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Published in:
Chemistry of Materials

DOI:
10.1021/acs.chemmater.2c01602

Published: 25/10/2022

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Direct Epitaxial Growth of InP Nanowires on MoS₂ with Strong Nonlinear Optical Response

Abde Mayeen Shafi,* Susobhan Das, Vladislav Khayrudinov, Er-Xiong Ding, Md Gius Uddin, Faisal Ahmed, Zhipei Sun, and Harri Lipsanen*

ABSTRACT: Mixed-dimensional van der Waals heterostructures are promising for research and technological advances in photonics and optoelectronics. Here we report vapor–liquid–solid (VLS) method-based van der Waals epitaxy of one-dimensional InP nanowires (NWs) directly on two-dimensional MoS₂. With optimized growth parameters (V/III ratio, flow rates of precursors, and growth temperature), we successfully grow high-quality InP NWs on MoS₂. The density and vertical yield of NWs on MoS₂ are significantly high. Due to the unique properties of both materials, we observe strong linear and nonlinear optical responses from the NW/MoS₂ heterostructures. Intriguingly, in addition to strong second and third harmonic responses, the mixed-dimensional heterostructures show odd-order high harmonic generation up to seventh order. Our findings can open new possibilities for advancing attosecond physics on a new platform of mixed-dimensional heterostructures.

INTRODUCTION

The discovery of graphene in 2004 created a landscape of atomically thin two-dimensional (2D) materials for exotic fundamental studies and numerous applications owing to their unique optical, electronic, and physical properties. Among the large number of materials in the 2D family, the layered transition-metal dichalcogenides (TMDCs) are the most extensively studied after graphene. The atomic thickness and tunable gap spanning from near-infrared to visible regions make TMDCs a potential building block for a plethora of nanoelectronic and photonic devices. Additionally, these materials exhibit outstanding properties such as strong intralayer covalent bonds between atoms and weak van der Waals (vdW) forces between layers, indirect-to-direct band gap transition from the bulk to the monolayer, and strong photoluminescence (PL) in monolayers because of quantum confinement. 2D materials possess inherently dangling-bond-free surfaces and can form vdW heterostructures with distinct 2D and non-2D (e.g., 0-, 1- and 3-dimensional) materials circumventing lattice matching requirements. Such on-demand heterostructures provide unprecedented opportunities for next-generation optoelectronics.

III–V semiconductor nanowires (NWs) possess excellent light trapping properties, low density of crystal defects, long-term stability, high carrier mobility, and relaxed lattice matching conditions due to their small footprint. These unique properties make NWs promising and suitable to be grown on different foreign substrates, including atomically thin 2D materials, e.g., graphene and MoS₂. Intriguingly, when 1D NWs are combined with 2D TMDCs having mechanical flexibility and tunable optical properties, the mixed-dimensional heterostructure can produce high-performance photonic and optoelectronic devices with complementary characteristics. However, controlling the growth of NWs on 2D materials for achieving high vertical yield is still very challenging due to the absence of nucleation sites. Additionally, the stability of 2D materials after NW growth (especially in TMDCs) is another important issue to consider for utilizing the benefit of the mixed-dimensional heterostructures.

Being a direct bandgap material with very low surface recombination velocity and excellent optical quality, InP nanowires provide a superior platform for many applications such as solar cells, light-emitting diodes, photodetectors, and lasers. In the class of 2D materials, MoS₂ is one of the well-studied TMDCs because of having a direct bandgap in monolayers, large excitonic binding energy, strong luminescence emission, and high carrier mobility. Additionally, the nonlinear optical properties of both InP and MoS₂ are quite fascinating for many photonic applications. Therefore, mixed-dimensional heterostructures based on InP NWs and MoS₂ show unprecedented prospects in advanced photonic research.

Received: May 28, 2022
Revised: September 30, 2022
Published: October 13, 2022
In this work, we demonstrate mixed-dimensional heterostructures consisting of vdW-epitaxially grown InP NWs on chemical vapor deposition (CVD)-grown monolayer MoS$_2$. The Au nanoparticle-assisted low-temperature growth produces high-quality vertical NWs on MoS$_2$ while ensuring the thermal and chemical stability of MoS$_2$. The heterostructures show excellent linear optical responses (i.e., Raman and PL). We also observe strong nonlinear optical phenomena such as second and third harmonic generations (SHG, THG) from the heterostructures. Interestingly, the heterostructures generate odd-order high harmonic (HHG) up to the seventh order. These results manifest the potential of epitaxially grown mixed-dimensional heterostructure for nanophotonics.

**EXPERIMENTAL DETAILS**

Monolayer (ML) and few-layer (FL) MoS$_2$ are grown on Si substrate covered with 285 nm of SiO$_2$. The precursors used for growing MoS$_2$ flakes are Na$_2$MoO$_4$ (99.9%, Sigma-Aldrich) and sulfur powder (99.5%, Alfa Aesar). First, we clean the SiO$_2$/Si substrates with acetone and 2-propanol, followed by O$_2$ plasma treatment for surface activation. The substrates are immediately spin-coated with Na$_2$MoO$_4$ solution (density 10 mg mL$^{-1}$) at 4000 rpm for 1 min. The growth is carried out in a dual-zone CVD furnace with a 50 mm diameter and 200 cm long quartz tube. A schematic of the system is presented in Figure S1. We load an aluminum boat containing ∼100 mg sulfur powder in the upstream (zone 1) of 70 sccm Ar flow. The substrate is placed in the growth zone (zone 2). Zone 1 and zone 2 are heated to 180 °C and 775 °C, respectively, with a 10 ºC min$^{-1}$ ramping rate in zone 2. The temperatures in both zones are maintained for 10 min when the temperatures reach the maximum. Afterward, the furnace is cooled naturally to room temperature. The size of the as-grown triangular MoS$_2$ flakes varies in the range of 10–100 μm.

We grow InP NWs on MoS$_2$ in a horizontal flow atmospheric pressure metalorganic vapor-phase epitaxy (MOVPE) system. As catalysts for the vapor–liquid–solid (VLS) growth, 40 nm diameter gold (Au) nanoparticles from a colloidal solution (BBI International, UK) are used. Before the spin coating of Au nanoparticles, poly-L-lysine (PLL) solution is dropped onto the MoS$_2$ powder in the upstream (zone 1) of 70 sccm Ar flow. The substrate is then kept in the air for 30 s. Trimethylindium (TMIn) and tertiarybutylphosphine (TBP) are used as the precursors for growing InP NWs. To grow NWs, the precursors are inserted in the reactor simultaneously for 300 s while the reactor temperature is kept at 430 ºC, and the nominal V/III ratio is ∼200 by adjusting the flow rates of TMIn and TBP to 80 and 100 sccm, respectively. We use hydrogen (H$_2$) as a carrier gas, and the total gas flow rate in the 1-in. horizontal reactor is ∼5 L/min (slm). The temperatures reported in this work are thermocouple readings of the lamp-heated graphite susceptor, which are slightly higher than the actual substrate surface temperature.

**RESULTS AND DISCUSSION**

Figure 1a shows a schematic of epitaxially grown NW/MoS$_2$ mixed-dimensional heterostructure. The scanning electron microscopy (SEM) image of an as-grown MoS$_2$ single crystal is presented in Figure 1b. The growth temperature and the amount of precursors during the CVD growth process are favorable for growing ∼10–100 μm size triangular shaped of mono- and few-layer MoS$_2$ single crystals. Figure 1c presents the 45º tilted-view SEM image of as-grown InP NWs on the MoS$_2$ flake. The average height of the grown NWs is measured as ∼5 μm, and the yield of vertically aligned NWs is significantly high (∼1/μm$^3$). The density of the NWs is nearly homogeneous all over the monolayer MoS$_2$. The TEM image of the NW is shown in Figure 2a. The Au nanoparticle sitting on the NW tip is visible in the image. The high-resolution TEM (HRTEM) image in Figure 2b shows the atomic structure of the NW.

In this experiment, we use the bottom-up VLS growth method for epitaxially growing InP NWs on MoS$_2$. The VLS NW growth mechanism is usually mediated by the metallic droplets (i.e., Au nanoparticles in this experiment) in the liquid state, which collect the vapor-phase growth species and initiate the nucleation and growth of solid-crystal NWs underneath the droplets. This Au-seeded method allows precise control over the NW diameter, density, and position and reduces the possibility of radial growth. However, the vertical NW growth on MoS$_2$ largely depends on the V/III ratio, flow rate of the precursors, and growth temperature. With optimized growth parameters, adatoms impinge the surface of MoS$_2$ under Au seeds and get adsorbed on the site to initiate nucleation and growth of NWs below the seeds. The high-density NW growth usually depends on the sticking coefficient (defined as the ratio of number of adsorbed atoms to the number of migrated or desorbed atoms) and the number of Au seeds on the substrate. MoS$_2$ has a relatively high sticking coefficient, and Au nanoparticles with PLL treatment ensure an adequate number of seeds on MoS$_2$. The nanoparticles are mounted on the substrate via MoS$_2$ fixation, which prevents...
Ostwald ripening of these particles. Therefore, the NWs can grow by self-supporting each other, leading to the growth of highly dense and vertical NWs on MoS$_2$

The dangling-bond-free surface of any 2D materials makes it difficult to grow III–V NWs with good coverage and adhesion. Only the edges of the 2D materials have dangling bonds which promote the growth of NWs. However, it creates defects in the crystal due to chalcogen vacancies, e.g., sulfur vacancies in MoS$_2$, which also lead to oxidation. This results in degraded intrinsic properties of the materials and also becomes one of the reasons for forming particulate islands on MoS$_2$ (see Figure S2). This also implies that the growth of NWs requiring a relatively higher temperature (close to the growth temperature of the MoS$_2$) could cause phase alteration and deformation of MoS$_2$ crystals. Therefore, the thermal stability of MoS$_2$ during temperature treatment and NW growth is crucial to ensuring high quality of NW/MoS$_2$ heterostructure-based devices. In our experiment, we use 430 °C for growing InP NWs on MoS$_2$ flakes. This temperature is suitable for maintaining the crystal stability of MoS$_2$ during the NW growth and ensures the growth of high-quality InP NWs.

The chemical stability of MoS$_2$ during the growth of NWs is another crucial factor, which needs to be addressed to ensure the intact quality of MoS$_2$ after growth. First, we perform PLL treatment and then deposit Au nanoparticles on the MoS$_2$ flakes. Subsequently, we anneal the MoS$_2$ flakes at 430 °C for 300 s in the MOCVD chamber while inserting only the TBP precursor into the chamber. We do not observe any change in the Raman and PL spectra of MoS$_2$ which partially confirms the chemical stability of MoS$_2$ during NW growth.

Although the thermal and chemical stability of MoS$_2$ is relatively lower than that of many other 2D materials such as graphene and hBN, MoS$_2$ possesses a higher potential for 1D integrated high-performance photonic and optoelectronic devices because of its bandgap within the common semiconductor range. Additionally, the surface lattice structure of MoS$_2$ is also an excellent platform for vdW epitaxial growth of NWs, as MoS$_2$ has a lattice parameter of 3.16 Å which is approximately 28% larger than that of graphene, making it more favorable for the growth of III–V NWs with large lattice parameters, such as InP. The wurtzite (WZ) and zinc-blende (ZB) phase InP lattice parameters are found to be 4.15 and 5.86 Å, respectively. Therefore, the lattice mismatch between WZ InP-MoS$_2$ is smaller than that of ZB InP-MoS$_2$. This suggests that the growth of InP NWs with the WZ phase is more favorable than with ZB.

The high-resolution XPS spectra are acquired from the NW/MoS$_2$ samples to characterize the composition and quality of the materials. In Figure 3, XPS spectra show emission peaks from In-3d, P-2p, Mo-3d, and S-2p. The core-level spectrum of In-3d and P-2p is deconvoluted into four peaks. Peaks at 444.3 eV (In-3d$_{5/2}$) and 451.8 eV (In-3d$_{3/2}$) are ascribed to InP, whereas peaks at 445.2 and 452.7 eV represent In$_2$O$_3$. Figure 3b shows the P-2p spectrum with InP peaks at 128.4 (P-2p$_{1/2}$) and 129.1 eV (P-2p$_{3/2}$), and the InPO$_4$ peak at 133 eV. In both In-3d and P-2p spectra, the relative intensity of InP emission peaks is much higher than that of In$_2$O$_3$ and InPO$_4$, which implies that the optimized growth parameters are suitable for suppressing oxide formation and growing good quality NWs on MoS$_2$. All the heterostructures also contain the characteristic emission peaks of MoS$_2$. Figure 3c exhibits three major peaks of S-2s, Mo-3d$_{3/2}$, and Mo-3d$_{5/2}$ at 226.3, 229.1, and 232.3 eV, respectively, assigned to MoS$_2$. As shown in Figure 3d, we observe a doublet from the S-2p peak at 161.9 and 163.1 eV corresponding to S-2p$_{1/2}$ and S-2p$_{3/2}$ attributed to the MoS$_2$ crystal. All the XPS results agree with previous reports.

We further characterize the NW/MoS$_2$ heterostructure with Raman and PL spectroscopy to investigate their optical response. A comparison of those responses from WZ NWs grown on Si (111) and NW/MoS$_2$ is also presented in Figure 4. Figure 4a shows the room temperature Raman spectra from NWs and the heterojunction of NW/MoS$_2$. The longitudinal
optical (LO) and the transverse optical (TO) phonon modes of InP NWs grown on Si substrate are observed at \( \sim 301 \) and \( \sim 340 \text{ cm}^{-1} \), respectively. When NWs are grown on MoS\(_2\), we observe similar LO and TO modes with a shift of \( \sim 2.2 \) and \( \sim 3 \text{ cm}^{-1} \), respectively. In addition, the in-plane \( E_{2g} \) \( (\sim 383.7 \text{ cm}^{-1}) \) and out-of-plane \( A_{1g} \) \( (\sim 403.1 \text{ cm}^{-1}) \) Raman modes appear in the Raman spectra that correspond to vibrational modes of MoS\(_2\). The Raman signals from both materials are in good agreement with the previously reported results. \(^{43,44,45}\) The PL spectra from the samples are shown in Figure 4b. The PL emission peak from the WZ phase of InP NWs grown on Si is observed at \( \sim 1.4 \) eV (880 nm). However, the peak is blue-shifted by \( \sim 80 \) meV in the NW/MoS\(_2\) heterostructure. NWs grown on MoS\(_2\) have a smaller effective diameter than those grown on Si due to surface depletion, which could explain the NW PL blue-shift in NW/MoS\(_2\) compared to the NW/Si samples. \(^{46}\) The average diameter of NWs on MoS\(_2\) is \( 63.3 \pm 5 \) nm for NW/MoS\(_2\). Few of the measured tapered-shaped NWs have a diameter of \( \sim 30 \) nm at the middle and \( \sim 20 \) nm at the top end. Therefore, the quantum confinement effect at the top of the NWs is also possible because the exciton Bohr radius of bulk InP is \( \sim 20 \) nm. This effect could cause the blue shift of NW PL in NW/MoS\(_2\) sample as reported previously. \(^{47}\) The heterostructure PL also contains the A-exciton and B-exciton of monolayer MoS\(_2\) at \( \sim 1.82 \text{ eV} \) (678 nm) and \( \sim 1.96 \text{ eV} \) (631 nm), respectively. The Raman and PL responses of MoS\(_2\) from the NW/MoS\(_2\) heterostructures elucidate the intact quality of the MoS\(_2\) flakes after epitaxial growth of NW on them. Further, as shown in Figure S3, we observe a strong enhancement of absorption from NW/MoS\(_2\) compared to MoS\(_2\) over a broad-spectrum range \( \sim 550 \) to 800 nm) from the differential reflectivity measurements.

We measure the nonlinear optical responses (i.e., SHG, THG, and HHG) from our NW/MoS\(_2\) heterostructure. In SHG, the incident photons of frequency \( \omega \) generate new photons of frequency \( 2\omega \) (inset of Figure 5a). Figure 5a shows the power dependency of SHG intensity from the heterostructure with the excitation wavelength at 800 nm. The calculated slope \( \sim (1.85) \) confirms the optical process is of second order. \(^{14}\) The SHG intensities also vary, with excitation wavelengths ranging from 800 to 1500 nm, and are presented in Figure 5b. The signal intensity is highest for 900 nm excitation because the laser energy is in resonance with the InP exciton energy. From the SHG mapping on the heterostructure presented in Figure 5c, we observe that strong SHG signals are generated from all over the sample. As shown in Figure 5d, bare MoS\(_2\) shows a 6-fold SHG pattern, whereas the SHG from the heterostructure is independent of the polarization of the pump due to the random growth directions of the NW on MoS\(_2\). However, the intensity of the signals from the heterostructure is twice as strong as compared to bare MoS\(_2\).

In the THG mechanism, the new photons of \( 3\omega \) are generated from incident photons of \( \omega \) (inset of Figure 5d). Figure 5d shows the variation in THG intensities from the heterostructure with the incident laser power. The exponent of power-dependent THG signals is about 2.81 with 1300 nm excitation, confirming its third-order optical nonlinearity. \(^{48}\) Strong THG signals are detected from the heterostructure, with different wavelengths ranging from 1200 to 1500 nm, as shown in Figure 5e. The signals show almost similar strength for all the excitation wavelengths. Interestingly, upon exposure of the heterostructure to a 4000 nm pump laser, we observe fifth- and seventh-order harmonic generations at 800 nm and \( \sim 571 \) nm, respectively, as shown in Figure 5f. However, the intensity of fifth HG is much stronger than that of seventh HG.

Both InP NWs with noncentrosymmetric crystal structures and MoS\(_2\) contribute to the high-intensity lower-order (SHG and THG) signals and relatively low-intensity HHG signals from the heterostructures. However, NWs contribute more than MoS\(_2\) because they are grown at a high density on MoS\(_2\) which results in more light–matter interactions in NWs than in MoS\(_2\). Therefore, the 6-fold symmetry of SHG from monolayer MoS\(_2\) vanishes at the heterostructure, as shown in Figure S4. However, NW growth density is not uniform on MoS\(_2\), making it difficult to accurately measure the quantitative contribution from each material in the nonlinear optical responses.

Notably, we do not observe any high even-order harmonic generation from the samples. This could indicate that the photophysical process for HHG is different from the lower-order harmonic generation, such as SHG, which is produced due to the nonzero second-order susceptibility resulting from the inversion symmetry breaking points at the surface of the WZ InP NW crystals. \(^{50}\) However, the absence of higher-order harmonics could theoretically mean that the inversion symmetry is retained, or a different mechanism is responsible for such a semiconductor crystal. \(^{51}\) Additionally, the even-order HHG from MoS\(_2\) has already been reported. \(^{52}\) Unexpectedly, we do not observe any influence of MoS\(_2\) even-order HHG in NW/MoS\(_2\) heterostructure. We speculate two possibilities for such results: (1) the reported intensities of the even-order HHG signals from MoS\(_2\) are very small compared to the odd-order HHG signals. The low signal-to-noise ratio in our home-built experimental setup might suppress the intensities of the even-order HHG signals.
Therefore, it is difficult to distinguish even-order HHG from the noise level of the system. Additionally, the input power was very close to the burning threshold of the samples. Therefore, a further increase in the laser power was damaging to our samples. (2) The light–matter interactions in NW and MoS$_2$ might suppress the even-order HHG from the heterostructures because of their distinctive crystal symmetries and orientations. Therefore, it requires further theoretical and experimental investigations to explain the unusual phenomenon.

Our findings show a simple way to fabricate InP-NW/MoS$_2$ mixed-dimensional heterostructures. The nonlinear responses, especially the HHG from the NW/MoS$_2$ heterostructure, could create new possibilities for numerous applications (e.g., lasers, electro-optic modulators, and frequency converters) of mixed-dimensional heterostructures.

**CONCLUSION**

In this work, we demonstrate the successful growth of InP NWs on MoS$_2$. The high yield of vertical NWs is achieved through optimized growth parameters. The low-temperature NW growth process ensures the stability of MoS$_2$ and produces high-quality NWs. Further, we show the excellent Raman and PL responses from the heterostructure. Our mixed-dimensional heterostructures exhibit strong nonlinear optical responses (SHG, THG, and HHG), promising numerous technological advances in photonics.

**MATERIALS AND METHODS**

For structural analysis, transmission electron microscopy (TEM) measurement of NWs is carried out with a JEOL 2200FS double aberration-corrected field emission gun microscope operated at 200 kV. As-grown NWs are transferred on a 400-mesh copper grid having 3 mm diameter and covered with a carbon film on one side to support the NWs.

To investigate the chemical composition and chemical bonding states of the samples, X-ray photoelectron spectroscopy (XPS) is performed in the Kratos Axis Ultra system with a monochromatic Al Kα source. The wide scans are performed with a 300 × 700 μm analysis area. The high-resolution scans are performed with a 20 eV pass energy and a 0.1 eV energy step. All spectra are calibrated by the C 1s peak at 284.6 eV.

The room-temperature Raman and PL spectra are recorded in a backscattering geometry with a confocal micro-Raman system (WITec alpha300 RA+). In both measurements, a 100× objective with a numerical aperture of 0.7 is used for illuminating with a 532 nm laser at a 2 mW power. The morphologies of MoS$_2$ and NW/MoS$_2$ are examined by a scanning electron microscope (SEM) (Zeiss Supra 40).

For SHG, THG, and HHG, an ultrafast femtosecond laser (Spectra-Physics, TOPAS) is used. The pulse width and the repetition rate of the laser are ∼230 fs and 2 kHz, respectively. The laser is focused on the sample through an objective lens (40×, 0.75 NA) with a spot size of ∼2.5 μm. The nonlinear responses from the samples are detected in reflection configuration by a photomultiplier tube (PMT) following a monochromator (Andor 328i). The SHG mapping is taken using a home-build multiphoton setup. For this measurement, the repetition rate of the ultrafast laser at 800 nm is 84.49 MHz. The beam is focused on the sample with an objective lens (40×, 0.75 NA), and the SHG is collected by the PMT, followed by multiple optical filters to block the seed laser.

**ASSOCIATED CONTENT**

*Supporting Information*

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.chemmater.2c01602.

Schematic of a dual-zone CVD system for growing MoS$_2$, SEM image of InP NW grown on annealed MoS$_2$,
differential reflectivity comparison of MoS₂ and NW/MoS₂, polarization dependent SHG from MoS₂ and NW/MoS₂ samples, SHG from a MoS₂ sample, and typical NW SEM images from the NW/MoS₂ samples.

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Notes
The authors declare no competing financial interest.

Acknowledgments

This research was supported by the GrapheneCore3 number 881603 and Academy of Finland [grant no. 320167 (PREIN Flagship - Aalto University)]. V.K. acknowledges the support of Aalto University Doctoral School, Walter Ahlström Foundation, Elektronikkinäsinörin Säätiö, Sähköinsinöörien Säätiö, Nokia Foundation, Finnish Foundation for Technology Promotion (Teknijärjestön Säätiö), Wallemar von Frenckell’s Foundation, and Kansallis-Osake-Pankki fund. The authors acknowledge Micronova for fabrication and characterization infrastructure and provision of facilities of Aalto University at Otanano – Nanomicroscopy Center (Aalto-NMC).

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