**Microwave Synthesis of Molybdenene from MoS2**

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**Abstract**

Dirac materials are characterized by the emergence of massless quasiparticles in their low-energy excitation spectrum that obey the Dirac Hamiltonian. Known examples of Dirac materials are topological insulators, d-wave superconductors, graphene, Weyl and Dirac semimetals, representing a striking range of fundamental properties with potential disrupting applications. However, none of the Dirac materials known so far shows metallic character. Here, we present evidences for the formation of free-standing molybdenene, a 2D material composed of only Mo atoms. Using MoS2 as a precursor, we induced electric field assisted molybdenene growth under microwave irradiation. We observe the formation of millimeter long whiskers following screw dislocation growth, consisting of weakly bonded molybdenene sheets, which, upon exfoliation, show metallic character, with an electrical conductivity of ~940 S m -1. Growth of molybdenene on 2D-hBN and 2D-MoS2 leads to a formation of hybrid layered-materials with tuneable optical and electronic properties. As a proof-of-principle, we also demonstrate the applications of molybdenene as surface enhanced Raman spectroscopy (SERS) platform for molecular sensing, as substrate for electron imaging and as scanning probe microscope cantilever.

**Keywords:** Molybdenene, Molybdenene Oxide, Microwave synthesis, Graphene catalyzed, AFM cantilever.

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**Main**

Elemental Dirac and Dirac-like materials are of intense research interests, as they typically do not suffer from impurities in structural phases, offering enhanced electronic mobility, in contrast to other, compound 2D materials. Elemental Dirac materials include graphene1, borophene2-4, phosphorene5, silicene6, 2D gold7 etc (**Fig S1**). Advancements in elemental Dirac materials are destined to involve higher atomic number metallic elements in the periodic table such as molybdenum, tungsten, titanium etc., for which the sea of electrons would be confined in 2D, potentially leading to exotic electronic and excitonic behaviour. In addition, these materials are structurally robust under mechanical load and at elevated temperature8. Moreover, transition metals exhibit variable oxidation states – a property essential to catalyze chemical reactions, thus their 2D confined atomic sheets could demonstrate extremely high catalytic activity. The advantage of 2D transition metals form could also be exploited in niche area, such as field emitters9, scanning tunneling microscopy (STM) tips10, nanoscale interconnects11, nano-electromechanical systems (NEMS)12, SERS based molecular sensing13. Growth of these 2D materials under ambient conditions is challenging due to their tendency to form clusters as well as their affinity towards oxygen14.

Here we provide evidences for the formation of free-standing atomic sheets of molybdenene, a 2D elemental Dirac material made of Mo atoms. We have prepared molybdenene under ambient conditions using microwave induced electrochemical reduction of commercially available molybdenum disulfide (MoS2) powder. Molybdenene shows an electric conductivity of 940 S m-1. We demonstrate the use of molybdenene to make AFM cantilevers, chemical and SERS-based molecular sensing and electron imaging. *Ab initio* density functional theory study corroborates the experimental findings, predicting a stable staircase-like molybdenene sheet with four-fold symmetry, as well as van der Waals type inter-layer interactions. We finally prepare 2D-2D hybrids of molybdenene with h-BN and MoS2. We have also shown the formation of molybdenene oxide.

**Experimental realization of molybdenene sheets**

Microwave assisted field-induced electrochemical transformation of MoS2 results in the synthesis of molybdenene (schematic of synthesis in **Fig 1a**, photograph of molybdenene whiskers in **Fig S2** and details in methods section**15-17**). When observed under field emission scanning electron microscopy (FESEM), the whiskers clearly appeared to be a layered material with large surface areas **Fig 1b-c** (details in **ESI Fig S3**). These whiskers, when exfoliated employing scotch tape, resulted in atomically-smooth metallic Mo atomic sheets, as observed in FESEM imaging. The formation of staircase-like features made up of atomic layers was revealed at the edges of the sheets, as observed in AFM images (**Fig 1d**). On the surface, evidence of screw dislocation mediated staircase-like growth with step height of 0.4 nm, typical for single crystals was observed (**Fig 1e**). Such growth mode has been earlier reported for GaN and MoS2**18-21**. Topography image of a molybdenene layer (thickness ~0.5 ± 0.05 nm) scotch tape exfoliated and transferred on silicon substrate is shown in **Fig 1f**.

The EELS spectrum of synthesized Mo sheets (large area image in **Fig 1g**) has two major high loss peaks L3 and L2 of Mo at 2552 and 2660 eV respectively, and one minor edge peak L1 at 2924 eV as shown in **Fig 1h (**peaks left shifted due to weakening of inter-layer coupling as compared to the bulk).**22** The low-loss region bestows zero-loss peak and low-loss Plasmon oscillation peaks without any signals of oxygen peak (see **Fig 1i**). The high-loss region (200-450 eV) beyond the low-loss Plasmon region has two major edges of Mo viz. M5 and M4 which are corresponding to energy loss near edge structure (ELNES) and two minor edges M3 and M2 are extended energy loss fine structure (EXELFS) (see **Fig 1j**). Moreover, the highly intense M4 edge of synthesized Mo reflects the high concentration of the Mo atoms, which is expected in Mo in comparison to MoO3.**22-23** Therefore, we can conclude that the synthesized 2D Mo is metallic in nature. Moreover, XPS study (ESI **Fig S4** and details in ESI) establishes that surface bound oxygen can be easily etched out by Ar+ plasma. After, 4 hours of Ar+ plasma irradiation, we indeed obtained XPS signal [Mo-3d5/2 peak at 228.13 eV (**Fig S4 e-f**, **Table S1)**] coming out of pure molybdenene metallic sheets. **Table S2** listing Mo 3d5/2 peak positions in XPS for pure molybdenum metal and its coumpunds clearly indicates that we have obtained pure metallic material. Thus, EELS and XPS together proves the chamical phase purity of the syntheized molybdenene samples.

When scrutinized under HRTEM, the molybdenene sheets were found to be entirely comprised of Mo atoms (**Fig 2a-b**) and highly crystalline (**Fig 2c**). Two distinct Eigen structures were revealed; one prominent phase with four-fold symmetry (region 1 marked in **Fig 2d** and FFT in the inset) and the other one strain-mediated 6-fold symmetry, found in very limited regions (region 2 marked in **Fig 2d** and FFT in the inset). Areal number density of atoms were 9.8 nm-2 and8 nm-2 for 4-fold and six-fold symmetry phase respectively. Incidentally, our results for Mo sheets are very close to that for Mo body centered cubic single crystalline molybdenum reported earlier**24-25.** Oxide formation can be safely excluded as MoO3 and MoO2 exhibit relatively higher inter-atomic distances ~3.6-4 Å and ~4.8-5.6 Å**26-30.** Thus, the synthesized material is proved to be metallic structure-wise. In addition, we confirmed the single crystalline nature of molybdenene sheets composed whiskers with four-fold symmetry through single crystal XRD pattern (ESI **Fig S5**, details in ESI). Material was found to be triclinic with crystal parameters a = 3.70 Å, b = 3.97 Å, c = 13.90 Å, α = 89.75°, β = 89.97° and γ = 89.99°. It should be noted that the sheets exhibited ~ 3.5 times larger c-parameter (characteristic of weak inter-layer coupling) than the bulk Mo crystal suggesting the arrangement of 2D sheets within the formed whisker.

Raman spectroscopy was also carried out to follow the exfoliation steps. As-prepared material exhibits Raman peaks at 284.01 cm-1, 364.5 cm-1 and 819 cm-1 which correspond to surface oxides (**Fig 2e**).Upon successive exfoliation, intensities of these peaks reduced. Moreover, after 8 such top layer removal exfoliation steps, Raman peaks arising due to pure metal (Mo-Mo) at 405 cm-1 and second harmonic peak at ~810 cm-1 were observed**31-33**. This establishes that oxygen is present primarily in top few atomic layers of synthesized Mo material. However, interior material just a few-layer inside is still intact and metallic. This is quite natural as the microwave exposure has been carried out in ambient conditions. Nevertheless, the EDS spectra of exfoliated oxygen-free molybdenene sheets, as shown in ESI **Fig S6**, provided clear evidence for the high purity of these metallic sheets. Separately prepared MO was also characterized by Raman spectroscopy, and it was established that characteristic Raman peaks corresponding to MO appears (AFM images in ESI **Fig S7**, Raman spectra in ESI **Fig S8**). In order to establish number of layer effect on the Raman peak positions, further exfoliation was carried out and it is found that the 810 cm-1 peak gets shifted and has indeed number of layer sensitive (**Fig 2f**). While on the other hand, 405 cm-1 peak does not shift much. Thinner sheets with less layers have less inter-layer coupling, however they couple better with silicon substrate. The metallic property of the molybdenene sheet was further confirmed by the Ohmic current-voltage characteristics in 2-probe I/V measurements using Keithley instrument on molybdenene sheets on lithographically fabricated device (ESI **Fig S9**) as shown in **Fig 2g**.

First-principles density functional theory study exhibits two constituent phases of molybdenene sheets namely four-fold and six-fold symmetry phases (**Fig 2h** and ESI **Fig S10-11**) with Mo-Mo distance ~2.9 Å (details in ESI). The four-fold symmetry phase consists of staircase-like structure while six-fold symmetry phase contains graphene-like surface. The phonon dispersion spectra of the four-fold and six-fold M sheets exhibit dynamic as well as phase instability of the free standing molybdenene sheet. However, four-fold symmetry structure is relatively more stable compared to six-fold with relatively small imaginary frequencies (ESI **Fig S10-11**, details of stability aspect in ESI). The inter-layer interaction in M sheets can be considered as van der Waals type interaction with an equilibrium inter-layer separation 3.20 Å and binding energy -1.17 eV (**Fig 2i**). However, the inter-layer separation is not very far away from Mo-Mo in-plane bond distance (2.91 Å) which indicates a very weak covalent interaction among the layers cannot be completely neglected. The metallic nature of atomic M sheets was further confirmed through DFT band structure calculations (**Fig 2j**).

Thin metallic flakes were examined using high-angle annular dark-field (HAADF) STEM (**Fig 3 a-b**). Several rectangular atomic layers lying over others in a stacked manner are visible (**Fig 3a**), consistent with the formation of molybdenene atomic sheets. The crystallographic structure evolution of molybdenene was explored by consecutive Ar plasma etching of the surface layers and subsequent HAADF STEM imaging. While our theoretical calculations confirmed the experimental result that the hexagonal phase is not stable and was indeed rarely observed, four-fold symmetry is a common feature in observation. Free standing molybdenene sheets hanging over bare copper grid were imaged in order to find accurate structures (**Fig 3c-d** and **3g-h**). Theoretical inter-atomic distance of 0.32 nm undisputedly validates value of inter-atomic distance of 0.33-0.36 nm 1.4 Å in graphene and 1.5 Å in borophene (due to large atomic radius of 139 pm), experimentally observed broadly over syntheized molybdenene sheets at various locations and on different sheets, as evident from atomistic line profiles. It should be noted that the average inter-atomic distance in molybdenene is distinctly different from that in its 2D oxides (MoO3 ~3.6-4 Å; MoO2 ~4.8-5.6 Å). Cross-sectional images (**Fig 3e-f**) further validates square lattice appearing for sheets on copper support and hanging in empty space. Thickness variation in sheets which were free-standingly hanging over empty spaces (**Fig 3i-j**) could be resolved and incidentally square lattice with inter-atomic distances 0.33-0.36 nm were invariably observed across the number of layers down to monolayers.

Our theory suggests that molybdenene has staircase-like features, with alternate atomic arrays lying below and other atomic arrays lying above. In high-resolution HAADF STEM imaging, both arrays are visible. In some areas, we found linear arrangements of atoms (**Fig 3j2**). **Fig 3g-h** shows intertwined atomic arrays. Line profiling vividly shows staircase and intertwined features. Free-standing molybdenene sheets exhibit local strain-mediated structural evolution in some areas (**Fig 3j3**) (ESI **Fig S12-16**, details of crystal growth aspect in ESI). Large area HAADF STEM imaging (**Fig S15**) vividly exhibits square lattice of molybdenene spread over the whole image area (~20 μm x 20 μm). Incidentally, sheet area extends typically to 50 μm x 50 μm. Keeping in mind the challenges to grow Xenes such as need of UHV and expensive precursor gases for bottom-up ALD/MBE/CVD techniques and liquid phase exfoliation resulting in low quality samples (lattice with structural defects and surface functionalities) with 50-500 nm lateral dimensions; present report is certainly significant. In the present study, graphene acts as a crystallization catalyst, as it absorbs microwave energy and transfers that heat to MoS2 sheets adjacent to it. Local heat at the G-MoS2 interface results in electrochemical reaction giving rise to decoupling of Mo atoms with S atoms which makes clusters. We found Moire pattern features (see **Fig S17**) in several areas during our detailed HAADF STEM imaging of molybdenene samples. This observation again attests to the formation of mono-/few- layered sheets. Thus, based on various microscopies (FESEM, AFM, TEM etc), especially HRTEM imaging and HAADF STEM high-resolution imaging, the atomic structure in molybdenene sheets was established. Raman spectroscopy, X-ray photoelectron spectroscopy and EELS spectroscopy established the chemical phase purity of the molybdenene sheets.

**Salient applications of molybdenene sheets**

Using molybdenene sheets of appropriate dimensions, we fabricated cantilevers for atomic force microscopy by gluing them onto silicon chips (**Fig 4a**). A typical molybdenene sheet chosen for cantilever fabrication is shown in the inset of **Fig 4a**. The most salient feature of our cantilevers is that they are made up of layered material with in-plane covalent bonding and inter-planar van der Waals interaction. This offers two possible ways of orientations (inset of **Fig 4a**). For the first orientation, where the layers are stacked parallel one above the other, the flexibility is dominant along the vertical direction and suppressed along the lateral direction34. This type of cantilever effectively minimizes the noise in lateral direction, thereby making it suitable for high resolution imaging in dynamic force mode. For the second orientation, where the layers are stacked laterally, the flexibility is limited in the vertical direction, thus minimizing the vertical deflection noise when used in lateral force mode35. We demonstrated successful imaging of versatile materials including hard silicon structures, smooth 2D surfaces and soft bio molecules using our cantilevers in the dynamic force mode. The tip was prepared by carefully bending the sharp end of the cantilever. The fabricated cantilever exhibited resonance frequency of about 135 kHz and Q-factor of about 160 (**Fig 4b**). These values closely matched with those of the commercially available cantilevers. The topography images of a standard silicon calibration grating with a pitch of 10 µm (**Fig 4c-d**), the surface of a molybdenene sheet with atomically resolved step heights (**Fig 4e-f**), and DNA strands on graphene oxide surface (**Fig 4g-h**), obtained with the fabricated cantilevers were at par with those of the commercial ones. It is noteworthy that our fabricated cantilevers are inherently metallic and highly reflecting which enhances the deflection sensitivity of laser-based photo diode detectors. In addition, our cantilevers are advantageous over the commercially available metal-coated silicon cantilevers which often suffer from failure due to delamination of metal coating during current-voltage measurements/imaging. 2D materials have been employed earlier as excellent nanomechanical resonators36-38 (details of 2D materials based resonator and chemical sensing in ESI).

SEM images of fabricated Mo cantilevers has been shown in **Fig 4i-j**. The cantilever in **Fig 4i** is shorter compared to that in **Fig 4j**. The longer cantilever has lower fundamental resonant frequency (89 kHz) and the shorter has higher (102 kHz) (**Equation (1)** of ESI). DNA detection has been carried out by attaching DNA to both the cantilever tips made up of Mo sheets, decrement in resonant frequency has been observed with the DNA attachment (**Equation (2)** of ESI). In **Fig 4i** the resonant frequency before DNA attachment was ~102 kHz (green curve) and it became 97 kHz (red curve) after DNA attachment. Similarly, in **Fig 4j** (long cantilever), the resonant frequency before DNA attachment was ~89 kHz (green curve) and after DNA attachment, it became 77 kHz (red curve). Secondary electrons are necessary in electron imaging through FESEM. Moreover, atomically flat anchoring surface provides enhanced imaging. Molybdenene metallic 2D sheets were therefore, explored as anchoring substrates for FESEM imaging. FESEM image of MWCNT over the glass substrate results in charging, which gives rise to poor-contrast in images (**Fig 4k**). However, charging is suppressed by taking molybdenene as an anchoring substrate for FESEM imaging of MWCNT and a clear topographic contrast is seen (**Fig 4l**).

The surface morphology of molybdenene sheets revealed flat terraces and staircase structures with a significantly high step density. Low coordination number of edge-atoms and high surface area-to-volume ratio make this material suitable for chemical sensing activities39. The chemical sensing properties have been tested by treating the material with ethanethiol which showed substantial changes in M morphology due to successful molecular attachment. The AFM topography images of the surface of molybdenene sheets before and after the treatment with ethanethiol are shown in ESI **Fig S18**. The surface stress and changes in mass and elasticity originating from molecules attachment play a crucial role in chemical sensing activity40,41. Hence, cantilevers made out of such material detect chemical species on its surface just by measuring the changes in frequency or force constant parameters. Molybdenene was also found competitive with gold and graphene42 as a material platform for surface-enhanced Raman spectroscopy (SERS) (ESI **Fig S19**, details in ESI).

**Molybdenene-based 2D-2D hybrids**

Raman spectroscopy of Mo-BN hybrid shows peaks corresponding to both BN and molybdenene (**Fig 5a**). Photograph of the Mo-BN hybrid is shown in the inset of **Fig 5a**. Structural modulation and reconfiguration of molybdenene is observed due to strong electrostatic inter-layer coupling in these new van der Waals heterolayers. The TEM image along with the elemental profiling of M-BN hybrid (**Fig 5b-e**) reveals overlying Mo and BN individual layers. The HRTEM image of M-BN overlap area (ESI **Fig S20**a) exhibits crisscross patterns, where the inset SAED pattern depict square crystal symmetry. HRTEM (region 1 marked in ESI **Fig S20**a) undisputedly reveals that an atomic sheet consisting of larger (Molybdenum) atoms is positioned above another atomic sheet constituted of smaller atoms (B and N both being smaller). It should be noted that the role of the inter-layer coupling between adjacent atomic sheets constituting 2D materials hybrids in triggering atomic structure evolution/reconfigurations is very crucial. The average inter-atomic distances of 0.35 nm were found equal along the two symmetry directions (**Fig 5f-g**). Optical band gap determined from Tauc plot is ~3 eV (**Fig 5h**). Electrical and optoelectronic measurements confirm the semiconducting nature of the M-BN hybrid (**Fig 5i**).

Raman spectroscopy of Mo-MoS2 hybrid shows peaks corresponding to both MoS2 and molybdenene (**Fig 5j**). Photograph of the Mo-MoS2 hybrid is shown in the inset of **Fig 5j**. The TEM and elemental profile of the M-MoS2 hybrid exhibits two different statistically distributed structures with signature of both Mo and MoS2 sheets (**Fig 5k-m**). The HRTEM image of region 1 marked in **Fig 5n** and inset FFT pattern **Fig 5o** reveals stripe patterns of atoms in M-MoS2 system with the average inter-atomic distance along two perpendicular directions of about 0.30 nm (see **Fig 5p**). In contrast, region 2 exhibits hexagonal symmetry in crystal structure with average inter-atomic spacing of 016 nm and 0.32 nm, respectively in the two directions (ESI **Fig S21a-c**). The optical band gap was determined to be 1.65 eV (**Fig 5q**). The semiconducting properties of M-MoS2 were confirmed by electrical measurement and the optoelectronic characterization shows a red-sensitive photocurrent due to band edge absorption of the hybrid (**Fig 5r**). Keeping electronic character of 2D materials43-47 in perspective, molybdenene being metallic even at monolayer with sufficient carrier concentration and expected high mobility (in contrast to its bulk counterpart), thermal conductivity higher than its bulk, enhanced flexibility, Young’s modulus higher than the bulk; will be apt for electrodes in electronics/optoelectronics, energy-storage and in catalysis. Molybdenene-based 2D-2D hybrids assumes significance for coupling quantum states evolving at the interfaces playing crucial role in determining physical/chemical properties of 2D-2D hybrids48-50.

**Conclusions**

In summary, we have shown evidenced for the formation of a new 2D Dirac elemental material we termed as molybdenene, through a facile single-step synthesis strategy via graphene-mediated microwave power absorption. Free-standing molybdenene sheets with atomically flat surfaces over large areas can be easily obtained by mechanical exfoliation of the initial molybdenene whiskers under ambient processing conditions within few seconds. Additionally, 2D-2D hybrid materials with h-BN and MoS2 can be created with tunable structure and properties. We demonstrated the successful application of molybdenene as scanning probe microscope cantilevers for both imaging and for chemical detection, chemical and SERS-based molecular sensing. Electron rich molybdenene is suitable for imaging platform and is expected to be excellent catalyst in chemical reactions. The present study is poised to inspire molybdenene-based electronic devices and chemical sensors.

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**Author Contributions Statement**

PK conceived the idea of synthesis of molybdenene sheet. TKS and SC synthesized and thoroughly characterized it under supervision of PK. NK explored AFM cantilever and nano sensing applications under the supervision of AN. SC synthesized and characterized Mo-BN and Mo-MoS2 hybrids and explored SERS based molecular sensing with molybdenene sheet as an anchor. AHT and REDB helped with atomically resolved HAADF STEM imaging. IV helped with understanding of field assisted growth mechanism. RJ, SP and MM carried out DFT study under the supervision of AD. PK, AN and IV wrote the paper together.

**Competing Interests Statement**

The authors declare no competing interest.

**Figure Legends/Captions (for main text figures)**

**Fig 1 Experimental realization of molybdenene:** (a) Schematic diagram depicting graphene catalyzed microwave synthesis of molybdenene sheet (Intense electric field breaks Mo-S bonds and Mo atoms sneaks out through expanded graphene network), Mo, S and C atoms are represented by pink, yellow and grey respectively, (b) and (c) FESEM images of flat atomic sheets obtained by sonication followed by centrifugation of Mo whiskers. (e) (d, e) AFM images showcasing staircase (each step being 0.4 nm)like features indicative of screw-dislocation mediated growth of Mo whiskers, and (f) showing monolayer Mo sheet i.e., molybdenene, transferred onto SiO2 substrate. (g) Large area HAADF image, which is taken to acquire EEL spectroscopy in the marked green region. (h) EELS spectrum of synthesized Mo sheets in the high loss region ranging from 2400 eV to 3300 eV. (i) EELS spectrum ranging from 0 to 400 eV bestows zero-loss peak and low loss plasmon oscillation peaks. (j) EELS spectrum acquired high loss region (200-450 eV) depicting highly intense M4 peak.

**Fig 2 Structural details and electronic character:** (a-b) TEM imaging formed molybdenene sheet and elemental mapping. (c) HRTEM image of top surface of molybednene. (d) Zoomed-in image of marked region in image (c). Criss-cross patterns of atoms are observed with four-fold symmetry (region 1 in image (d)). Hexagonal arrangements of atoms are observed with six-fold symmetry (region 2 in image (d)). (e) Raman spectra of as synthesized Mo whiskers and surface attained after subsequent peeling of top layer (bottom spectrum for final peeling step clearly shows distinct Raman peaks at ~405 cm-1 which is characteristic of metallic phase and its second overtone at ~810 cm-1. For visual clarity oxide Raman peaks are red shaded and metallic Raman peaks are green shaded. Distinct metallic Raman signatures and flat sheets observed in FESEM, TEM and AFM establishes synthesis of free standing molybdenene. (f) Layer dependence Raman spectra of molybdenene. (g) *I*-*V* characteristics of Mo sheet placed on SiO2 substrate. (h) Molybdenene structure with four-fold symmetry obtained by DFT calculations. (i) The minimum energy curve to determine equilibrium inter-layer separation for molybdenene surface with four-fold symmetry. (j) DFT band structure calculations of four-fold structure of molybdenene.

**Fig 3 Atomic resolution HAADF STEM imaging:** (a-b) Low magnification HAADF STEM images of molybdenene highlighting the electron-transparent nature of the sheets. Stacked atomic layers are visible. (c, d, g, h) High-resolution HAADF STEM images of different areas along with atomistic line profiles. (e-f) High-resolution cross-sectional images of molybdenene atomic sheets. Four-fold atomic arrangements are observed. Layer dependent resolved images (i, j) along with zoomed-in imges in selected locations 1-3 are shown.

**Fig 4 Applications of molybdenene:** (a) Optical images of fabricated and commercially available cantilevers. Upper left inset shows FESEM image of a typical molybdenene sheet used for cantilever fabrication. Parallel and perpendicular orientations of molybdenene sheets are schematically shown in the upper right inset. (b) Amplitude and phase versus frequency plots of a fabricated cantilever. Comparison of topography images of standard calibration grid (c, d), 2D structure of molybdenene sheets (e, f) and DNA on graphene oxide sheets (g, h) obtained with fabricated and commercial cantilevers, respectively. (i-j) SEM images of Mo cantilevers (inset having amplitude (V) vs frequency (Hz) sweep before (green curve) and after (red curve) DNA attachment) for molybdenene-fabricated tips of two different dimensions (length and breadth). (k-l) SEM images of MWCNTs over glass and Molybdenene as an anchoring substrate respectively.

**Fig 5 Molybdenene based 2D-2D hybrids:** (a) Raman spectrum of synthesized Mo-BN hybrid and a digital image with UV light exposure (inset). (b) TEM image of Mo-BN. (c-e) Elemential mapping of showing Mo-BN, boron (red dots), nitrogen (blue dots) and Mo (green dots). (f) HRTEM image of showing interwinded atomic arrangements. (g) Atomic line profile of image (f) as marked by the white and red line has average inter-atomic distance of 0.35 nm and 0.35 nm. (h) Tauc plot for optical band gap of Mo-BN hybrid. (i) I-V/PC behaviour of Mo-BN hybrid. (j) Raman spectrum of synthesized Mo-MoS2 hybrid and a digital image with UV light exposure (inset). (m-n) Elemential mapping showing Mo-MoS2. (m) TEM image of Mo-MoS2. (n) HRTEM image taken at the of two layers. (o) Zoomed-in HRTEM image of region 1 marked in (l) (inset FFT pattern). (p) Atomic line profile of image (o) has average inter-atomic distances 0.31 (green plot) and 0.30 nm (red plot). (q) Optical band gap plot of Mo-MoS2 hybrid. (r) I-V/PC measurements of Mo-MoS2 hybrid.

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**Methods:**

**1. Experimental protocols:** Initially molybdenum disulfide powder (MoS2) having 20 μm crystalline flakes were dried (at 150 °C on a hot plate for 2 hours). Then graphene powder was mixed with MoS2 (50:50) weight percentage to explore its role as a catalyst for the reaction. The mixed power was exposed to microwave at 720 W with shots of 10 secs for up to 1 minute in closed alumina vessel under ambient conditions. After process, crucible is allowed to cool down for efficient heat dissipation to attain thermal equilibrium and avoid the breaking of alumina crucible as well as a bottom glass plate. The subsequent process was again repeated 3 times to have enough long whiskers. In order to avoid breaking of the tip, we used electrostatic force to stick it on the edge of the sharp paper and gently place in a collecting box. These micro-whiskers were used for further characterization. Morphological feature was studied by FESEM by transferring Mo needles on a conducting carbon tape. I-V measurement was done in probe station at moderate vacuum conditions to avoid the oxidation of metallic needles. For photoconductivity measurements, blue, green and red lasers of wavelengths 505, 532 and 650 nm were used for optical excitation in PC measurements of Mo hybrids. Thiol functionalization was achieved by treating the obtained micron sized needles in ethane dithiol solution for 24 hrs under ambient conditions. Treated samples were removed from solution and washed with DI water to remove the unattached thiol group and dried under vacuum. For SERS, 10 ppm solution of methylene blue was used to demonstrate the SERS behaviour of Mo, also we have compared it with the graphene (Gr) and gold (Au) (gold sputtering done for 1 minute on SiO2 substrate) having good SERS capabilities.

**2. Synthesis of molybdenene and molybdenene oxide**

MoS2 powder (purity-99.99 %) and boron nitride powder (purity 99 %) were purchased from Sigma Aldrich, graphene powder from Ultra Nanotech (99 %) and ethane dithiol (98 %) from Merck, alumina substrate (99.7 %) from Ants Ceramic. Iso-propanol (IPA) was purchased from Merck (99 %). All chemicals were used as-received without any further purification. Molybdenene (M) was synthesized by microwave exposure of a mixture of MoS2 and graphene powder in the ratio 1:1 under ambient conditions. MoS2, in contrast to graphene, has lower thermal conductivity and higher dielectric loss, which supports heat accumulation at the interface. Graphene acts as a catalyst for effective microwave power absorption, which converts to heat and elevates temperature. Consequently, microwave absorption further increases, which in turn further increases the temperature. This dynamic process is characterized by production of thermal spikes creating highly energetic, strongly non-linear conditions at the interface. Details of microwave dissipation for 2D materials and its impact on peak temperature and rate of heating are presented in ESI section.

While generated thermal conditions instantly melt MoS2 (loosen bonds) as soon as temperature reaches its melting point (Tm = 1185 °C), simultaneously acting local vertical electric field of the microwave being extremely high (>106 V cm-1) results in breaking of Mo-S bonds. Released Mo atoms (rich in electrons) respond to such an enormous electric field and migrate through the graphene-MoS2 mixture, constituting molybdenene layer. Anisotropy in growth features arises from microwave electric field16-17. As time passes, several hot spots generate and the whole surface is thermally activated, and enhanced participation of Mo atoms takes place resulting in whiskers formation. These whiskers consist of weakly bonded molybdenene sheets. The initial surface temperature increase with time as heat accumulates in the system inducing a vertical thermal gradient. Of this reason bottom sheets grow larger compared the topmost layer, giving rise to the staircase like structure made up of molybdenene sheets. The process is schematically presented in **Fig 1a**. As a result, we obtained molybdenene whiskers of variable length and width. They appeared highly reflecting and flat under optical microscope with size ranging from a fraction of micrometer to a few millimeters. The essential role of graphene as a catalyst can be inferred from ESI **Fig S2**. Molybdenene oxide (MO) was further produced from M by heating it at 200 °C and at 500 °C under ambient atmosphere for 2 hours (AFM images in ESI **Fig S7** and Raman spectra in ESI **Fig S8**).

**3. Synthesis of molybdenene based 2D-2D hybrids**

Hybrids of molybdenene with boron nitride (BN) and molybdenum disulfide (MoS2­) were synthesized sonochemically. First, the BN and MoS2 2D materials were synthesized. In order to attain them, two different container having dispersions of 50 mg each of BN and MoS2 in 25 ml isopropanol (IPA) were kept in an ultra-sonicator (Cole Parmer, 40 kHz) and sonicated for 12 hours. The supernatant was separated from both sonicated solutions by centrifuge machine (Remi, R-24) at a centrifugation speed of 3000 rpm, equivalent to relative centrifuigal force of 704 x g; for 2 minutes. The obtained supernatant was dried at 100 ˚C for 2 hours. Now for the molybdenene and BN hybrid (M-BN) synthesis, 5 mg of molybdenene was dispersed in the 10 ml of IPA solvent along with the 5 mg of synthesized 2D BN. The resultant mixture was then ultra-sonicated for 6 hours and thus M-BN hybrid was obtained. Similarly, For Mo and MoS2 hybrid (M-MS), 5 mg of both molybdenene and synthesized MoS2 was dispersed in 10 ml of IPA solvent and sonicated for 6 hours. The supernatants were taken from synthesized M-BN and M-MS hybrid solutions and were used for further characterizations.

**4. Material characterization**

Synthesized 2D Sheets of Mo is first diagnosed by optical microscope (Olympus 100X). Single crystal XRD (AXS D8 Quest system from Bruker) is performed to check the type of crystal structure along with its related parameters. Further, its surface morphology was studied by FESEM (Hitachi S-4800) at operating voltage 30 kV and current 10 µA. Placing the rods on carbon tape. TEM, HERTM and SAED were obtained by JEOL (JEM 2100) of free standing Mo 2D sheets (dispersed in IPA sonicated for 4 hrs.). HAADF STEM images were recorded using a probe aberration corrected FEI Titan G2 80-200 ChemiSTEM. AFM (Agilent Model No.5500) measurements were performed in non-contact mode to check minutes details such as roughness and thickness of synthesized Mo sheets transferred on Si/SiO2 substrate. XPS measurements of Mo sheets were performed in solid phase under high vacuum condition by ESCA+Omicron Nano Technology GmbH to check its chemical purity. Raman spectroscopy of Mo sheets was carried out in solid phase before and after successive peeling steps within the wave number range of 100-1500 cm-1 in the back scattering geometry employing Confocal Micro-Raman Spectrometer (Seki Tech. Corporation, Japan) with 633 nm He-Ne LASER source.

**5. Computational**

All the spin-polarized calculations were performed within the framework of density functional theory (DFT) using the plane-wave technique as implemented in Vienna Ab Initio Simulation Package (VASP). The generalized gradient approximation method (GGA) parameterized by the Perdew-Burke-Ernzerhof (PBE) was used to account for the exchange-correlation energy.M1 DFT+U method was used to account for the on-site coulomb repulsion and improve the description of localized Mo d-electrons in Mo sheet with Ueff = 2.0 eV as recommended by the previous studies.M2-M3 The projector augmented wave potential (PAW) was used to treat the ion-electron interactions. To describe the effect of van der Waals interactions, DFT-D2 empirical correction method proposed by Grimme was applied.M4 In all computations, the kinetic energy cut off is set to be 500 eV in the plane-wave expansion. All the structures were fully relaxed (both lattice constant and atomic position) using the conjugated gradient method and the convergence threshold was set to be 10-8 eV in energy and 0.001 eV/Å in force. The Brillouin zone was sampled using a 9×9×1 Monkhorst-Pack k-point mesh both for geometry optimization and band structure calculation. Phonon dispersion spectra have been obtained using the density functional perturbation theory (DFPT) as implemented in the PHONOPY code.M5 Ab initio molecular dynamics (AIMD) simulation was performed using the VASP simulation package.Interlayer binding energy (Eb) was calculated as Eb = E(bilayer) – 2×E(single layer).

**6. Fabrication of AFM cantilever**

Initially, the even-sized Mo whiskers were separated from the microwave treatment graphene-MoS2 powder mixture. Suitable Mo sheets were obtained by exfoliation. Commercially available silicon chip was used as a supporting material for cantilever. Under Olympus binocular microscope (CX-21), the chip and Mo sheets were arranged to fabricate a cantilever. Prior to fixing rods on chip, small amount of fevikwik instant glue was applied to have good adhesion with chip and rods. Following this protocol, more than 10 cantilevers was prepared and tested in Multimode Agilent 5500 AFM setup. AFM tip was prepared by gently bending the apex region of the cantilever using a tweezer. The performance of each bended cantilever was then checked by scanning the standard calibration grids provided by Agilent AFM.

**Data availability**

Supplementary information includes (a) various sections on microwave heating mechanism **(Section 1),** X-ray photoelectron spectroscopy (**Section** 2), Comparison of areal atomic number density of molybdenene with other 2D materials **(Section** 3), Details of DFT theoretical calculation (**Section** 4), Structural details from STEM HAADF imaging **(Section** 5), Chemical sensing using AFM cantilever **(Section** 6) and SERS based molecular sensing **(Section** 7), (b) Figures **S1-S21**, and (c) Tables **S1-S4**. Further, data is available upon request to corresponding author.

**Methods only references**

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