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Significant pressure-induced enhancement of photoelectric properties of WS₂ in the near-infrared region

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ABSTRACT

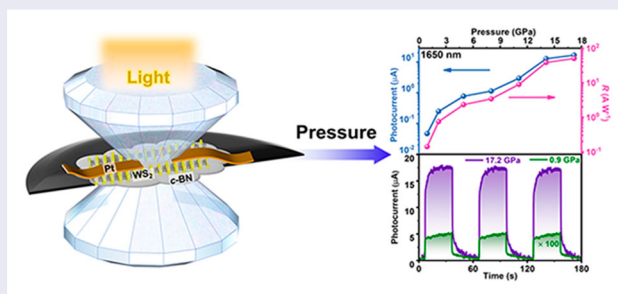
Here, a WS₂-based photodetector is fabricated and its detection range is extended to optical communication waveband by pressure. At ambient pressure, WS₂ exhibits a fast photoelectric response speed (rising/decaying time: 35/49 μs) under 635 nm laser illumination. Upon compression, the responsiveness and external quantum efficiency increase by two orders of magnitude under NIR illumination with different wavelengths as the pressure increases up to 17.2 GPa. The pressure-induced band gap reduction is mainly ascribed to the enhanced S-S interlayer interaction, leading to significant enhancement in the WS₂ photoresponsiveness. This study paves the way for designing high-performance broadband photodetectors through pressure modification.

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KEYWORDS

WS₂; high pressure; transition metal dichalcogenides; photoelectric property; crystal structure



IMPACT STATEMENT

Fast photoelectric response speed and extended detection range were achieved in the WS₂-based photodetector via pressure modulation, which paves a new way for designing high-performance broadband photodetectors.

Introduction

High-performance infrared photodetectors have attracted widespread attention owing to their wide applications in industrial and scientific fields [1–5]. Layered materials, such as graphene and black phosphorus, are often used as promising infrared photodetectors, presenting novel physical properties and excellent optoelectronic properties. However, with the ever-increasing demands, the disadvantages of the zero bandgap in graphene and the environmental instability in black phosphorus limit their further development in infrared photoelectric detection technology [6–8]. Therefore, there is an urgent requirement for novel infrared detection materials and feasible

methods for the improvement of the detection performance of photoelectric materials in the infrared waveband.

Recently, transition metal dichalcogenides (TMDs) have attracted extensive attention in the scientific community [9–12]. TMDs exhibit various outstanding physical properties such as tunable band gap, high optical absorption, and strong spin–orbit coupling that have immense potential in the fields of energy, catalysis, and optoelectronics [13–19]. TMDs have a typical sandwiched structure. The middle layer consists of transition metal atoms, and the top and bottom layers are made of chalcogen atoms. The intralayer atoms are covalently

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bonded, while the interlayers are connected by van der Waals forces [20]. Therefore, they can be easily exfoliated into multilayers or even monolayer nanosheets via ultrasonic or mechanical methods [21–24]. Similar to the most well-known TMDs, 2H-WS₂ is a widely studied TMD with thickness-dependent indirect-direct bandgap transitions and various bandgap values [25–27], for its individual use as a substrate material for photodetectors, the performance-tuning methods for WS₂-based photodetectors often focus on improving the quality of the prepared WS₂ and tuning its number of layers. Substantial efforts have been devoted to improving the WS₂ photoelectronic properties since Perea-Lopez et al. [28] first used it in the preparation of photodetectors, increasing its responsiveness, response time, and response range to 53.3 A W⁻¹, 560 μs, and 1064 nm, respectively [29–33]. This series of studies provide new prospects for enhancing the photoelectronic properties of WS₂. However, improving the photoelectric properties of WS₂ devices further with faster response speeds and wider spectral response ranges is still an enormous challenge.

Employment of high pressure is an effective method of modulating the bandgap and electronic states of materials that can further tune photoelectronic-related properties. Unlike chemical doping and molecular modifications [34,35], the application of pressure does not introduce chemical impurities and is more controllable. Recently, pressure-induced enhancement of photoelectric properties has been realized in a variety of materials, such as perovskites, Bi₂O₂S, AgIn₅S₈, and iodine [36–40]. In TMDs, enhanced photoelectric properties have been observed in ReS₂ and PtS₂ under pressure [41,42]. 2H-WS₂ has excellent mobility and carrier density than PtS₂ and ReS₂, which shows the advantages in photoelectric applications [43–48]. Previous experimental and theoretical calculation results have revealed that the applied pressure not only effectively regulates the charge carrier characteristics of WS₂ but also substantially modulates the light absorption ability of WS₂ [47,49]. All these properties are inextricably linked to the photoelectronic properties of a semiconductor, and therefore, it is anticipated that pressure regulation may be an effective method of modifying the photoelectric performance of WS₂.

In this study, we systematically investigate the photoelectric properties of WS₂ under pressure. The WS₂-based photodetector prepared in a pressure cell simultaneously achieves an ultra-fast response speed and a broad detection range at ambient pressure. The photoelectrical properties of WS₂ are significantly enhanced under high-pressure modulation in the detection range from visible to NIR. The continuous tuning of the photoelectric properties provides a platform for developing

pressure-modulated devices for future functional material technologies.

Materials and methods

Sample characterization

WS₂ crystal was purchased from 6Carbon Technology (Shenzhen, China). The sample was characterized by X-Ray diffraction (XRD, Rigaku Synergy Custom FR-X) with Mo Kα radiation ($\lambda = 0.7099 \text{ \AA}$) and scanning electron microscopy (SEM, FEI Magellan 400). A JEOL (JEM-2200FS) instrument with an accelerating voltage of 200 kV was employed for high-resolution transmission electron microscopy (HRTEM) and selected area electron diffraction (SAED).

In-situ XRD and Raman measurements

High pressure was generated using a diamond anvil cell (DAC) with a pair of diamond anvils (300 μm culet). Pressure was measured via the ruby fluorescence method. Silicone oil was used as the pressure-transmitting medium in the experiments. *In-situ* high-pressure XRD was measured by Rigaku Synergy Custom FR-X. The Raman spectra were recorded using a Renishaw inVia notch filter spectrometer equipped with a 514.5 nm exciting laser.

Photocurrent

In-situ high-pressure photocurrent measurements were conducted in a symmetrical DAC with a pair of diamonds with a culet size of 400 μm. A T301 stainless steel gasket was pre-indented to $\sim 50 \text{ μm}$ (thickness), and a sample chamber with a diameter of $\sim 380 \text{ μm}$ was drilled at the center of the indentation by a laser. An epoxy–boron nitride layer was inserted between the steel gasket and the diamond culet to achieve electrical insulation between the electrical leads and the metal gasket. Two platinum sheets were arranged to contact the sample in the chamber for photocurrent measurements as electrodes. The sample area between the two electrodes was approximately $8 \times 10^{-9} \text{ m}^2$. No pressure-transmitting medium was used for the photocurrent measurements. The photocurrents under pressure were recorded using a Keithley 2461 semiconductor characterization system under the illumination of a xenon lamp. A high-precision photocurrent scanning test microscope system (MStarter 200) with a source meter (Keithley 6482) was used for laser illumination of different wavelengths, including 635, 980 nm (power density: 15.3 mW cm^{-2}), 1270 nm (power density: 5.7 mW cm^{-2}), 1450 nm (power density:

5.4 mW cm⁻²) and 1650 nm (power density: 8.3 mW cm⁻²), the light power density under different wavelengths were measured by an optical power meter, the dark environment was required to avoid the interference of environmental light.

Computational details

All DFT calculations were performed using the projector augmented wave method (PAW) as implemented in the Vienna ab initio simulation package (VASP). The Perdew–Burke–Ernzerhof (PBE) was treated as the exchange–correlation potential within the generalized gradient approximation (GGA). We calculated the lattice parameters using the optB88b-vdW functional under pressure. The band gap and optical properties of WS₂ under pressure were postprocessed using VASPKIT. The cut-off energy for the plane-wave was determined to be 600 eV, and Monkhorst–Pack k-point meshes spanning not more than 0.03 Å⁻³ in the Brillouin zone were chosen. The convergence for energy was set to 10⁻⁶ eV on each atom, and the internal atomic positions and lattice constants under each hydrostatic pressure were optimized until the force on each atom was less than 0.01 eV Å⁻¹.

Results and discussion

The XRD pattern of layered WS₂ at ambient pressure is shown in Figure 1(a) which indicates that our sample has a 2H structure with space group *P6₃/mmc*, and its structure is schematically represented in Figure 1(b). Moreover, SEM and HRTEM measurements were obtained to investigate the crystal structure of WS₂, and the results are displayed in Figure 1(c,d). These images clearly illustrate the layered stacking structure with a lattice spacing of 0.27 nm in WS₂, and the corresponding SAED pattern (inset of Figure 1(d)) proves its hexagonal structure.

To evaluate the photoresponse performance of WS₂, a series of optoelectronic measurements was performed within the DAC device using a two-point probe. The inset image of Figure 2(a) shows a schematic of the prepared installation. The relationship between the photocurrents and different biases with the light on/off time interval of 5 × 10⁻³ s is shown in Figure 2(a). In addition, the response speed is an important parameter to describe the quality of a photodetector, the rise time τ_{rise} (and decay time τ_{decay}) is defined as the time intervals for the response rising (falling) from 10% (90%) to 90% (10%) of its peak value. As shown in Figure 2(b), under 635 nm laser illumination, τ_{rise} and τ_{decay} are calculated as ~35 μs and ~49 μs, respectively, at ambient pressure. Compared with the previously reported WS₂

photodetectors [28–33,50–55], our measured response and recovery times are the most rapid (Table S1). In addition, the effect of the illumination intensity (P_{in}) on the photoelectronic properties of WS₂ was also studied. Figure 2(c) plots the photocurrents of WS₂ under different P_{in} ranging from 0.1 to 25.7 mW cm⁻² under 5 V bias. It is clearly observed that the photocurrents increase with increasing P_{in} , which could be understood that more photogenic electron–hole pairs contribute to the photocurrent due to the higher illumination intensity [56]. Figure S1 displays the sublinear relationship between the photocurrent and P_{in} extracted from Figure 2(c), this phenomenon has been observed in several photoconductive detectors and is considered to be the result of electron–hole generation, trapping, and recombination in semiconductors [4,57–59].

The effects of pressure on the photoresponsiveness of WS₂ were further investigated. Figure 3(a) displays the photocurrents of WS₂ device under xenon lamp irradiation ($P_{in} = 1.2$ mW cm⁻²) at selected pressures with 5 V bias, and the observed linear I–V curves demonstrate that there is good Ohmic contact between WS₂ and electrodes, as shown in Figure S2a. The responsivity (R)

$$R = \frac{I_{ph}}{P_{in} \times S} \quad (1)$$

is described as the photocurrent generated per unit power of the incident light on the effective area, that is, $I_{ph} = I_{illumination} - I_{dark}$, S is the effective illuminated area [60,61]. During the compression process, the increase photoelectric response time of WS₂ is mainly owing to enhanced interatomic interaction, as shown in Figure S2b [62]. In addition, the effect of pressure on the WS₂ optoelectronic properties can be expressed by the dependence of the photocurrent and R on the pressure, as presented in Figure 3(b). The value of R reaches ~692.9 A W⁻¹ at 20.1 GPa that is ~20 times that at 1.4 GPa (35.6 A W⁻¹). In addition, according to the given formula, when P_{in} and S remain constant, as the pressure increases, ~20-fold increase in the photocurrent of the WS₂ device is achieved by pressure tuning. However, the absence of a significant increase in specific detectivity is considered mainly due to the inevitable increase in dark current with the applied pressure, as shown in Figure S2c and d. However, compared to the previous literatures, the specific detectivity under high pressure remains basically stable at a relatively high order of 10¹¹ [29–32,50,51], as shown in Table S1. The pressure-induced photocurrent increase is closely related to the decrease of bandgap, which is calculated in the following theoretical section. As the band gap decreases, electrons in the conduction band are more likely to be excited into the conduction band by the same illuminated wavelength. This phenomenon has also been

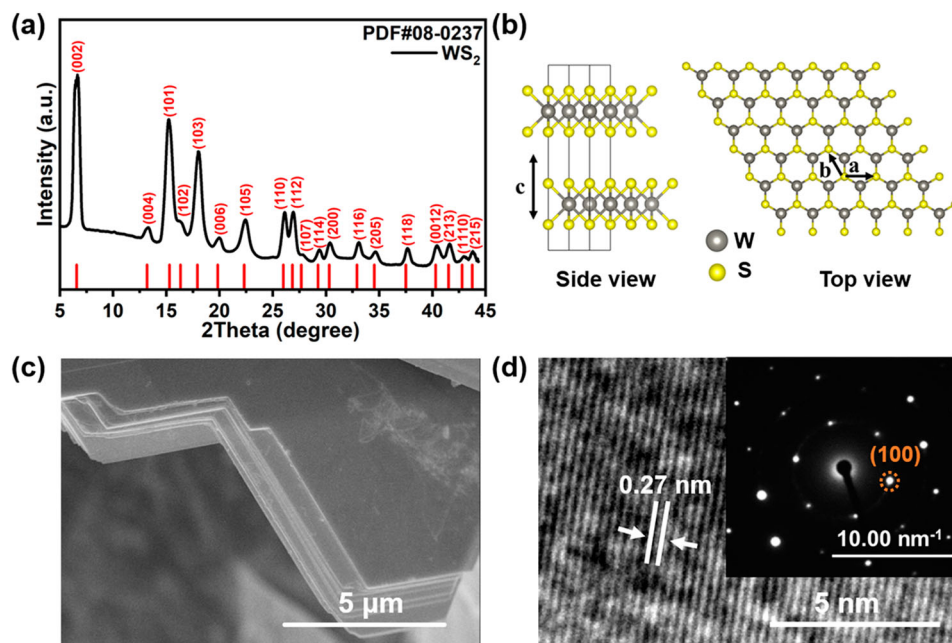


Figure 1. Sample characterization of 2H-WS₂. (a) XRD pattern of WS₂ at ambient pressure, the vertical lines at the bottom represent the theoretical diffraction peaks of 2H phase (space group $P6_3/mmc$). (b) Schematic of 2H-WS₂ along the c -axis (left) and the ab plane (right), respectively. (c) The SEM image of WS₂ indicates its layered structure. (d) HRTEM image of WS₂, the inset SAED image shows the hexagonal structure of WS₂.

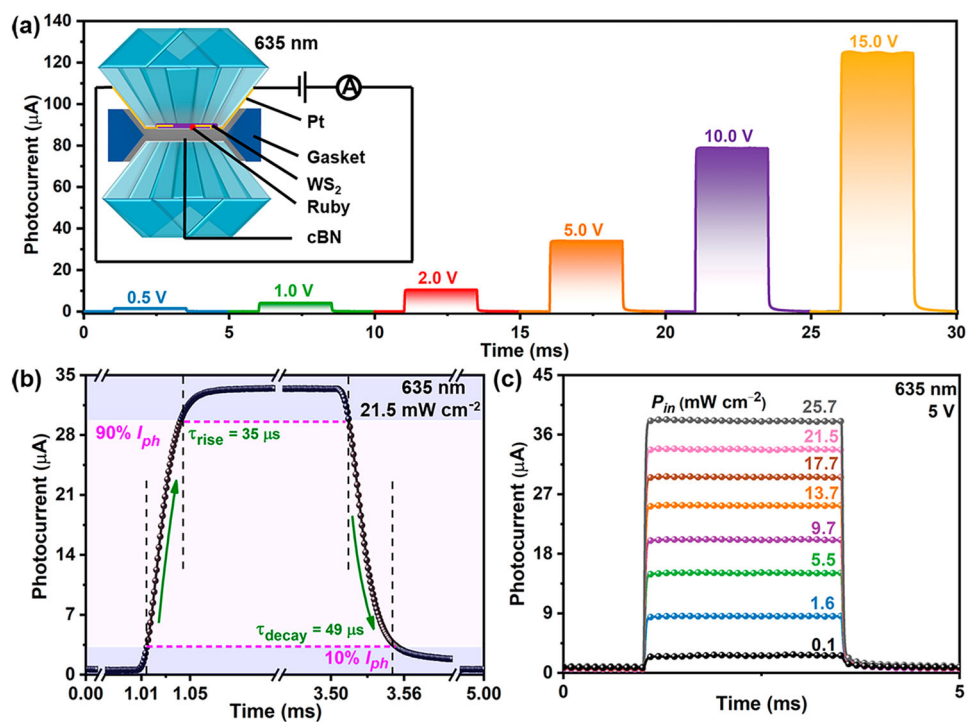


Figure 2. (a) Photocurrents of WS₂ photoelectrical devices dependent on different biases under ambient pressure. The inset is the schematic of our photocurrent measurements. (b) τ_{rise} and τ_{decay} of WS₂ under 635 nm laser illumination at 5 V bias. (c) Photocurrents of WS₂ under different P_{in} .

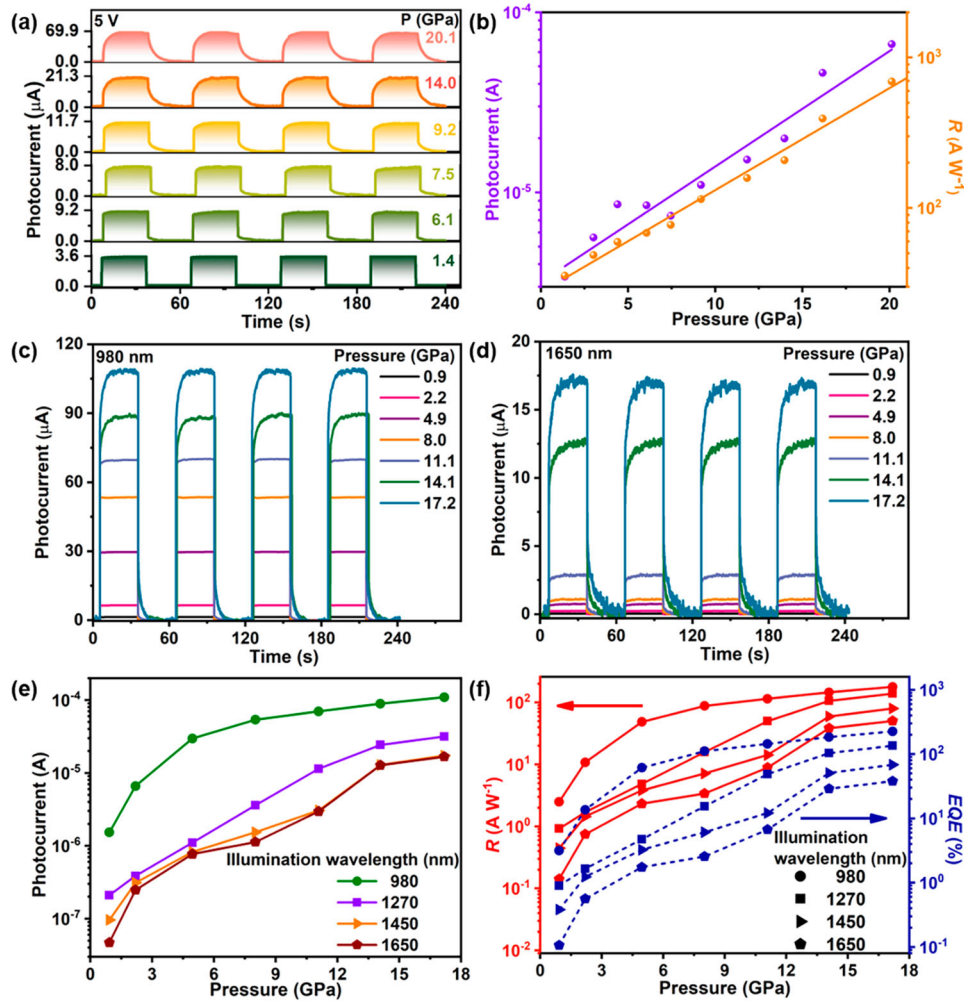


Figure 3. (a) Photocurrents at selected pressures of WS₂ under xenon lamp irradiation. (b) Pressure-dependent photocurrents (left axis) and responsivity (right axis) under xenon lamp irradiation during compression. (c–d) Photocurrents of WS₂ under the illumination wavelength of 980 nm and 1650 nm at selected pressures. (e) Photocurrent–pressure dependence of layered WS₂ with 980, 1270, 1450, and 1650 nm NIR wavelengths. (f) R and EQE of WS₂ as a function of pressure under illumination of selected NIR wavelengths.

observed in other single-crystal TMDs, such as PtS₂ and ReS₂, however, the photocurrent enhancement of WS₂ is much higher than the previous reports [41,42].

Enhancing the response of optoelectronic materials is still an immense challenge for developing the functionalities and applications of high-performance NIR photodetectors. Hence, we used several specific NIR wavelength lasers as the light source, including 980, 1270, 1450, and 1650 nm, as shown in Figure 3(c,d) and Figure S3a and b, to observe the variation in the photoelectric properties of WS₂ under applied pressures up to 17.2 GPa. Similar to the variation observed under xenon lamp illumination, when compared with the photocurrents of WS₂ under ambient pressure (Figure S4), the photocurrent increased significantly upon the application of pressure under illumination of different NIR wavelengths. It is worth mentioning that, as shown in Figure 3(e), in the spectral range of 1450–1650 nm, the pressure-induced

photocurrent increasement reaches up to three orders of magnitude compared with the initial pressure. The external quantum efficiency (EQE) is another important parameter for photoelectric behavior characterization and can be calculated using the relationship

$$EQE = R \frac{hc}{e\lambda} \quad (2)$$

where c is the light velocity, e is the electron charge, and λ is the wavelength of illumination. As shown in Figure 3(f), under the illumination of the selected NIR wavelengths, the photoelectric characteristic parameters R and EQE, like the photocurrent, exhibit a significant increase upon compression. As the pressure increases to 17.2 GPa, WS₂ exhibits an increase of approximately two orders of magnitude in R and EQE in the NIR region relative to the initial pressure. Intriguingly, although the pressure leads to an enhancement of the photoelectric

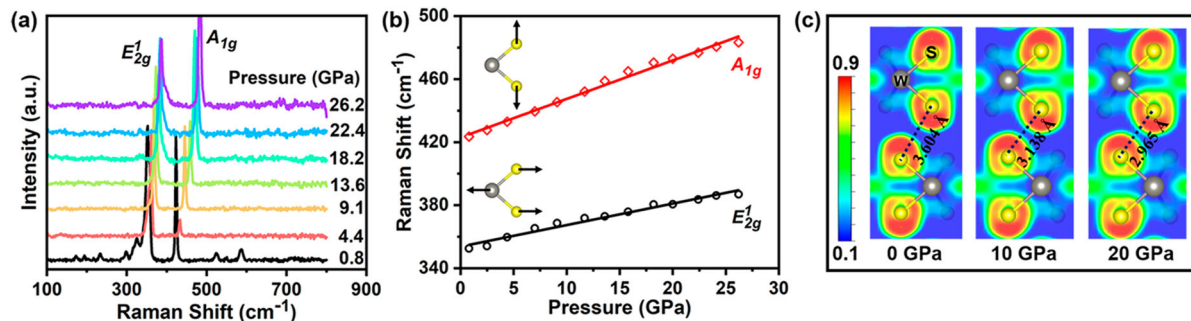


Figure 4. (a) Raman spectra of WS₂ with 514.5 nm laser excitation wavelength at selected pressures. (b) Raman shifts of the E_{2g}^1 and A_{1g} Raman modes with increasing pressure. (c) ELF of WS₂ at selected pressures.

properties throughout the NIR waveband, the photoelectric properties at shorter wavelengths is consistently better than those at longer wavelengths, as shown in Figure 3(e,f). This is in good agreement with the optical absorption properties of WS₂ obtained from our theoretical calculation results (the insets in Figure 5(b,d)). Furthermore, we compared the photocurrent at 0.5 GPa in the compression process with that under ambient pressure after decompression. We surprisingly found that the photocurrent of WS₂ after compression treatment is seven times as large as that at 0.5 GPa (upon compression), as shown in Figure S3c. This indicates that pressure is of great significance as a means of adjusting high-performance infrared photodetectors.

To further understand the mechanism of the pressure-modified photoelectric property enhancement of WS₂, *in-situ* XRD and Raman spectroscopy measurements were conducted to decipher the structural modification upon compression. It can be seen in Figure S5 that when the applied pressure is up to 21.8 GPa, the *in-situ* high-pressure XRD patterns prove that there is no structural phase transition occurring within pressures up to 21.8 GPa, suggesting that the pressure-influenced enhancement of photoelectric properties is not associated with it. To provide insight into the effects of the applied pressure, we investigated the Raman spectra of WS₂. As shown in Figure 4(a), at 0.8 GPa, due to the 514.5 nm excitation wavelength is close to the B excitonic resonance for WS₂, there are several second-order Raman peaks could be observed in the Raman spectrum. As the pressure increases, the B exciton energy shows a blue shift, and the second-order Raman vibration modes disappeared above 4.4 GPa (as shown in Figure S6), which is consistent with the previous literature results [63]. Besides that, there are two obvious first-order Raman peaks at ~ 355 and 421 cm^{-1} that belong to E_{2g}^1 (in-plane vibrational mode of W and S atoms) and A_{1g} (out-of-plane S-atom vibrational mode) during the whole compression process. With an increase

in pressure, these two Raman modes undergo rapid hardening (Figure 4(b)). The slope of the A_{1g} vibration mode is larger than that of the E_{2g}^1 mode, which is not only indicating the A_{1g} mode is more responsive to the external pressure, but also means the S-S interlayer interaction is enhanced under compression. To further understand the chemical bonding, we performed calculations on the electron localization function (ELF), as shown in Figure 4(c). The enhanced interlayer interaction is also evident from the evolution of the S-S interlayer distances that reduce from $3.604\text{ \AA}$ at ambient pressure to $2.965\text{ \AA}$ at 20 GPa. The Raman spectra and ELF calculations reveal that the reduction of the interlayer spacing leads to the enhancement of S-S interlayer interaction upon compression.

To explicitly provide information regarding the orbitals involved in the charge transfer of the band structure, we also performed theoretical calculations of the WS₂ electronic structure under different pressures using the VASP as shown in Figure S7. Upon compression, the bandgap of WS₂ shows a successive decrease due to the broadening of valence band maxima and conduction band minima with the contribution of the W 5d and Se 3p orbitals. Similar to MoS₂ and ReS₂, the enhanced S-S interlayer interaction of WS₂ leads to a reduction in the bandgap during compression [42,47,64]. The narrowed bandgap causes the valence electrons to be more easily excitable by illumination, such that they may cross over the bandgap into the conduction band, resulting in more photo-generated carriers, eventually causing higher photocurrents during compression [42,65].

Evaluating the variations of optical parameters (particularly the absorption coefficient $\alpha(\omega)$ and the photoconductivity $\sigma(\omega)$ obtained by theoretical calculations) with pressure facilitates the further understanding of the photoelectric properties of WS₂ under high pressure. As shown in Figure 5, under ambient pressure, the $\alpha(\omega)$ and $\sigma(\omega)$ is consistent with the experimental results [66,67], which proves the reliability of the

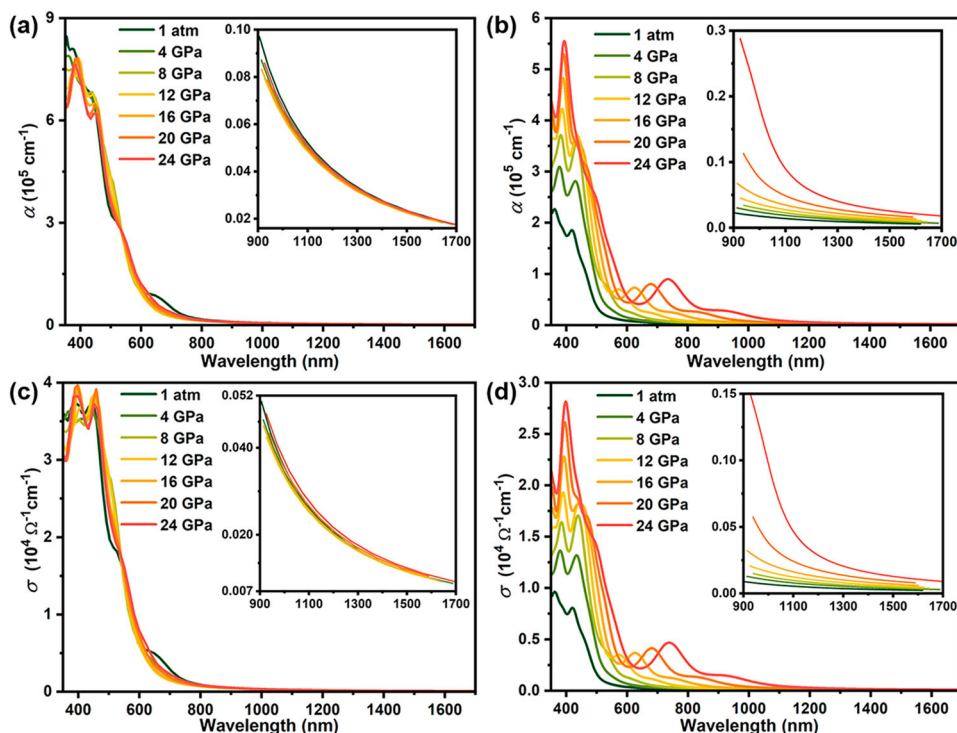


Figure 5. Optical properties of WS₂ at various pressures. (a) Absorption coefficient $\alpha(\omega)$ and (c) the photoconductivity $\sigma(\omega)$ of WS₂ perpendicular to c axis at selected pressures. The (b) $\alpha(\omega)$ and (d) $\sigma(\omega)$ parallel to c axis under pressure, respectively. The four inset images illustrate the changes in the WS₂ optical properties in the NIR waveband.

theoretical calculations. With the increase of pressure, $\alpha(\omega)$ and $\sigma(\omega)$ both increase to varying degrees, which is in accordance with our experimental observation of pressure-induced photocurrent increases. In addition, as shown in Figure S2a, the slope change of I–V curves for WS₂ under xenon lamp irradiation also provides reliable evidence of the increasing trend of $\sigma(\omega)$ with increasing pressure. However, the $\alpha(\omega)$ and $\sigma(\omega)$ are anisotropic, with considerably different values for different incident light polarization directions. Upon compression, the $\alpha(\omega)$ and $\sigma(\omega)$ perpendicular to c axis are almost unchanged (Figure 5(a,c)); however, these two parameters parallel to c axis exhibit a significant enhancement and redshift. These results suggest the experimentally enhanced photoelectric properties of WS₂ during compression is principally attributable to changes in the optical parameters along the c axis, and the red-shifted absorption edge is attributable to the pressure-induced bandgap reduction.

Conclusion

In summary, we investigated the photoelectric properties and structural changes of WS₂ under pressure through *in-situ* XRD, Raman spectroscopy, photocurrent measurements, and theoretical calculations. The WS₂-based photodetector prepared in the pressure cell exhibits an

ultra-fast response speed of $\sim 35 \mu\text{s}$ and a broad photodetection range up to 1650 nm. Moreover, the photoelectronic properties of WS₂ are significantly enhanced from the visible to the NIR range upon the application of pressure. Extensive high-pressure spectroscopic analyses and theoretical calculations reveal that upon compression, more photogenerated carriers are modulated by the decrease in the WS₂ bandgap owing to the enhanced S–S interlayer interaction, leading to significantly enhanced photoelectric characteristics of WS₂. The employment of high pressure as a modulation strategy leads to a remarkable enhancement in the photoelectronic properties of WS₂. Our work provides an experimental and theoretical basis for the practical application of WS₂ as a functional material in future optoelectronic devices.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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