

Synthesis of phase-pure monolayer 1T-MoS₂ and 2H-MoS₂ colloidal nanomaterials with negative photoconductivity

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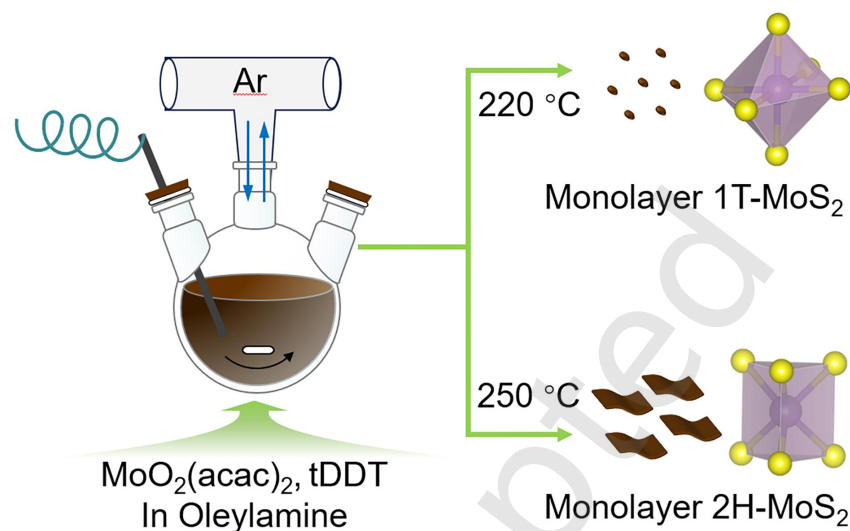
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We report a temperature-controlled wet-chemical strategy to synthesize monolayer 1T-MoS₂ and 2H-MoS₂ colloids. Despite their distinct exciton dynamics and electronic structures, both phases exhibit trap-state-mediated negative photoconductivity under near-infrared (825 nm) illumination

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ABSTRACT: MoS₂ is an important two-dimensional transition metal dichalcogenide (TMD), whose physical and chemical properties are closely related to its layer number, size, defects, and crystal structure. Therefore, the controllable synthesis of phase-pure monolayer MoS₂ represents a key challenge. Herein, we report a simple and efficient wet-chemical synthesis strategy for 1T-MoS₂ and 2H-MoS₂, with the crystal phase controlled by the reaction temperature. At 220 °C, ultra-small (1–2 nm) monolayer 1T-MoS₂ colloids are obtained; at 250 °C, monolayer 2H-MoS₂ nanosheets are produced. Both materials exhibit good dispersibility in nonpolar solvents and can be readily spin-coated into thin films. Interestingly, the 1T-MoS₂ and 2H-MoS₂ exhibit different exciton dynamics in transient absorption (TA) spectra, but similar trap state-induced negative photoconductivity (NPC) under near-infrared (825 nm) illumination. These findings provide new perspectives for the design of MoS₂-based optoelectronic devices.

KEYWORDS: 1T-MoS₂; 2H-MoS₂; colloidal nanomaterials; wet-chemical synthesis; negative photoconductivity

1 Introduction

As a quintessential representative of transition metal dichalcogenides (TMDs), molybdenum disulfide (MoS₂) occupies an important position in two-dimensional material research due to its unique layered structure and tunable physicochemical properties [1, 2]. The properties of MoS₂ are strongly dependent on its crystallographic phase. The 2H-MoS₂ is a semiconductor with a trigonal prismatic coordination (D_{3h}), demonstrating significant potential in field-effect transistors and photodetectors. In contrast, the 1T-MoS₂ exhibits an octahedral coordination structure (O_h), while the 1T'-MoS₂ possesses a distorted octahedral structure. The 1T and 1T' phases are metastable and display semimetallic or metallic properties, showing excellent performance in energy-related fields such as electrocatalytic hydrogen evolution reactions, supercapacitors, and batteries [3, 4].

To obtain high-quality MoS₂, researchers have developed various preparation strategies, which can be categorized into "top-down" and "bottom-up" approaches. The former primarily involves exfoliation methods, where few-layer or monolayer MoS₂ nanosheets are obtained from multilayer MoS₂, while the latter encompasses various synthetic methods. These approaches

can also be classified into solid-phase processes (e.g., mechanical exfoliation, etc.), solution-phase processes (e.g., liquid-phase exfoliation, hydrothermal/solvothermal methods, electrochemical exfoliation, etc.), and vacuum-based processes (e.g., CVD, ALD, sputtering, etc.). Among them, wet-chemical synthesis offers distinct advantages, including relatively mild reaction conditions, feasible operation, low cost, scalability for batch production, and facile control over nanoscale morphology [1].

Currently, although various wet-chemical methods for MoS₂ have been developed, a series of synthetic issues remain. One major challenge is phase purity. Due to its metastable nature, the 1T-MoS₂ tends to transform into the stable 2H-MoS₂, making it difficult to achieve high phase purity. While mixed-phase MoS₂ has been reported to exhibit superior performance compared to pure phases in some applications [5], the quantitative control and characterization of mixed phases remain challenging. Starting from phase-pure materials, however, enables precise control over arbitrary proportions of mixed phases. Phase-pure 2H-MoS₂ can be synthesized via hydrothermal methods [6], room-temperature liquid-liquid interface synthesis [7], hot-injection methods [8], and single-precursor thermal

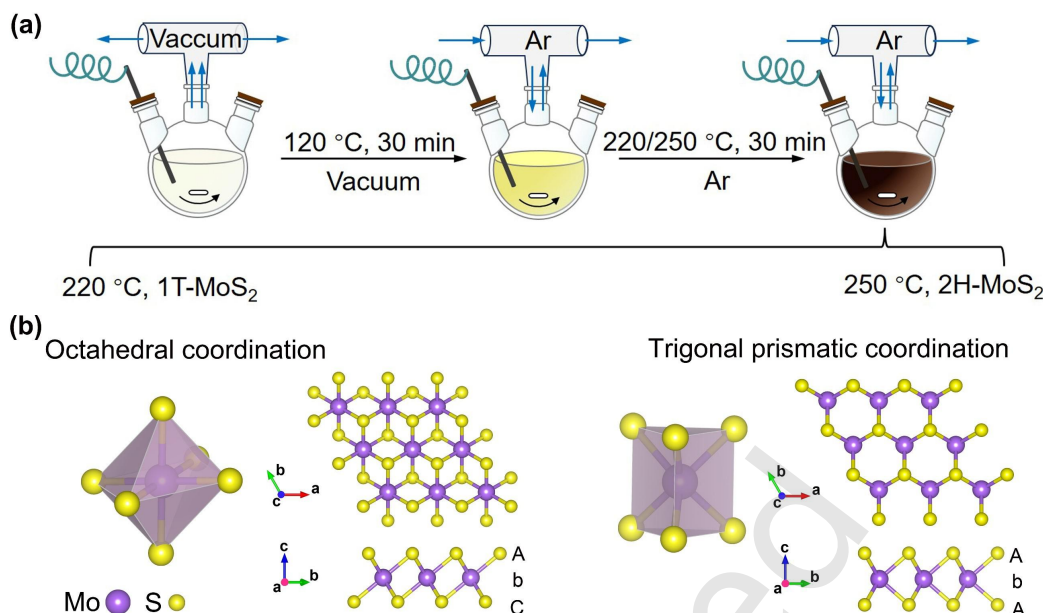


Figure 1. Schematic illustration and structural models of monolayer 1T-MoS₂ and 2H-MoS₂.

decomposition [9]. 1T-MoS₂ can be achieved via hydrothermal synthesis [10]. Generally, strategies involving hybridization with foreign ions, molecules, or nanostructures are employed to stabilize or confine the 1T phase [11–14], or the 2H-to-1T phase transition is employed during the intercalation–exfoliation process [15].

The second challenge lies in controlling the number of layers. MoS₂ possesses a two-dimensional layered structure, and its electronic properties are closely correlated with the layer count. For instance, 2H-MoS₂ exhibits a tunable bandgap: ~1.8 eV (direct) for monolayers and ~1.2 eV (indirect) for multilayers [16]. While most syntheses yield few-layer or multilayer products, monolayer 2H-MoS₂ has been successfully achieved through methods such as electrochemical intercalation–exfoliation [17] and high-temperature (320 °C) solution reactions [18]. One approach to preparing monolayer phase-pure 1T-MoS₂ involves epitaxial growth of 1T-MoS₂ on Bi nanocrystals, followed by dissolution of the Bi core with acid [19]. Using the interlayer space of layered double hydroxides (LDHs) as nanoreactors, MoS₂ with a monolayer percentage of 97% and a 1T phase content of 89% can be synthesized [20]. Additionally, hot-injection in strongly coordinating solvents (oleylamine and octadecene, OLA-ODE), utilizing electrostatic interactions to stabilize negatively charged nuclei with positively charged ligands, has successfully produced 1T-MoS₂ monolayer nanosheets [21].

To address the challenges in phase purity and layer number control as mentioned, this work develops a simple and efficient solution-phase synthesis strategy that enables the preparation of phase-pure monolayer 1T-MoS₂ and 2H-MoS₂ within the same synthetic system (i.e., using the same reactants, solvent, and method), with particular emphasis on the synthesis of ultra-small, free-standing colloidal 1T-MoS₂. The crystallographic phase of MoS₂ is controlled by simply tuning the reaction temperature. By setting the reaction temperature to 220 °C and 250 °C, we successfully synthesized monolayer 1T-MoS₂ and 2H-MoS₂, respectively. The crystal structure, morphology, size, as well as

the optical and optoelectronic properties of these colloidal MoS₂ were systematically characterized and investigated.

2 Experimental

2.1 Materials

Bis(acetylacetonato)dioxomolybdenum(VI) (MoO₂(acac)₂, 97%), Oleylamine (OLA, 80%–90%), and tert-dodecylmercaptan (tDDT, 98%) were purchased from Aladdin. Cyclohexane (≥99.7%), methanol (≥99.5%), and ethanol (≥99.7%) were obtained from Sinopharm Chemical Reagent Co., Ltd. All chemicals were used as received without further purification.

2.2 Synthesis of colloidal 1T-MoS₂ and 2H-MoS₂

The synthesis procedure is illustrated in Figure 1a. First, 32.6 mg of MoO₂(acac)₂, 0.19 mL of tDDT, and 15 mL of OLA were added into a 100 mL three-neck flask. The mixture was heated at 120 °C under vacuum for 30 min to form a clear solution. Subsequently, the temperature was raised to 220 °C under an argon atmosphere and maintained for 30 min under stirring, yielding a black solution. After cooling to room temperature, excess methanol was added, and the product was collected by centrifugation to obtain 1T-MoS₂. For the synthesis of 2H-MoS₂ nanosheets, the reaction was carried out at 250 °C for 30 min, and the product was isolated by centrifugation with ethanol. Both as-synthesized 1T-MoS₂ and 2H-MoS₂ could be dispersed in cyclohexane to form colloidal solutions.

2.3 Device Fabrication

The MoS₂ cyclohexane dispersion was spin-coated (600 rpm) onto the surface of indium tin oxide (ITO, 15 Ω·sq^{−1}) interdigitated electrodes (7×13 mm², with an interdigital spacing of 200 μm). The film thickness was controlled by adjusting the solution concentration and the number of spin-coating cycles. An 825 nm laser (LSR825NL) was used as the light source, and the photoelectric characteristics were measured using a Keithley 2636B sourcemeter. The responsivity (R, defined as the photocurrent generated per unit power of incident light on the effective area) and specific

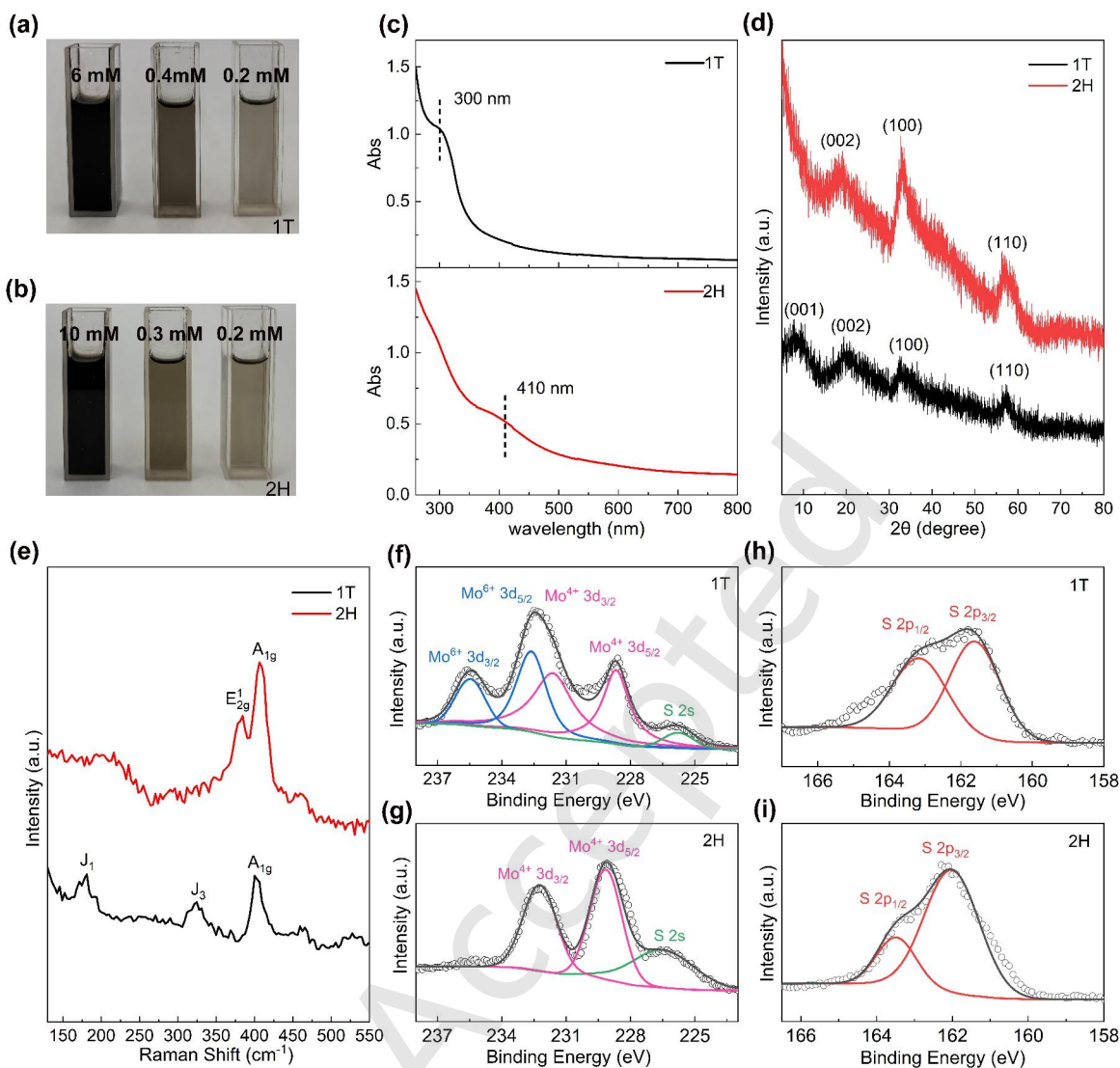


Figure 2. (a, b) Images of 1T-MoS₂ and 2H-MoS₂ cyclohexane solutions with various concentrations. (c) UV-Vis absorption spectra of 1T-MoS₂ and 2H-MoS₂ solutions. (d) XRD spectra. (e) Raman spectra (excitation wavelength: 532 nm). (f) and (g) XPS spectra of Mo 3d and S 2s of 1T-MoS₂ and 2H-MoS₂, respectively. (h) and (i) XPS spectra of S 2p of 1T-MoS₂ and 2H-MoS₂, respectively.

detectivity (D^* , assuming shot noise from dark current) were calculated.

2.4 Characterization

The morphology and structure of MoS₂ were characterized using transmission electron microscopy (TEM) (HT7800, 120 kV), high-resolution transmission electron microscopy (HRTEM) (JEM-2100, 200 kV), and high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) (Arm300, 300 kV). Raman spectra of MoS₂ were acquired using a confocal Raman spectrometer with an excitation wavelength of 532 nm (Xplora Plus). Annealing treatment was performed in a tube furnace (OTF-1200X-S) under a nitrogen atmosphere. X-ray diffraction (XRD) patterns were recorded (Rigaku D/Max 2500V/PC) using solid powder samples (the centrifuged product was dried in an oven at 60 °C for 6 h). Film thickness and surface morphology were characterized by scanning electron microscopy (SEM) (JEOL JSM-7600F). Absorption spectra were measured using a Hitachi U-3010 ultraviolet-visible (UV-vis) spectrophotometer. X-ray photoelectron spectroscopy (XPS) was performed on a Thermo Scientific K-Alpha spectrometer. Transient absorption (TA) spectra were acquired using a TA100-

2431 ultrafast transient absorption system with a laser pulse width of 35 fs, a repetition rate of 1 kHz, average power of 0.16 mW and 0.098 mW (20.382 $\mu\text{J}/\text{cm}^2$ and 12.484 $\mu\text{J}/\text{cm}^2$, respectively).

3 Results and Discussion

A monolayer MoS₂ consists of three atomic layers (S–Mo–S), and different stacking configurations of these S–Mo–S layers give rise to distinct crystallographic phases. In 1T-MoS₂, the S–Mo–S atomic layers adopt an A–b–C stacking sequence, with Mo atoms in octahedral coordination. In 2H-MoS₂, the S–Mo–S layers are stacked in an A–b–A fashion, and Mo atoms are in a trigonal prismatic coordination environment [22], as illustrated in Figure 1b.

The MoS₂ synthesized by MoO₂(acac)₂ and tDDT in OLA were characterized by UV-vis, TA, Raman spectroscopy, XRD, XPS, and TEM. The results confirm that the product obtained at 220 °C corresponds to 1T-MoS₂, while that synthesized at 250 °C is 2H-MoS₂.

The as-synthesized 1T-MoS₂ and 2H-MoS₂ exhibit good

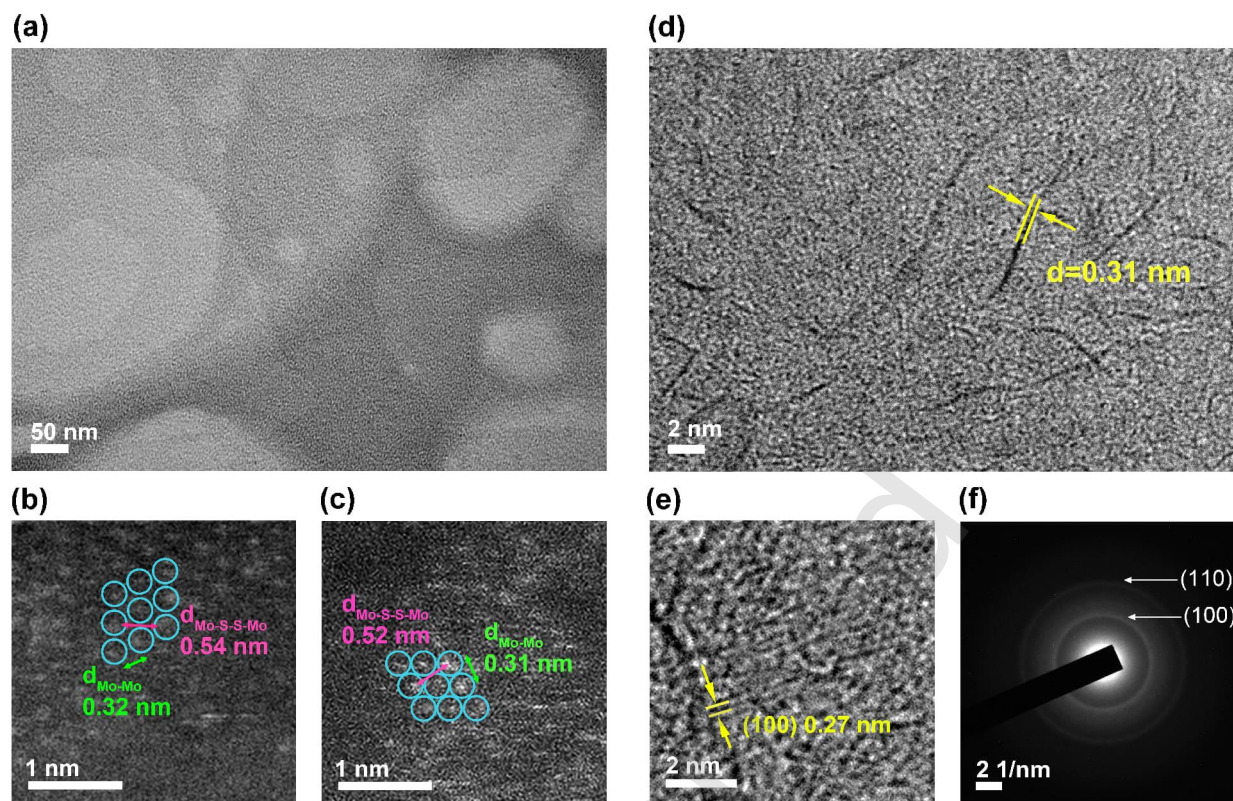


Figure 3. (a) TEM image, (b) and (c) HAADF-STEM images of the 1T-MoS₂ colloids. (d) TEM image, (e) HRTEM image, and (f) SAED pattern of the 2H-MoS₂ nanosheets.

dispersibility in cyclohexane, as shown in Figure 2a,b. At a concentration of 6–10 mM (based on the MoS₂ formula), both 1T-MoS₂ and 2H-MoS₂ appear as black solutions. At lower concentrations (0.1–0.4 mM), 1T-MoS₂ appears gray, while 2H-MoS₂ exhibits a grayish-green color, consistent with reported observations [23]. This color difference is corroborated by the UV-Vis absorption spectroscopy (Figure 2c). The absorption spectrum of the 1T-MoS₂ shows a peak at around 300 nm and no distinct absorption features in the visible to near-infrared range, with absorbance monotonically decreasing with increasing wavelength, indicative of its metallic character. In contrast, 2H-MoS₂ exhibits a broad absorption band centered at 410 nm (spanning 360–460 nm), a characteristic feature of semiconducting 2H-MoS₂. Compared to multilayer 2H-MoS₂ nanosheets, where this absorption band is commonly centered at 440 nm, the blueshift is likely attributable to quantum confinement effect in monolayer 2H-MoS₂ nanosheets. Both colloidal 1T-MoS₂ and 2H-MoS₂ are nonfluorescent. The reason could be that the metallic-phase 1T-MoS₂ is inherently nonfluorescent; while the absence of photoluminescence from the 2H-MoS₂ nanosheets may be attributed to quenching by surface defect states. The synthetic yield of monolayer 2H-MoS₂ nanosheets is nearly 100%, while that of monolayer 1T-MoS₂ is 30%, assuming the same ligand mass fraction for the 1T-MoS₂ and 2T-MoS₂ (~26%). It suggests a relatively lower nucleation and growth rate of the 1T-MoS₂ under the lower-temperature conditions.

The XRD patterns of the as-synthesized 2H-MoS₂ and 1T-MoS₂ are shown in Figure 2d. The broadened diffraction peaks suggest their nanoscale sizes. For 2H-MoS₂, the diffraction

peaks at 18.8°, 33.0°, and 58.0° correspond to the (002), (100), and (110) crystal planes, respectively. For 1T-MoS₂, the diffraction peaks at 32.6° and 57.1° are assigned to the (100) and (110) planes, respectively. The peaks at 8.7° and 20.2° are assigned to the (001) and (002) planes, respectively [23–26], which is commonly observed for 1T-MoS₂ distinguishing from 2H-MoS₂ with a mono-peak. Compared with the bulk MoS₂, the shifts in the (001) and (002) peaks are typically attributed to surface ligands. In some literatures, these two peaks are also indexed as (002) and (004) [27–29]. The d-spacing corresponding to 8.7° is 1.0 nm. If indexed as (002), the (001) spacing would be 2.0 nm. Given that the size of the 1T-MoS₂ here is only 1–2 nm, it is difficult to support a parallel arrangement with a 2.0 nm spacing. Therefore, the former indexing set is more reasonable in this concern. Both indexes do not affect the identification of the 1T-MoS₂ phase.

Figure 2e shows the Raman spectra of the 1T-MoS₂ and 2H-MoS₂. The peaks at 176, 324 and 400 cm⁻¹ of the 1T-MoS₂ are assigned to the J₁, J₃ and A_{1g} modes, respectively [22, 30]. The absence of the characteristic E_{12g} peak in the Raman spectrum of 1T-MoS₂ indicates that no 2H-MoS₂ coexists. The J₁ peak exhibits a shift compared to its typical position around 140–170 cm⁻¹, which may arise from the effect of ultrasmall size. The J₂ peak is not observed around 220–230 cm⁻¹, because it is the least intense of the J series [30]. For 2H-MoS₂, characteristic peaks are observed at 384 cm⁻¹ (assigned to the E_{12g} mode, originating from in-plane vibrations of S atoms relative to Mo atoms in opposite directions) and at 406 cm⁻¹ (assigned to the A_{1g} mode, originating from out-of-plane vibrations of S atoms in opposite directions). The shift of the

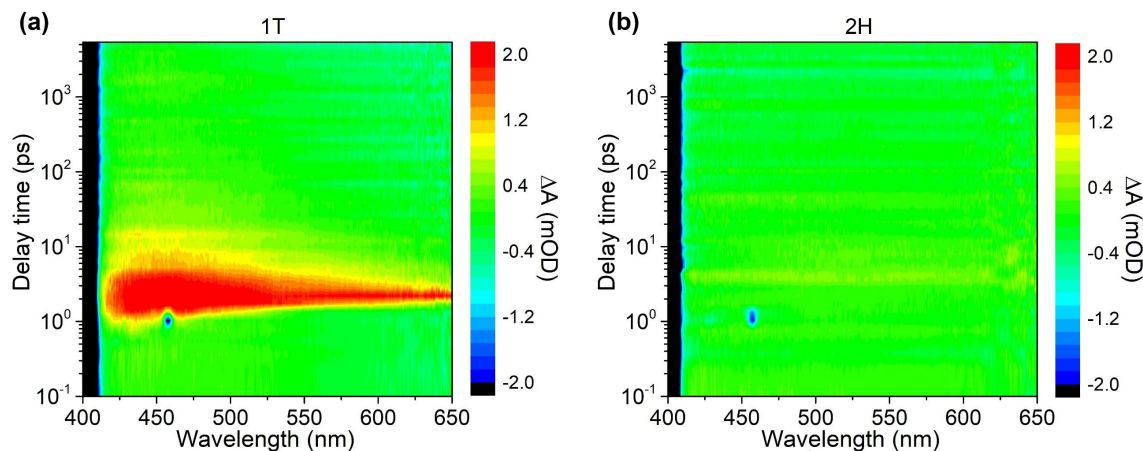


Figure 4. TA spectra of the (a) 1T-MoS₂ and (b) 2H-MoS₂.

A_{1g} peak to that of multilayered 2H-MoS₂ (~408–410 cm⁻¹) is consistent with the monolayer feature, where the weakened interlayer van der Waals forces reduce the restoring force for out-of-plane (A_{1g}) vibrations, leading to a decrease in frequency [31, 32].

The synthesized 1T-MoS₂ and 2H-MoS₂ were further characterized by X-ray photoelectron spectroscopy (XPS). Figure 2f–i shows the XPS spectra of the Mo 3d, S 2s, and S 2p orbitals. For 1T-MoS₂, the binding energies of Mo⁴⁺ 3d_{5/2} and Mo⁴⁺ 3d_{3/2} are 228.6 eV and 231.6 eV, respectively, and those of S 2p_{3/2} and S 2p_{1/2} are 161.5 eV and 162.8 eV, respectively. The fitting of 1T-MoS₂ also shows a pair of peaks that are associated with Mo⁶⁺, which could be induced by some oxidation species [30], likely due to the Mo⁶⁺ in precursor and abundant edges of the ultrasmall 1T-MoS₂ where Mo⁶⁺ may anchor. The Mo⁶⁺ species do not exist in the form of MoO₃, for no feature peaks corresponding to MoO₃ are found in the XRD pattern and the Raman spectrum. For 2H-MoS₂, the binding energies of Mo⁴⁺ 3d_{5/2} and Mo⁴⁺ 3d_{3/2} are 229.2 eV and 232.3 eV, respectively, and those of S 2p_{3/2} and S 2p_{1/2} are 162.2 eV and 163.6 eV, respectively. Compared with the 2H phase, these binding energy peaks of 1T phase exhibit a shift toward lower binding energy, with an offset of 0.6 eV–0.8 eV, which is a typical feature distinguishing the 1T and 2H phases [12, 32, 33].

In this synthetic system, the reaction temperature plays a decisive role in determining the phase and size of MoS₂. The 1T-MoS₂ synthesized at 220 °C exhibits ultrasmall nanocrystals with a size of 1–2 nm (Figure 3a). HAADF-STEM image (Figure 3b,c) reveals that the Mo atoms in the crystalline particles are arranged in a hexagonal pattern. The adjacent Mo–Mo distance ranges from 0.31 to 0.32 nm, showing some fluctuations. It is slightly smaller than the 0.32 nm spacing observed in bulk 1T-MoS₂. This modulation is likely attributable to the ultrasmall size and the monolayer structure, which is consistent with the observed shift of the J₁ vibrational mode. The HRTEM image shows that the 2H-MoS₂ synthesized at 250 °C has a lateral size of 5–10 nm (Figure 3d) and a thickness of 0.31 nm, indicating the monolayer structure. The lattice fringes and the corresponding selected area electron diffraction (SAED) pattern (Figure 3f) yield d spacings of 0.27 nm and 0.16 nm for the (100) and (110) planes of 2H-MoS₂, respectively, which are consistent with the JCPDS card No. 75-1539. The

TEM image of the sample prepared at 240 °C for 30 min (Figure S1(a)) shows both ultrasmall nanoparticles and nanosheets, suggesting a mixture of 1T-MoS₂ and 2H-MoS₂. TEM image of the sample synthesized at 250 °C for 10 min (Figure S1(b)) reveals the presence of smaller MoS₂ nanosheets with sizes of 2–4 nm, whereas the sample obtained after 30 min at the same temperature contains only larger nanosheets. We further investigated the thermal stability of 1T-MoS₂ by annealing its film at 250 °C in N₂ for 30 min. The Raman spectrum of the annealed sample shows the J₁, J₂, and J₃ modes (Figure S2). The absence of the A_{1g} mode and the emergence of the J₂ mode indicate a phase transition of 1T-MoS₂ to the distorted octahedral phase 1T'-MoS₂ [21]. These results indicate that the temperature and chemical environment govern the MoS₂ phase, while the reaction time controls the size of the monolayer 2H-MoS₂ nanosheets.

Transient absorption (TA) spectroscopy was employed to study the exciton dynamics of the 1T-MoS₂ and 2H-MoS₂. The 1T-MoS₂ exhibits photo-induced absorption (PIA) bands with positive signals in the visible spectrum range (Figure 4). Since 1T-MoS₂ is a metallic phase with its Fermi level located in the conduction band, the PIA (photoinduced absorption) signal originates from the absorption of high-energy carriers generated by the pump excitation. This signal predominantly lies in the range of 430–530 nm, centered at ~470 nm, and exhibits a significant attenuation at longer wavelengths. In contrast, 2H-MoS₂ shows no ground-state bleaching (GSB) or PIA signals. It is due to the rapid relaxation of excitons by an ultrafast recombination process. The TA features of 1T-MoS₂ and 2H-MoS₂ are unlikely to originate from Mo⁶⁺ species, which would induce a GSB. Nevertheless, Mo⁶⁺ may play a role in defect engineering, affecting the carrier transport dynamics in devices.

Uniform thin films of 1T-MoS₂ and 2H-MoS₂ were obtained by spin-coating their cyclohexane solutions (Figure 5a). The film thickness can be controlled by adjusting the concentration of the spin-coating solution, the number of spin-coating cycles, and the spin speed. Typically, we fabricated a 2.9 μm thick 1T-MoS₂ film (concentration: 6 mM, 600 rpm for 15 s, 25 coating cycles) and a 1.3 μm thick 2H-MoS₂ film (concentration: 10 mM, 600 rpm for 15 s, 15 coating cycles) on planar ITO interdigitated electrodes (Figure 5b,c).

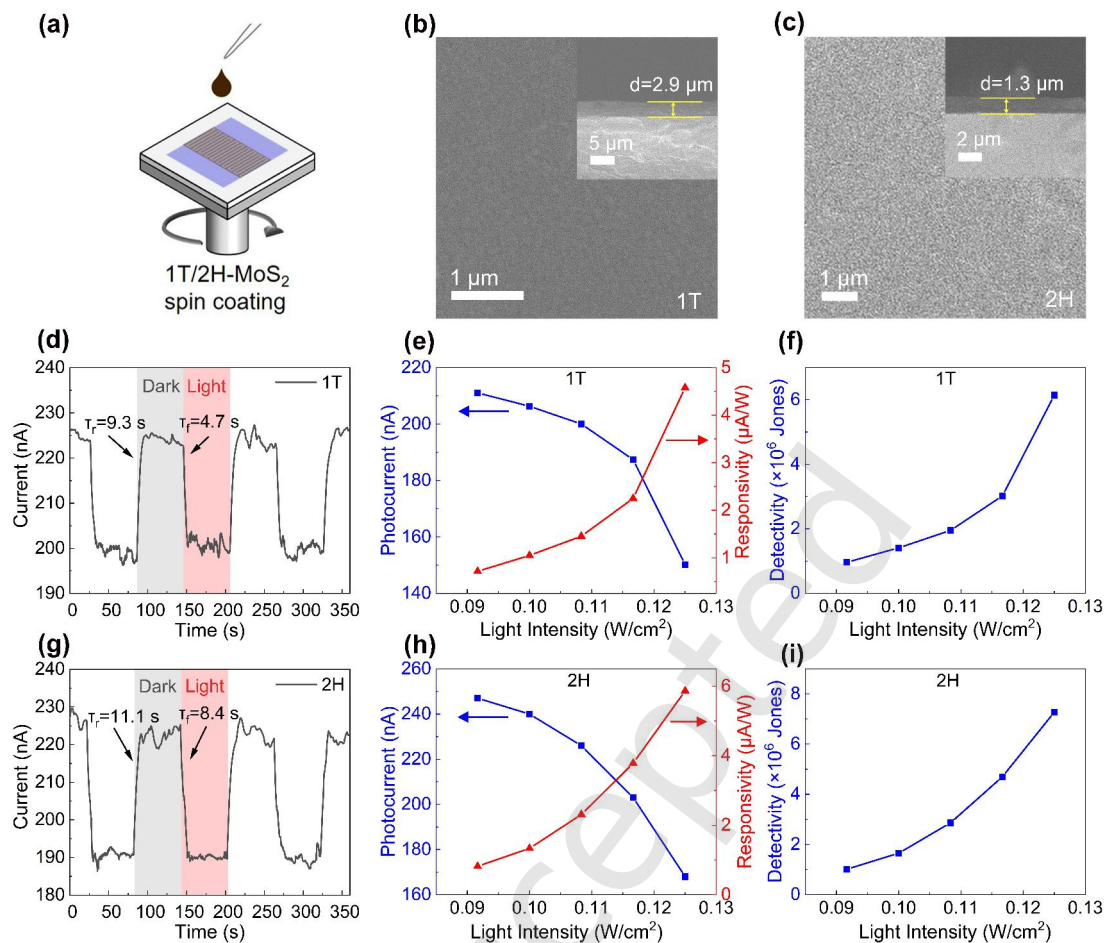


Figure 5. (a) Schematic illustration of the fabrication process of ITO/(1T/2H-MoS₂)/ITO devices by spin-coating. SEM top-view and side-view (inset) of spin-coated films of (b) a 1T-MoS₂ film and (c) a 2H-MoS₂ film. (d) and (g) Periodic photocurrent response of the devices under 5 V bias and 825 nm laser illumination (0.108 W/cm²). (e) and (h) Light intensity dependent photocurrent and responsivity. (f) and (i) Light intensity dependent detectivity.

Under 825 nm illumination with a bias voltage of 5 V, both devices exhibited negative photoconductivity (NPC) responses (Figure 5d,g). The NPC effect in MoS₂-based devices can arise from various mechanisms, including thermal effects, defect/trap capture, ion modulation, and heterojunction interface effects [34-41]. For the simple planar interdigitated electrode employed in this study, the response mechanism excludes the influence of external ion modulation, gate field or complex heterojunction interfaces. Furthermore, the relationship between light power and device response characteristics shows that for both 1T-MoS₂ and 2H-MoS₂, responsivity increases with increasing light power, while photocurrent decreases (Figure 5e,h, Figure S3). This trend contrasts with the characteristics of thermally dominated negative photoresponse mechanisms in MoS₂ [34]. The detectivity increases with light intensity, with the maximum detectivity reaching 6.14×10^6 Jones for the 1T-MoS₂ device and 7.28×10^6 Jones for the 2H-MoS₂ device (Figure 5f,i). Given that the devices are based on monolayer MoS₂ colloidal nanocrystals with abundant surface defects, the carrier dynamics governed by defects or trap states represent the most plausible mechanism for the NPC effect. Comparison of the NPC of this work with reported MoS₂ NPC devices was provided in the supporting information (Table S1). The rise time (τ_r) and decay time (τ_d) for the 1T-MoS₂ device were 9.3 s and 4.7 s, respectively, while those for the 2H-MoS₂ device were 11.1 s and 8.4 s, respectively.

In both cases, the decay times were shorter than the rise times, indicating that the filling of trap states occurs faster than the relaxation process. Consistent with this, our previous studies on NPC devices based on colloidal nanocrystals, such as CsPbX₃ quantum dots, also demonstrated that the NPC behavior originates from trap states, with response characteristics similarly showing shorter decay times than rise times [42-44]. Defect states in MoS₂ typically include sulfur vacancies (V_S) and molybdenum vacancies (V_{Mo}). In the semiconducting phase, V_S are shallow donor levels (close to the conduction band) that induce localized lattice perturbations and act as scattering centers, leading to reduced carrier mobility. In contrast, V_{Mo} are deep acceptor levels (close to the valence band or mid-gap) that cause significant lattice relaxation, and the captured carriers are difficult to be released by thermal activation [45]. These characteristics account for the slower response rates compared to mechanically exfoliated MoS₂ [36, 38]. The slightly faster response rate of the 1T-MoS₂ than 2H-MoS₂ device is attributed to the synergistic effects of photoexcitation, trapping, and transport of the charge carriers, determined by the phase, size, and defects of MoS₂. On the one hand, the 1T phase is metallic with no intrinsic bandgap, resulting in higher carrier mobility than the semiconducting 2H phase. On the other hand, the smaller size and distorted crystalline of 1T-MoS₂, along with the presence of abundant organic surface ligands and oxide species,

mean that its conductivity is dominated by electron hopping between particles and it has a higher defect state density. Consequently, although the 1T-MoS₂ device exhibits a slightly faster response rate, it does not show significantly higher current.

4 Conclusions

In summary, we have developed a facile and robust wet-chemical strategy for the phase-controlled synthesis of monolayer MoS₂ colloidal nanomaterials. By simply tuning the reaction temperature within the same synthetic system, we successfully achieved the selective preparation of phase-pure metastable 1T-MoS₂ (at 220 °C) and thermodynamically stable 2H-MoS₂ (at 250 °C). Notably, the as-obtained 1T-MoS₂ exhibits an ultrasmall lateral size of 1–2 nm while maintaining its monolayer nature—a structural feature rarely reported for metastable-phase TMDs. Both 1T-MoS₂ and 2H-MoS₂ demonstrate excellent dispersibility in nonpolar solvents, enabling the facile fabrication of uniform thin films via spin-coating. Photoelectronic measurements reveal that both phases exhibit negative photoconductivity under near-infrared (825 nm) illumination, a phenomenon that may originate from defect-induced carrier trapping effect and warrants further investigation. This work not only provides a simple and efficient route for phase engineering of MoS₂ nanostructures but also offers new insights into the structure–property relationships of two-dimensional transition metal dichalcogenides, potentially guiding the design of future optoelectronic devices based on phase-tailored TMD materials.

Data availability

All data needed to support the conclusions in the paper are presented in the manuscript. Additional data related to this paper may be requested from the corresponding author upon request.

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Declaration of competing interest

All the contributing authors report no conflicts of interest in this work.

Author contribution statement

F. H. G., N. N. Z., W. Y. Z.: synthesis, characterization, and device investigation. Z. H. Y., Y. Y. S., Z. X. G.: TA characterization. D. D. X., J. C. B., X. X. X.: project administration and supervision. F. H. G., X. X. X.: writing manuscript. All the authors have approved the final manuscript.

Use of AI statement

None.

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Electronic Supplementary Material

Synthesis of phase-pure monolayer 1T-MoS₂ and 2H-MoS₂ colloidal nanomaterials with negative photoconductivity

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1. Figures

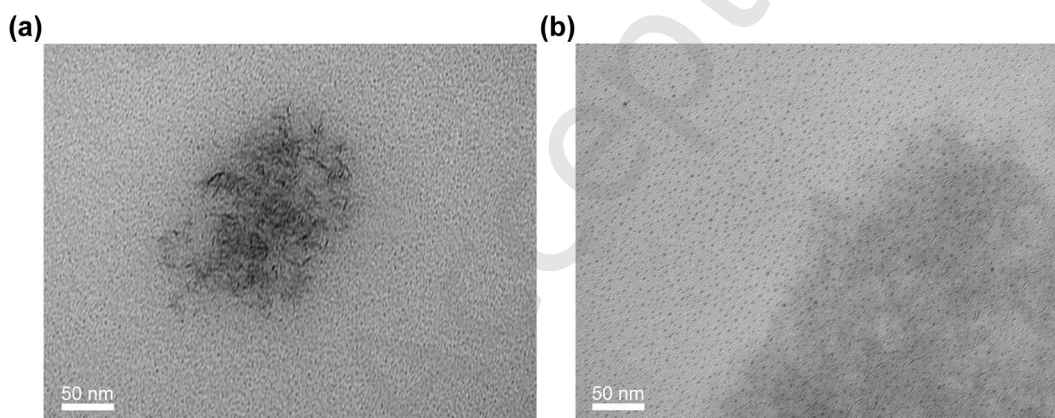


Figure S1. The TEM images of the MoS₂ synthesized at (a) 240 °C for 30 min, (b) 250 °C for 10 min.

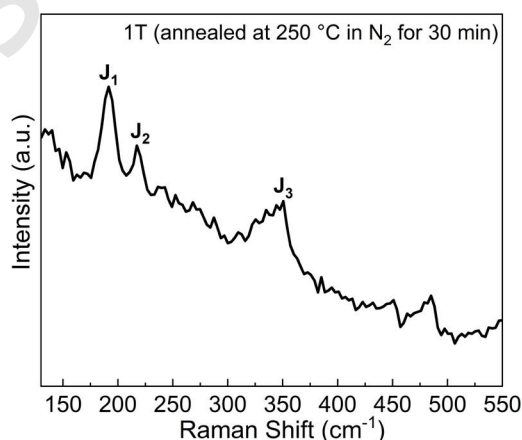


Figure S2. The Raman spectrum of the 1T-MoS₂ annealed at 250 °C in N₂ for 30 min shows 1T'-MoS₂ peaks.

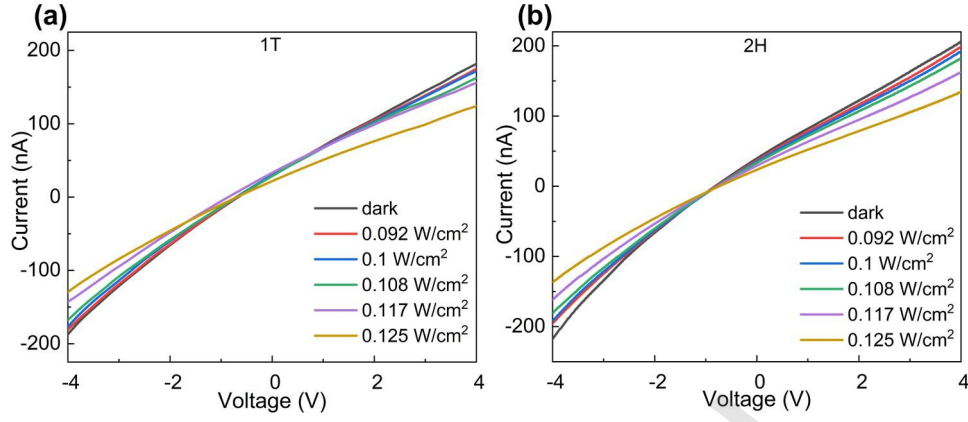


Figure S3. The incident light intensity dependent I-V curves of the (a) 1T-MoS₂ and (b) 2H-MoS₂ devices.

2. Responsivity (R) and detectivity (D*)

The formulas for responsivity (R) and detectivity (D*) are identical for both negative photoconductivity (NPC) and positive photoconductivity (PPC) [42]. Responsivity defines the photocurrent generated per unit incident optical power over the effective area of the photodetector and is given by:

$$R = \frac{|I_{\text{on}} - I_{\text{off}}|}{P_{\text{in}} A}$$

Where I_{on} is the photocurrent, I_{off} is the dark current, P_{in} is the optical power, and A is the effective area of the device. The detectivity (D*) characterizes the weakest optical signal detectable by the device. When the dark current is dominated by shot noise, D* is calculated as:

$$D^* = \frac{R}{\sqrt{(2q \frac{I_{\text{off}}}{A})}}$$

Where q is the elementary charge, and R is the responsivity.

The cross-sectional area of MoS₂ between the electrodes is proportional to the thickness d , and the conductance is proportional to this cross-sectional area. In the regime of relatively high film transmittance, the absorption, to a first-order approximation, is proportional to the thickness. According to the formulas for responsivity (R) and detectivity (D*), where I_{on} and I_{off} are approximately proportional to the thickness, it follows that R is approximately proportional to d and D^* is approximately proportional to $d^{1/2}$. Therefore, based on the data in Figure 5, for the same thickness, 2H-MoS₂ exhibits higher R and D^* values than 1T-MoS₂, although they remain within the same order of magnitude.

3. Table

Table S1. Comparison of positive and negative photoresponses of MoS₂-based optoelectronic devices.

Reference	Device structure	Layers	Method	λ (nm)	τ_r/τ_f (s)	R (A/W)	D* (Jones)	Type	Mechanism
[34]	Back-gate FET	Multilayer	Mechanical exfoliation	980 454	0.05/— s level	2.3 10 ⁵	— —	NPC PPC	Photothermal effect Photogating effect
[35]	Two-electrode	Monolayer	CVD	660 450, 532	— —	— —	— —	NPC PPC	Defect-trapped excitons Free exciton dominated
[36]	MoS ₂ /Ge FET	Multilayer	Mechanical exfoliation	1550 532	4 × 10 ⁻⁵ —	— 66	4.43 × 10 ⁶ —	NPC PPC	Photothermal & photogating competition, net negative photocurrent MoS ₂ photoconductive effect
[37]	Electrolyte-gated FET	Multilayer	Mechanical exfoliation	520 520	— —	— —	— —	NPC PPC	Au ³⁺ →Au NPs trapping photoelectrons Photogenerated carriers increase conductance
[38]	MoS ₂ /Si NP FET	Multilayer	Mechanical exfoliation	780 780	sub-ms sub-ms	— —	— —	NPC PPC	NP junction photogating (negative photovoltage) PN junction photogating (positive photovoltage)
[39]	Ion-gel top-gate FET	Monolayer	CVD	900 500	— —	0.63 21.79	10 ¹⁰ 2.45 × 10 ¹¹	NPC PPC	Ion desorption + photothermal synergy Photogenerated carriers increase conductance
[40]	MoS ₂ /MXene two-electrode	Monolayer	Solution-processed + annealed	750 550	$\tau_r < 0.15$ s level	1.9 —	2.1 × 10 ¹⁰ —	NPC PPC	Interfacial defect trapping electrons Band-edge excitation
[41]	Ferroelectric HZO-gate FET	Multilayer	Mechanical exfoliation	650	0.5	8.44 × 10 ³	—	NPC	Interfacial trap + ferroelectric polarization
This work	planar interdigitated electrodes	Monolayer	Wet-chemical synthesis + spin-coating	825	1T:9.3/4.7 2H:11.1/8.4	1T:4.58 × 10 ⁻⁶ 2H:5.87 × 10 ⁻⁶	1T:6.14 × 10 ⁶ 2H:7.28 × 10 ⁶	NPC	Trap-state-assisted carrier capture and release