip-sample heights over individual circumcoronene (Fig. 2b,c) unambiguously reveal the molecular skeleton consisting of 19 fused benzene rings with six zigzag edges. Close-up image reveals that the C-C bonds at molecule’s corners exhibit the shortest length when imaged at small tip-sample separations (Fig. 2c). At a moderate tip-sample distance (Fig. 2b), the corner bonds also show a brighter AFM contrast, suggesting a higher bond order of these C-C bonds as compared to other ones. The calculation of the free-standing circumcoronene based on B3LYP DFT functional unambiguously reveals a localization of the shortest C-C bonds (or bonds with highest order) at the molecular corners (Fig. 2h). Such a peculiar bond localization within circumcoronene can be described based on the Clar’s Aromatic Sextet rule, which predicts the formation of maximal number of aromatic sextets (seven in circumcoronene) with a C=C localization at the molecular corners. It is worth mentioning that ncAFM contrast of peripheral C-C bonds in circumcoronene associated with an increased bond localization of aromatic rings also resembles that of Kekulene molecule\textsuperscript{[25]}.

In order to uncover the atomic registry of the circumcoronene-based superlattice on Cu(111), we imaged both individual molecules within the superlattice and adjacent Cu(111) lattice, respectively (Supplementary Fig. S8). Superimposing the Cu(111) lattice over the circumcoronene array reveals that all the circumcoronene molecules adopt identical adsorption with commensurate registry with respect to the Cu(111) lattice, corroborated by the DFT-predicted model (Fig. 2f). Furthermore, DFT calculations also unveil that circumcoronene adopts a slightly bent geometry, with peripheral benzene rings pulled towards the underlying Cu(111)
surface (Fig. 2g). Such a bent structural conformation of circumcoronene is manifested by a brighter contrast of the central benzene ring in the constant-height ncAFM images. The simulated ncAFM images of DFT-relaxed structures using a modified probe-particle mode\cite{26,27} reproduce all the key features of experimental ncAFM images including a tip-sample dependent ∆f contrast over the corner C-C bonds with the highest bond order and higher ∆f contrast of the central benzene ring attributed to non-planar geometry of the circumcoronene (Fig. 2d,e).

**Electronic properties of the circumcoronene-based superlattice.** We then probed the electronic properties of individual circumcoronene molecules and their superlattice via performing differential conductance spectroscopy (dI/dV) measurements (Fig. 3). In contrast to rather featureless dI/dV spectrum acquired over bare Cu(111) surface (only Shockley surface state induced peak around -0.4 V), the dI/dV curve collected over circumcoronene at the periphery of superlattice (blue curve in Fig. 3a) exhibits three characteristic peaks, located at energies of -1.6 V, +0.9 V and +1.8 V respectively, resembling that acquired over an isolated molecule (green curve in Fig. 3a). Interestingly, the dI/dV curve acquired over the circumcoronene in the interior of the superlattice (red curve in Fig. 3a), reveals additional prominent peaks centered at +0.36 V (labeled as $F B_1$) and +1.15 V (labeled as $F B_2$). We further probed the spatial distribution of these electronic states via dI/dV mapping with a CO-functionalized tip. The dI/dV maps collected at the corresponding energies of -1.6 V, +0.9 V and +1.8 V reveal the characteristic nodal patterns localized over the molecular backbone, which can be assigned to the highest occupied (HOMO), lowest unoccupied (LUMO) and second lowest unoccupied (LUMO+1) molecular orbitals respectively as supported by their excellent resemblance with the calculated dI/dV maps of these frontier orbitals using a CO-decorated
tip\textsuperscript{[28,29]} (inset of Fig. 3c,e,g). In contrast, $dI/dV$ maps collected at energies of the additional electronic states ($FB_1$ and $FB_2$) within the circumcoronene superlattice, reveal remarkable intensity in-between molecules (Fig. 3d,f), which cannot be explicitly ascribed to tunneling into any frontier molecular orbitals. Furthermore, $FB_1$ and $FB_2$ states largely attenuate at the edge of circumcoronene superlattice as evident from the 2D contour plot of $dI/dV$ spectra acquired across the superlattice (Fig. 3b).

**Flat bands in the chiral electronic Kagome-honeycomb lattice.** We performed DFT calculation combined with tight-binding (TB) model calculations to understand the physical origin of both $FB_1$ and $FB_2$ states in the circumcoronene superlattice. These new electronic states arise from the formation of circumcoronene superlattice that confine 2D electron gas (2DEG) of the Cu(111) surface into chiral electronic Kagome-honeycomb lattice. Calculated electrostatic potential landscape over circumcoronene superlattice shows a notable accumulation of the negative charge in the plane of circumcoronene (Fig. 4b). This negative electrostatic potential is balanced by a positive potential accumulated in the Cu(111) surface regions between molecules. In addition, a comparison of the calculated density of states (DOS) plots of the freestanding circumcoronene and Cu(111)-supported circumcoronene, shows that the latter exhibits a downshift of the energetic positions of the molecular orbitals (Supplementary Fig. S9), attributed to the electron transfer from the Cu(111) substrate to circumcoronene. Each molecule hosts a negative electrostatic potential and thereby acts as a hexagonally-shaped repulsive barrier for creating a periodic modulation and confinement of the 2DEG\textsuperscript{[30-32]}. Notably, our DFT calculations also reveal a non-negligible out-of-plane buckling of Cu atoms in the topmost layer of the Cu(111) regions forming a periodic buckling pattern (Fig. 4c) reminiscent to the chiral
Kagome-honeycomb lattice (Fig. 4a). Due to a computational feasibility, the semi-infinite surface Cu(111) surface is represented by only three Cu layers in our DFT calculations, which therefore cannot provide an accurate description of the electronic structures of surface state and the confined states observed experimentally.

The unique electronic structures of artificial electronic lattice can be well described using TB model that involves the electron hopping strength between atomic orbitals associated to lattice sites. TB band structures of a standard Kagome and Kagome-honeycomb lattice are shown in Fig. 4d and 4e, respectively. The common Kagome lattice is composed by a network of corner-sharing triangles, leading to the destructive phase interference of electron hopping pathways between nearest-neighbor lattice sites. As such, it precludes electron hopping between neighboring unit cells and results in the localization of electronic state within single Kagome hexagon\(^{[33-34]}\). Such a unique real-space localization of the electronic state further manifests itself by the formation of the empty state dispersionless band (i.e. flat bands) alongside Dirac bands in a reciprocal space, as revealed by TB band structure derived from hopping parameters of \( t_1 = t_2 = t_3 = -0.5 \, eV \). In contrast, Kagome-honeycomb lattice can be virtually derived from the Kagome lattice by introducing an additional bond between frustrated Kagome’s triangles, associated with additional hopping constant \( t_4 \). The TB band structure of Kagome-honeycomb lattice (Fig. 4e) with hopping constants of \( t_1 = t_2 = t_3 = t_4 = -0.5 \, eV \), exhibits two flat bands below and above Dirac bands, which can be attributed to a pair of the intense peaks (\( FB_1 \) and \( FB_1 \)) in the electronic spectrum of the circumcoronene-based superlattice on Cu(111) (Fig. 4f).

The electronic Kagome-honeycomb lattice emerges as “antilattice” to the circumcoronene superlattice, whereby individual molecule acts as a hexagonal repulsive barrier for 2DEG. The underlying physical picture is remarkably similar to the artificial electronic lattices\(^{[35-41]}\).
patterned by CO molecules on Cu(111) surface via STM manipulation, wherein each CO molecule also acts as a repulsive scatter for 2DEG. Although STM manipulation endows an ultimate control over the lattice topology, this approach is unsuitable for practical applications due to a lack of scalability. Our findings surpass this limitation, paving a viable route towards a scalable bottom-up fabrication of the extended 2D electronic lattices, where NG with tailored topology is employed as a “building block”. We envisage that a rational design of nanographenes with different size and edge topologies can potentially serve as a versatile “tuning knob” for the efficient modulation of the hopping strength and electronic structure of artificially-engineered electronic lattices.

**Conclusions**

We have demonstrated a conceptually new on-surface synthesis route towards the ultra-high yield fabrication of the hexagonal-shaped circumcoronene molecule on Cu(111) via surface-assisted intramolecular cyclodehydrogenation of the rationally-designed precursor molecule. A simultaneous assembly of large-area circumcoronene array results in a periodic confinement of the 2D electron gas into unique chiral electronic Kagome-honeycomb lattice associated with the peculiar electrostatic potential landscape of circumcoronene array on Cu(111). Our results open up a new avenue for high-yield fabrication of atomically-precise elusive nanographenes with desired topologies towards large-scale fabrication of artificial 2D lattices with possible non-trivial electronic properties. 2D Kagome-honeycomb lattices with flat band electronic structure can be particularly interesting in a wide scope of condensed-matter physics, due to their prosperous potential in the realization of a variety of exotic many-body phenomena, including
anomalous quantum Hall states\textsuperscript{42-44}, Wigner crystallization\textsuperscript{45} and topological insulating transitions\textsuperscript{46}.

Methods

The qPlus STM/ncAFM measurements: The STM and ncAFM experiments were performed in UHV conditions at 4.4 K using a commercial Omicron LT STM/AFM machine. We used the commercial qPlus sensor\textsuperscript{47} with a resonant frequency of $f_0 = 28$ kHz, a stiffness of $k = 1800$ N m$^{-1}$ and quality factor 11500, which was operated in the frequency-modulation mode with an oscillation amplitude of $A = 100$ pm. All simultaneous STM/ncAFM images were collected in constant height mode. The tip apex was routinely decorated by a CO molecule by its controllable picking from the bare Cu(111) surface.

Sample preparation: Cu(111) single crystal (MaTeck GmbH) was cleaned by multiple cycles of Ar$^+$ sputtering and annealing. The precursor molecules were deposited from Knudsen cell (MBE-Komponenten GmbH) at 350 °C onto clean Cu(111) surface under ultrahigh vacuum conditions (base pressure $< 3 \times 10^{-9}$ mbar). After the deposition of the precursor molecules, the sample was annealed at 140 °C for 1 hour for the fabrication of circumcoronene arrays. Subsequently, the sample was transferred into the STM/AFM head held at temperature 4.5 K for ncAFM/STM imaging and characterization.

DFT simulations: Density Functional Theory calculations were performed using the FHI-AIMS code\textsuperscript{48} and GGA-PBE approximation of the exchange-correlation potential\textsuperscript{49,50} in order to obtain the electronic properties of the circumcoronene molecule on Cu(111) surface. The
Tkachenko-Scheffler treatment of the Van der Waals (vdW) interactions was used during the calculations\cite{51}. We modeled the Cu(111) surface by a 3×3 supercell composed of three layers. After placing the molecule on the surface, we performed structural relaxations of all the atoms of the slab, except the last Cu layer. The optimization procedure was stopped when the total energy and the remaining atomic forces were found below $10^{-5}$ eV and $10^{-2}$ eV/Å, respectively. A Monkhorst-Pack grid of 3×3×1 was used to integrate the Brillouin zone. Furthermore, the calculated total charge density and the Hartree potential were used to analyze the electronic interactions between the circumcoronene and the surface.

**Tight-binding calculations:** We carried out calculations of a tight-binding (TB) model using the nearest neighbour interaction between nodes of Kagome-honeycomb lattice shown in Fig. 4e. All on-site energies have been set to zero and the nearest neighbor hopping was set to $t=-0.5$ eV to mimic the experimental evidence. To obtain band plot shown in Fig. 4e, TB Hamiltonian was diagonalized along a dense set of high-symmetry k-points.

**SPM simulations:** AFM image simulations were performed based the Probe Particle code\cite{26,27}, that includes vdW and electrostatic interactions between the CO tip and the surface. Lennard-Jones potential was used for the treatment of Pauli repulsion and vdW interactions. The Hartree potential obtained from DFT calculations was used to calculate the electrostatic forces. All the simulations were done with an effective atomic radius $R_C = 1.661$ Å and a lateral stiffness of $k=0.25$ N/m. $dI/dV$ maps were simulated using the PP-STM code\cite{28} with an CO rigid tip (without tip-apex relaxation). We first calculated the eigenstates of the circumcoronene orbitals (with the FHI-AIMS code) which were subsequently used in the simulations. The $dI/dV$ maps were obtained at characteristic energy positions corresponding to frontier orbitals (-1.6 V, +0.9 V and +1.8V). Note that the Lorentzian width parameter for broadening of the eigenstates was set to be 0.001 eV.
Data Availability: The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Author contributions: J.L. supervised the project. M.T., performed experiments related to on-surface synthesis, STM/ncAFM measurements and data analysis. G.L. and J.W. designed and synthesized the precursor molecules. P.M. and P.J. performed the DFT calculations. D.S.-P. and P.J. performed tight-binding calculations. X.P., J.S., S.S. and M. J. K. contributed to the scientific discussion. The manuscript was written by M.T. and L.J., with contributions of all co-authors.

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References


**Fig. 1** On-surface synthesis of circumcoronene-based superlattice. 

**a**, Precursor molecule II undergoes cyclodehydrogenation on Cu(111) via methyl radical-radical coupling towards formation of the circumcoronene. The cyclodehydrogenation results in ultra-high yield circumcoronene molecules. **b**, STM image acquired after deposition of the precursor II onto Cu(111) at room temperature. Inset shows STM image of individual precursor molecule. **c**, STM image collected after sample annealing at 140 °C, revealing presence of the circumcoronene-based self-assembly on Cu(111). **d**, Schematic illustration of the chiral Kagome-honeycomb lattice.
**Fig. 2** | **Bond-resolved ncAFM imaging of circumcoronene superlattice.**

a, Large-scale ncAFM image of the circumcoronene array, collected with CO-decorated tip. Defective circumcoronene molecule (indicated by yellow arrow) constitutes an approximately 2% of total products. b-e, Constant-height ncAFM images of individual circumcoronene molecule, collected at tip sample distances $z=40$ pm in (b), $z=60$ pm in (c), with respect to setpoint $V=20$ mV; $I=1$ nA. d-e, ncAFM images simulated using numerical probe-particle approach, for physical tip-sample distance of 0 pm in (e), 20 pm in (d) with respect to setpoint 14.4 Å. f, DFT-relaxed model of circumcoronene-based superlattice on Cu(111). g, Top and side view of single circumcoronene molecule on Cu(111). h, DFT (B3LYP) calculations of the bond lengths within circumcoronene molecule.
**Fig. 3** Electronic structures of the circumcoronene array. **a**, $dI/dV$ spectra collected over bare Cu(111) substrate (grey), circumcoronene molecule residing at periphery of superlattice (blue), circumcoronene molecule located in the interior of superlattice (red) and isolated molecule (green). **b**, Two-dimensional contour plot of $dI/dV$ spectra acquired along line indicated in inset panel in **a**. **c-g**, Topography STM image (1 V; 1 nA) and corresponding $dI/dV$ maps collected at energetic positions of states HOMO (**c**), $FB_1$ (**d**), LUMO (**e**), $FB_2$ (**f**) and LUMO+1 (**g**). Inset images in panels (**c**), (**e**) and (**g**) are the calculated $dI/dV$ maps of the HOMO (**c**), LUMO (**e**) and LUMO+1 (**g**) of circumcoronene using a CO-decorated tip.
Fig. 4 | Origin and electronic structure of Chiral Kagome-honeycomb lattice. a, Schematic illustration of the chiral Kagome-honeycomb lattice. b, Electrostatic potential of the circumcoronene superlattice. c, The out-of-plane buckling of Cu atoms in topmost Cu(111) layer. d-e, A $k$-space dispersion of the electronic bands of the Kagome and chiral Kagome-honeycomb lattices, calculated using TB model for hopping constants $t_1 = t_2 = t_3 = -0.5 \text{ eV}$ (d) and $t_1 = t_2 = t_3 = t_4 = -0.5 \text{ eV}$ (e). f, Experimental $dl/dV$ spectra reveal the existence of two characteristic peaks ($FB_1$ and $FB_2$) associated to two flat bands of the chiral electronic Kagome-honeycomb lattice.