Deterministic Thermal Sculpting of Large-Scale 2D Semiconductor Nanocircuits

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2D transition metal dichalcogenide semiconductor (TMDs) nanocircuits are deterministically engineered over large-scale substrates. This original additive nanolithography approach combines large-area physical growth of 2D TMDs layer with high resolution thermal-scanning probe lithography, to reshape the ultra-thin semiconducting layers at the nanoscale level. The additive nanofabrication of few-layer MoS₂ nanostructures of controlled thickness, grown in the 2H-semiconducting phase, is demonstrated as shown by their Raman vibrational fingerprints and by their optoelectronic response. The electronic signatures of the MoS₂ nanostructures are locally identified by Kelvin probe force microscopy providing chemical and compositional contrast at the nanometer scale. Finally, the potential role of the 2D TMDs nanocircuits as building blocks of deterministic 2D semiconducting interconnections is demonstrated by high-resolution local conductivity maps showing the competitive transport properties of these large-area nanolayers. This work thus provides a powerful approach to scalable nanofabrication of 2D nano-interconnects and van der Waals heterostructures, and to their integration in real-world ultra-compact electronic and photonic nanodevices.

1. Introduction

Emerging 2D materials belonging to the class of transition metal dichalcogenide semiconductors (TMDs) have recently gained a broad interest by the scientific community offering new routes to nanoscience and nanotechnology.[1-12] Thanks to their exceptional optoelectronic response and tunable bandgap in the visible and near-infrared spectrum, combined with the atomically thin structure, functional properties can be obtained with a strong impact in various fields ranging from nanoelectronics and nanophotonics, to energy conversion and quantum technologies.[13-26] In this context the possibility to achieve a controlled reshaping of TMDs layers is particularly attracting in order to develop engineered and/or quantum confined 2D materials as a building block for functional nanodevices in electronics, photonics, and quantum technologies.[6,27-33] Recently the possibility to promote tuning of the optoelectronic response via shape engineering of 2D layers has been demonstrated using micrometric TMDs flakes,[34-36] highlighting the impact of this approach in photonics and quantum optics. However few attempts can be found on the arbitrary nanolithography of 2D TMDs layers on large-scale wafers, typically applied to isolated micro-flakes,[32,37-38] or characterized by spatial resolution at the micro-scale.[39] So far, the most diffuse technique for 2D TMDs materials preparation has been mechanical exfoliation of crystals that provides randomly distributed 2D flakes, endowed with a size typically limited at the micro-scale. The urgent demand for potentially scalable platform and devices has recently motivated alternative growth methods for TMDs layers mainly relying on the chemical vapor deposition approach[40-47] which leads to a random distribution of triangularly shaped 2D TMDs islands typically sized on a micrometer scale. Complex multi-step lithography processes, requiring chemical or plasma etching of the 2D islands (subtractive approaches), are typically used for nanopatterning 2D TMDs, introducing unwanted contaminations or surface damages to the fragile ultra-thin layers.[48,49] Scalable growth and non-invasive nanopatterning approaches are thus urgently required to engineer the optoelectronic and photonic response of 2D TMDs layers.

The thermal-scanning probe lithography (t-SPL) has recently emerged as a very promising technique, uniquely providing local modification of materials properties in ambient conditions with nanoscale spatial resolution provided by a sharp conductive probe.[50-52] This approach is optimal for the fragile 2D layers as demonstrated by few recent experiments showing the nanolithography of high quality metallic contacts on 2D TMDs,[47] and their thermomechanical reshaping when exfoliated as micrometric flakes randomly distributed on the surface.[31,54] Scalable growth and non-invasive nanopatterning approaches are thus urgently required to engineer the optoelectronic and photonic response of 2D TMDs layers.

In this work, we demonstrate a new additive approach enabling the scalable growth of ultra-thin 2D TMDs layer and their direct and high-resolution nano-sculpting via t-SPL. The
nanolithography of ultra-thin MoS₂ nanocircuits deterministically located onto a large-scale wafer is demonstrated by combining the t-SPL of sacrificial polymeric films, with large area ion beam-assisted physical deposition of few-layer TMDs films. By exploiting the t-SPL-defined polymer nanomask, it is possible to additively grow few-layer MoS₂ films, easily controlling the thickness of the final MoS₂ nanocircuits from a few layers up to the bulk regime (details in Figure S1, Supporting Information). The Raman micro-spectroscopy maps have shown the characteristic vibrational response of the 2D semiconductor nanopaths, spatially engineered thanks to the t-SPL-based method. The non-invasive t-SPL has been further exploited to precisely align simple nanodevices based on high-quality metallic contacts onto the 2D TMDs nanopaths, uniquely preserving their electronic response. The capability to control the electronic transport properties at nanometer lateral scale is demonstrated via high-resolution Kelvin probe nanoscopy and local probing of the electric transport via conductive-atomic force microscopy (AFM) nanoscopy. The local electrical and compositional contrasts and the electrical conductivity, resolved at the nanometer scale on the MoS₂ nanopaths, qualify them as building blocks of next generation nanocircuitry. The exceptional uniformity of the 2D TMDs layers over large-area (cm²), combined with the non-invasive t-SPL nanolithography enables the precise nanofabrication of ultra-thin nanocircuits and van der Waals heterostructures nanodevices over large-scale wafers, opening new perspectives in electronics, photonics, and quantum technologies.

2. Results and Discussion

The homogeneous growth of ultra-thin MoS₂ layers over large-scale is demonstrated exploiting a new ion-beam-assisted approach. Controlled deposition of ultra-thin semiconductor films is achieved via collimated ion beam sputtering of a stoichiometric MoS₂ target, faced to the substrate (e.g., silica and silicon). Under this configuration large-area homogeneous films can be achieved on areas exceeding several cm², as highlighted by the sample picture of Figure 1a, which shows a few-layer MoS₂ film (thickness ≈ 5.5 nm) deposited on a transparent silica substrate. The deposition process of the pristine amorphous MoS₂ films takes place at room temperature and is thus compatible with flexible polymer substrates. Recrystallization of MoS₂ films can be obtained via high temperature (750 °C) annealing in a tubular furnace, in presence of sulfur background pressure to avoid altering MoS₂ stoichiometry. The structural quality of the ultra-thin TMD film is confirmed by the Raman micro-spectra (Figure 1b) characterized by the E₁²g and by the A₁g mode, resonant at 383 and 408 cm⁻¹, respectively, as expected for an ultra-thin MoS₂ layer.[55] Remarkably the Raman response, detected over sub-micrometric optical spots, is homogeneous up to the cm scale, as demonstrated by the spectra acquired along a diagonal axis of the sample, 5–6 mm apart one from the other (red spots in Figure 1a). The presence of a 2H-semiconducting MoS₂ ultra-thin film is further confirmed by the optoelectronic response detected in far-field extinction spectroscopy (Figure S1, Supporting Information), and characterized by the A and B exciton resonances.[56]

Figure 1. a) Picture of a large-scale ultra-thin MoS₂ layer grown onto a transparent silica substrate, and b) micro-Raman spectra detected in different spots of the samples shifted several mm apart from one another (region A, B, C in the picture). c–f) Sketch of the t-SPL nanolithography process. g) Large-scale optical microscopy showing a series of MoS₂ nanocircuits fabricated onto a Si/SiO₂ substrate. h) SEM image of a few-layer MoS₂ nanocircuit (darker regions in the secondary electron signal) showing the logo of our laboratory (DIFILab facility – UNIGE) at the nanoscale, and zoom on a detail of the MoS₂ nanopaths. i) AFM image of a high resolution nanocircuit, showing ultra-thin MoS₂ nanopaths (Scale bar = 200 nm).
The large-scale homogeneity of these layers, combined with the novel t-SPL technique, provides the unique opportunity to develop a scalable approach for the nanoscale-resolved reshaping of ultra-thin TMDs layers. The t-SPL indeed allows to pattern arbitrary nanopaths onto a sacrificial thin polymeric bilayer (see Experimental Section for further details) by exploiting a sharp hot nanoprobe (sketch in Figure 1c). In this way engineered nanopatterns can be written at high resolution (tip radius is approximately few tens of nm) on the soft layer, which selectively exposes the substrate after the development process (Figure 1d). The sacrificial bilayer acts as a nanopatterned mask with appropriate negative angle profile for the following few-layer MoS2 growth (Figure 1e), thus avoiding any change of the few-layer material structures (e.g., oxidation) induced by the lithography process. The final lift-off of the polymer film allows to achieve engineered few-layer MoS2 nanopaths, as sketched in Figure 1f, characterized by the same structure of the corresponding large-area film.

The large-scale optical microscopy image of Figure 1g demonstrates the scalability of the method, enabling the additive nanofabrication of few-layer MoS2 nanocircuits (darker regions of the image) in arbitrarily defined positions over large-scale, achieving uniform condition up to the cm² scale. The zoomed scanning electron microscopy (SEM) image on a specific nano-layout) in arbitrarily defined positions over large-scale, nanofabrication of few-layer MoS2 nanocircuits (darker regions of the image) forming the logo of our facility and obtained thanks to the t-SPL nanolithography. The zoomed-in SEM image well highlights the contrast of the few-layer MoS2 on the substrate with nm spatial resolution. Further MoS2 nanopatterns designed at higher resolution are shown in the AFM image of Figure 1i, which evidences nanostripe widths below 200 nm. In particular, we achieve best lateral width down to 130 nm, and minimum inter-spacing of about 135 nm. The characteristic thickness of these MoS2 nanopatterns reads (5.5 ± 0.5) nm (= 8 –9 layers), determined by the statistical analysis of different AFM topographies (further details in Figure S2, Supporting Information). Remarkably, the additive nanolithography approach here described enables fabrication of clean ultra-thin MoS2 nanostructures, avoiding damaging and/or contamination of the 2D TMDs layer intrinsic in the reactive ion etching-based approach, typically used for nanopatterning 2D materials and enabling the accurate control and tailoring of the nanostructure thickness.

To confirm the material structure and to show the reshaping capabilities of the few-layer material, Raman micro-spectra have been measured both on the engineered nanopaths (red curve in Figure 2b) corresponding to blue regions of the optical microscopy image of Figure 2a) and few hundred nanometers apart on the bare substrate (black curve in Figure 2b). The Raman micro-spectrum detected on the MoS2 nanopaths shows the characteristic E1g and A1g vibrational modes excited at 383 and 408 cm⁻¹, respectively, while on silica substrate (black curve) we measure an unstructured background.

In parallel, the high homogeneity of the few-layers MoS2 nanopaths is demonstrated by the micro-Raman maps shown in Figures 2c and 2d, respectively, corresponding to the Raman image of a whole nano-logo and of a zoomed detail highlighted in Figure 2a. These Raman maps, obtained at an excitation wavelength of 532 nm, recover the morphology of the few-layer MoS2 nanostructures, with a spatial resolution in the range of few hundred nm’s which is only limited by optical diffraction (Figure 2d).

In order to reveal the spatial arrangement of the MoS2 nanostructure, overcoming the optical diffraction limit, and to investigate the local electrical properties, Kelvin probe force microscopy (KPFM) was employed. Such analysis was carried out by using a Pt-coated conductive tip operating in single pass configuration, in order to extract contact potential difference (CPD) maps of MoS2 nanopaths lying on a Si/SiO2 substrate. We define the CPD referred to SiO2 work function as:

$$\Delta \phi = \frac{1}{e} (\phi_{SiO2} - \phi_{MOS2})$$

where e is the elementary electron charge, $\phi_{SiO2}$ is the silica work function and $\phi_{MOS2}$ is the work function of the surface underneath the tip during the scan. Figure 3a shows the $\Delta \phi$ map obtained on the MoS2 nanopaths, highlighting their strong electrical contrast in terms of surface potential. An example of a $\Delta \phi$ cross-sectional profile acquired across the MoS2 nanotrack (white line in Figure 3a and corresponding profile in Figure 3b) shows a work function difference between MoS2 nanopaths and silica substrate of about 200 meV. This electrical response is uniform over the CPD map, highlighting the MoS2 to silica contrast with spatial resolution in the range of few tens of nm, determined by the Kelvin nanoprobe radius and by lock-in modulation voltage. In particular, the histogram of the $\Delta \phi$ map (Figure 3c) is characterized by two distributions, respectively, centered at 0 and ~200 mV, as confirmed by the fit and by the cross-sectional profile at the MoS2-silica edge. The former peak arises from the silica substrate and the latter from the MoS2 nanopaths. The measured $\Delta \phi$ map characterized by 200 mV contrast at the MoS2-SiO2 interface allows to quantify the work function of the MoS2 nanopaths as $\phi_{MoS2} = 5.25 eV$, calculated considering the value of SiO2 work function ($\approx 5.05 eV$), in good agreement with respect to recent reports.

Figure 2. a) Optical microscopy image of a few-layer MoS2 nanocircuit. b) Micro-Raman spectra detected on the MoS2 nanopaths (red line) and on the neighboring Si region (black line). c,d) Micro-Raman maps detected on the whole MoS2 nanocircuit and onto a detail (highlighted region in [a]), respectively. The maps show the signal detected in the range from 370 to 429 cm⁻¹, corresponding to E1g and A1g modes.
In order to show the potential of the 2D-TMDs nanocircuits as active optoelectronic interconnects, we devise a simple ultra-thin device thanks to the non-invasive t-SPL approach. High quality metallic contact can be precisely aligned onto the nanocircuits by exploiting the peculiar in-situ imaging and real-time nanolithography capabilities (i.e., direct overlay method), avoiding undesired damage and contamination for the fragile layers. In this way, the electronic transport properties of the TMDs layers can be deeply investigated with nanoscale spatial resolution via conductive-AFM nanoscopy. Figure 4 shows an example of 2D-TMDs device based onto a few-layer MoS$_2$ nanofinger. A metallic nanocontact (brighter finger at the bottom of the image) has been precisely aligned onto the few-layer nanofinger, thus enabling the local electric probing of the material with the conductive-AFM nanoprobe (ResiScope technology, sensitive across wide conductivity ranges). This c-AFM approach enabled the high-resolution detection of current and resistance maps onto the MoS$_2$ nanodevice, avoiding damage of the sample and/or the probe. The c-AFM maps were acquired in contact-AFM configuration by applying a DC bias voltage of 0.5 V to the sample (metallic nanoelectrode) with respect to a p-doped single crystal diamond tip.

Figure 4a,b shows the local-current and -resistance map of the MoS$_2$ nanofinger device, detected in real-time with the topography (Figure 4c). A strong electrical contrast is detected at the MoS$_2$-substrate edges within both the current and the resistance map, as highlighted in Figure 4d by a comparison between topography and local current extracted along a horizontal profile (white dashed line in Figure 4a,c). The electrical maps demonstrate the semiconducting behavior of the MoS$_2$ nanocircuit device with nanoscale spatial resolution, precisely corresponding to the AFM topography. The local resistance maps allow estimating a resistivity value of about 5 $\Omega \cdot \text{m}$ for these large-area few-layer MoS$_2$ under the approximation of a long channel device. This result is comparable to state-of-the-art MoS$_2$ layers$^{[33,64,65]}$ and very promising in view of deterministic 2D TMDs nano-interconnects. Furthermore, these few-layer nanofinger configurations highlight the possibility to engineer ever more complex...
few-layer van der Waals semiconducting nanodevices, taking advantage of the non-invasive t-SPL nanolithography for the arbitrary and precise alignment of 2D materials endowed with their original optoelectronic properties.

3. Conclusion

We have shown the additive nanolithography of few-layer MoS2 nanocircuits arbitrarily and precisely engineered onto large-scale wafers, thanks to the non-invasive t-SPL approach combined with the homogeneous growth of few-layer TMDs across several cm². The thermal nanolithography of a sacrificial polymeric layer, combined with the large-scale physical deposition of the few-layer TMDs, uniquely enables the on-demand reshaping of ultra-thin TMDs layers with nanoscale spatial resolution. The so formed MoS2 nanocircuits grow into the 2H-TMD semiconductor phase, as shown by their vibrational fingerprint in Raman micro-spectroscopy, and by their electrical work function in Kelvin probe nanoscopy. High quality few-layer TMDs nanodevices have been further engineered by exploiting the non-invasive t-SPL approach to precisely align metallic nanocontacts onto the MoS2 nanocircuits. This configuration has enabled the local probing of the electronic transport properties of the few-layer MoS2 nanocircuits, resolved via conductive-AFM nanoscopy. The local-conductivity maps highlight the competitive transport properties of these reshaped MoS2 nanodevices as few-layer semiconducting interconnects in ultra-thin devices and components. Remarkably these results highlight the possibility to engineer even more complex van der Waals heterostructure nanodevices by this original t-SPL-based technique, uniquely preserving the optoelectronic properties of the fragile few-layer materials. These reshaped 2D-TMDs nanocircuits thus open new perspectives for the integration of 2D semiconducting layers in scalable next-generation devices with impact in electronics, photonics, renewable energies, and quantum technologies.

4. Experimental Section

Large Area Growth of 2D TMDs: A fused silica (SiO2) substrate was cleaned by ultrasonic sonication both in acetone and isopropyl alcohol and loaded into a custom-made vacuum chamber with pressures of 10⁻¹⁰⁻¹² mbar, faced toward a MoS2 target. The latter was then irradiated by an ECR Plasma Source TPIS (TECTRA), which generated a 1.44 keV Ar+ ion beam (gas purity N5.0) at a pressure of 6.0 × 10⁻⁴ mbar. The ion beam formed a 45° angle with respect to the MoS2 target surface normal. The ion beam irradiation induced the spattering deposition of MoS2 thin films on the SiO2 substrate, while the thickness of the deposited material was monitored by means of a calibrated quartz crystal microbalance.

A single zone tubular furnace was used for the recrystallization process (sulfurization). The sample was placed at the center of the furnace, while a 10 scm flux of inert gas (Ar) was flowed inside the pipe as a carrier gas. A quartz boat with sulfur powder was placed between the origin of the Ar flux and the sample. The furnace was then brought to the temperature of 750 °C with a heating ramp of 20 °C min⁻¹ and maintained at high temperature for a soaking time of 10 min. The heater was then turned off, allowing the cool-down of the sample.

Thermal-Scanning Probe Lithography: The few layer MoS2 nanocircuits were achieved thanks to the t-SPL patterning of a sacrificial bi-layer polymer mask, deposited by spin-coating onto a thermal oxide coated (SiO2; 300 nm) p-doped silicon substrate. The underlying film was a PMMA/MA (thickness 95 nm) while a thermally sensitive film of polyphthalaldehyde (PPA, thickness = 25 nm) was spin-coated on the surface. The new Nanofrazor Scholar setup (Heidelberg Instruments) enabled the high-resolution t-SPL of the sensitive PPA film in ambient condition, thanks to the action of a sharp silicon tip (radius tip was approximately few tens of nm) locally heating the surface. The system was able to provide temperature- and time-controlled heat pulses through the nanoprobe (temperature at the cantilever sensor tunable in the 500–1100 °C range), thus enabling the arbitrary nanolithography of complex shapes and/or local modification of material properties.

In this experiment, the t-SPL nanopatterning was performed with the Nanofrazor Instruments operating in continuous heating mode, and the temperature of the cantilever sensor (so called “writer”) was set to about 900–950 °C, while the scanning rate was in the range of 50 μm s⁻¹. A chemical development of the PMMA/MA film underlying the patterned nanopaths was finally performed in order to expose the patterned areas of the substrate (solution of deionized water in isopropyl alcohol at 5% vol).

The sample with the patterned bi-layer polymer mask was transferred in the UHV system for the physical large-scale MoS2 growth, as for the extended flat films (see “Large area growth of 2D TMDs” section). The sample was finally rinsed in acetone for the lift-off of the non-patterned areas achieving MoS2 nanopaths on the substrate, avoiding damages and contaminations by the lithographic process. Under this condition, high quality MoS2 nanostructures could be easily achieved with the high-temperature recrystallization process.

Metallic nanocontacts based on a Ti/Au thin film (thicknesses 2 nm/20 nm) were precisely aligned onto the MoS2 nanocircuits (see as a sake of example the few-layer MoS2 nanofinger device of Figure 4) thanks to its in situ imaging and direct t-SPL overlay nanopatterning based on the polymeric bilayer process.

Atomic Force Microscopy Characterization: High resolution AFM images were acquired by a JPK NanoWizard AFM (Bruker) operating in Quantitative Imaging mode.

The KPFM maps were acquired by a Nano Observer AFM (Concept Scientific Instruments) operating in single-pass mode and equipped with a platinum-coated silicon tip. The conductive AFM maps (current, resistance) were acquired by using the same Nano Observer AFM, equipped with a ResiScope module enabling c-AFM nanoscopy over wide current (and resistance) ranges avoiding damage to the sample. The c-AFM maps were detected with a conductive p-doped diamond tip scanning in contact mode.

The analysis of all the AFM images was performed with the software Gwyddion (open source).

Scanning Electron Microscopy Imaging: The top-view SEM topographies of the MoS2 nanocircuits (secondary electron signal) were acquired by exploiting a thermionic electron source biased at 5 kV (Hitachi SEM SU3500).

Raman Micro-Spectroscopy: Raman micro-spectra and maps were detected by using an NRS-4100 Raman microscope (JASCO) operating in back-scattering configuration with sample excitation with a 514 nm laser source.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

Data Availability Statement
The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords
additive nanolithography, few-layer MoS 2 nanocircuits, Kelvin probe nanoscopy, large-area 2D TMD semiconductors, thermal-scanning probe lithography

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