



metal dichalcogenides (TMDs), such as  $\text{MoS}_2$ ,  $\text{MoSe}_2$ ,  $\text{WS}_2$  and  $\text{WSe}_2$ , the energy band structure changes as the number of layers decreases; the band gap changes from a blocky indirect band gap to a direct band gap in a single thin film; and the optical, electrical and mechanical properties also change significantly [5–8]. TMDs can interact directly and strongly with light, with a band gap covering the near-infrared to visible wavelengths, and have promising applications in optoelectronic devices [9–11]. However, the conductivity of TMDs is low [12], and by introducing various highly conductive materials (e.g., carbon materials, MXenes) as conductive networks for compounding with TMDs [13–18]; the interface modulation between conductive networks and TMDs is achieved to fully utilize the advantages of composites [19, 20].

In low-dimensional TMDs materials, the Coulomb interaction in these atomic thin layers is highly enhanced due to the reduced dimensionality and weak dielectric shielding [21]; the ground-state electrons in two-dimensional TMDs materials are excited to jump to the conduction band with valence band holes in Coulomb interaction, form tightly bound excitons (a bound state of one electron and one hole) [22], trions (a bound state of an exciton plus an additional electron or hole) [23, 24], and biexcitons (a bound state of double excitons) [25, 26]. These excitonic complexes have a large binding energy between a few hundred millivolts, and these values are more than an order of magnitude larger than that in conventional III-V-based quasi-2D semiconductor quantum wells (QWs), so they can be easily observed at room temperature and the photoresponse of TMDs is dominated by these excitons [27–32].

In this work, we introduced edge quantum wells (QWs) as electron capture centers through vertical graphene nanosheet-embedded carbon (GNEC) films, and explored the photoluminescence (PL) capability of carbon films combined with monolayer  $\text{WSe}_2$ . In order to study the effect of carbon film on the exciton state in monolayer  $\text{WSe}_2$ , the thin and thick carbon films were manufactured separately for comparison. We also performed photoluminescence measurements related to the incident power to confirm the emission properties of different exciton states, which show a linear increase with increasing excitation power at different slopes. Photoluminescence at different temperatures was also tested to explore the thermal stability of excitons in different bound states with increasing temperature. The geometric limit of monolayer TMDs causes enhanced Coulomb interactions and modulates the many-body exciton state of monolayer TMDs materials through edge traps of graphene carbon films, which can govern the optical properties and carrier transport properties. A microscopic understanding of how excitons are formed from other carriers is essential for elucidating the potential physical mechanism of many-body interactions in TMDs

materials as well as for applications in optoelectronic devices.

## 2 Methods

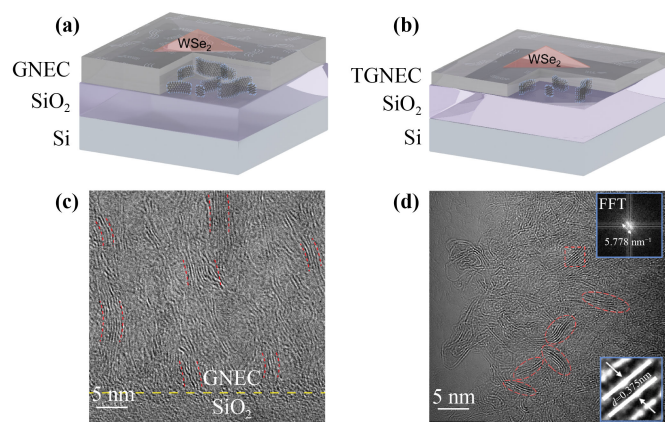
### GNEC film deposition and characterization.

Monolayer  $\text{WSe}_2$  samples were produced by (CVD). Then, monolayer  $\text{WSe}_2$  chip was wet-transferred onto a  $\text{SiO}_2/\text{Si}$  substrate (280 nm thermal oxide on  $n^+$ -doped silicon), with half on a GNEC film and half on a TGNEC film. The GNEC film was deposition by ECR process. The thickness of GNEC film is about 70 nm. A polyimide (PI) film was used as an isolation layer to deposit thin GNEC film. The nanostructures of the carbon films were analyzed by TEM (FEI, Titan3 Themis G2) and AFM (Dimension ICON).

**Optical characterization.** Raman and PL measurements were conducted using a Horiba LabRAM system equipped with a confocal microscope, a charge-coupled device (CCD) Si detector, and a 532 nm diode-pumped solid-state laser as the excitation source. The laser light was focused on the sample surface via a  $50\times$  objective lens (Olympus, LMPLFLN50X). The spectral response of the entire system was determined with a calibrated halogen-tungsten light source. The PL signal was collected by a grating spectrometer, thereby recording the PL spectrum through the CCD (Princeton Instruments, PIXIS). All the PL spectra were corrected for the instrument response. For temperature-dependent measurements,  $\text{WSe}_2/\text{GNEC}$  film chips were put into a Linkam THMS 600 chamber and the temperature was set to a constant (range from 298 to 77 K) during the PL measurements using a low-temperature controller with liquid nitrogen coolant.

## 3 Results and discussion

Vertical graphene nanosheet-embedded carbon (GNEC) films were made using an electron cyclotron resonance (ECR) plasma sputtering system. The ECR plasma sputtering system has been described in detail in our previous work [33–35]. The involvement of low-energy electrons leads to the vertical growth of graphene nanocrystals embedded in amorphous carbon on the substrate. Microwaves with power of 500W are used in the vacuum chamber, followed by plasma generation at an argon pressure of  $4 \times 10^{-2}$  Pa. By applying a symmetrically confined magnetic field to the system to increase the plasma density, the negatively biased carbon target is sputtered by high-speed bombardment of  $\text{Ar}^+$  ions, and the carbon atoms separated by the bombardment move toward the substrate. In the deposition manufacturing process, we divide the  $\text{SiO}_2/\text{Si}$  substrate into two equal areas. Polyimide (PI) film is applied on one side and the other side is left untreated. PI polymer



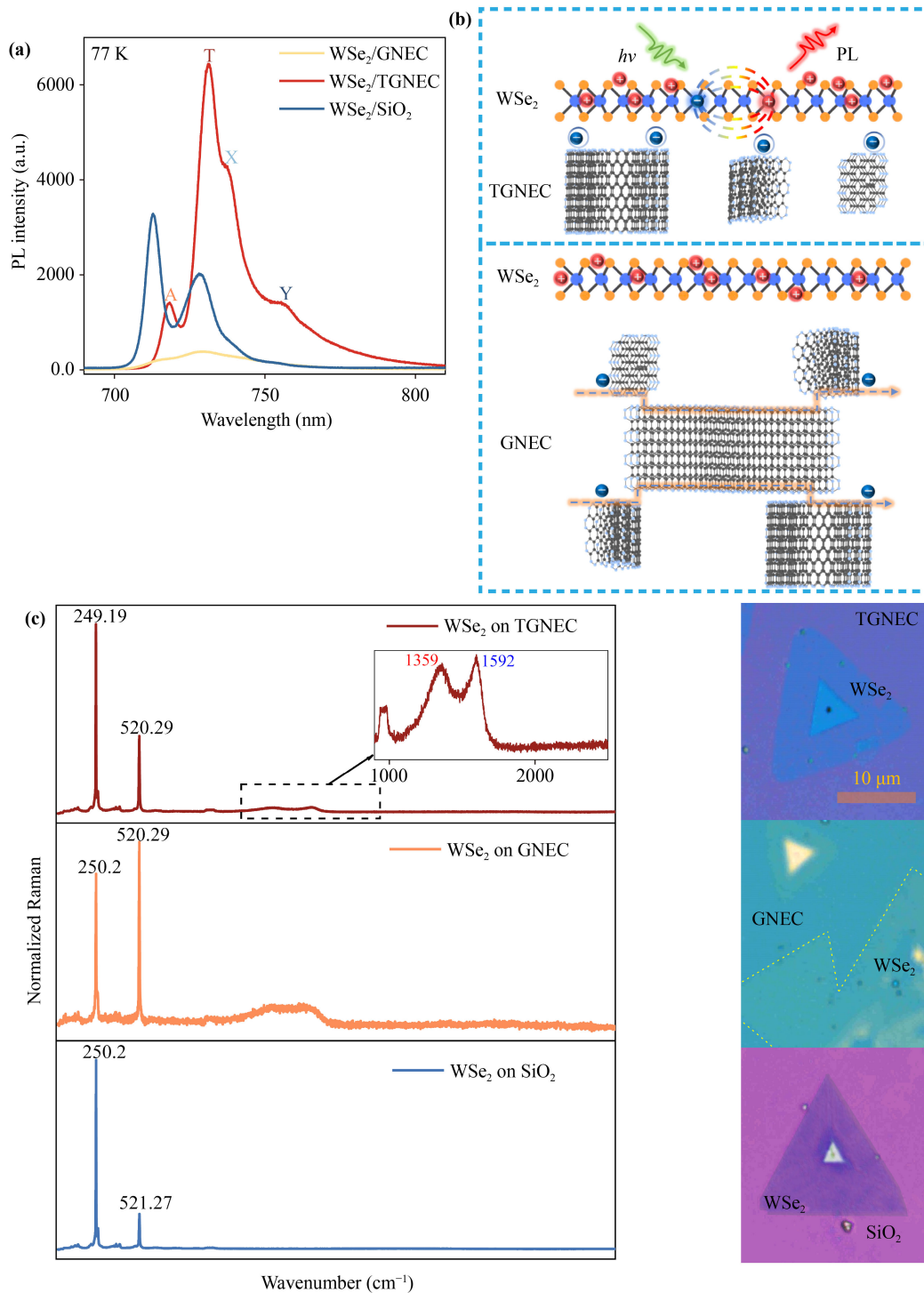
**Fig. 1** (a) Structural model of vertical graphene nanosheet-embedded (GNEC) film. (b) Structural model of vertical thin graphene nanosheet-embedded (TGNEC) film. (c) High-resolution TEM micrographs of 80 V GNEC film cross-section sample. (d) Top-view TEM images of carbon films. Insets show the FFT images and the crystal plane spacing.

film plays a filtering and screening role, carbon atoms will be deposited on the silicon substrate through the polymer film to form a thin graphene nanosheet-embedded carbon (TGNEC) film [Fig. 1(b)], and the other side without treatment will be deposited as a thicker GNEC film [Fig. 1(a)]. Deposition time is set to 30 minutes at a bias voltage of 80 V.

The graphene nanosheets in the thick GNEC film are rich in content [Fig. 1(a)], diverse in morphology and dense, and easy to form a conductive network, with excellent carrier conduction ability. The thin GNEC film contains few graphene nanosheets [Fig. 1(b)], which are scattered and almost non-conductive, and the high density of the edges will capture electrons, which will easily make the monolayer WSe<sub>2</sub> giving rise to excitonic complexes and enhance the photoluminescence of the device. The surface characteristics of GNEC were observed by using transmission electron microscope (TEM). We can clearly observe the presence of graphene nanocrystals from the cross-section [Fig. 1(c)] and the top view [Fig. 1(d)] surface of the carbon film. In addition, the Laue spots are clearly observed from the FFT image of GNEC; the reciprocal lattice distance between the two spots is 5.778 nm<sup>-1</sup>; the crystal plane spacing in this direction is calculated to be  $d = 0.375$  nm; corresponding to the (0001) plane of multilayer graphene, indicating that the nanocrystal is multilayer graphene [36, 37].

WSe<sub>2</sub> samples grown by chemical vapor deposition (CVD) were wet-transferred onto SiO<sub>2</sub>/Si wafers with two different thicknesses of carbon films deposited. WSe<sub>2</sub> is n-doping induced by defect engineering and contains a large number of free electrons inside, which greatly improves its conductivity [16]. The same WSe<sub>2</sub> samples were also transferred on the SiO<sub>2</sub>/Si substrate

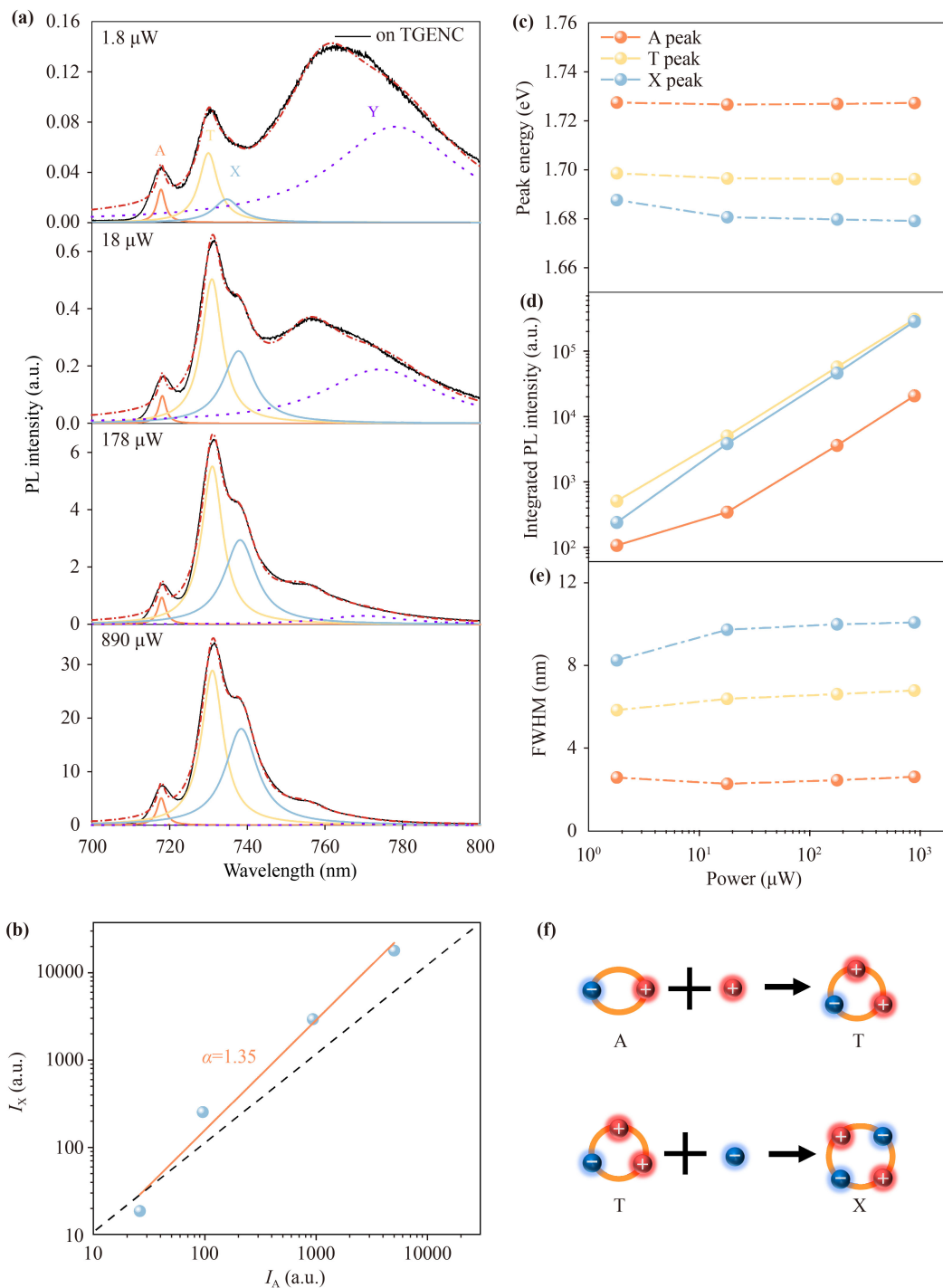
without carbon film growth as a control reference. The PL spectra of the samples were then measured, and the WSe<sub>2</sub>-GNEC samples were kept in a Linkam microscopy-compatible cryostat at 25 °C during the measurements. The liquid nitrogen flows continuously to maintain the low temperature during the tests. The single-layer WSe<sub>2</sub> combined with thick GNEC film emits light weakly [Fig. 2(a)]. Due to the high content of graphene nanocrystals in the thick carbon film, it is prone to form a conductive network [Fig. 2b]. The photogenerated carriers flow away in direct conduction and are not easily formed into emitted photons. The photoluminescence of the WSe<sub>2</sub> sample coupled with a thin carbon film (WSe<sub>2</sub>/TGNEC) is significantly stronger than that of the monolayer WSe<sub>2</sub> on SiO<sub>2</sub> [Fig. 2(c)]. There are four different peaks at wavelengths of 720 nm, 730 nm, 735 nm and 755 nm [Fig. 2(c)], respectively designated as A, T, X and Y peaks. In the following study, we will confirm which exciton complexes are responsible for these characteristic peaks. The highest peak is at 730 nm, which is much larger than the highest peak in conventional WSe<sub>2</sub>, and the A peak is decreased compared to conventional monolayer WSe<sub>2</sub>. The reason for the different peaks in the WSe<sub>2</sub>/TGNEC is the formation of edge quantum wells (QWs) at the high-density edges of GNEC film [Fig. 2(b)] [38], where a large number of electrons are concentrated. The optical response of WSe<sub>2</sub> is also dominated by excitons. We also tested the luminescence ability of different WSe<sub>2</sub> samples, and the highest peak was found to be basically stable at around 745 nm with stable luminescence performance in multiple measurements. The monolayer WSe<sub>2</sub> is heavily p-type doping containing a large number of holes, which combine with photoexcited leaping electrons to form tightly bound excitons, trions and biexcitons. When the energy of the photon is sufficient to excite the electron from the valence band to the conduction band, the photon is absorbed in the semiconductor or insulator. Photoluminescence is simply an opposite process in which electrons in the conduction band recombine with holes in the valence band under the emission of photons. Strong Coulomb interactions due to less charge shielding form special excitonic complexes with large binding energies that remain stable even at room temperature [39–41]. At low temperatures, the emission of bound excitons and charged trions usually dominate the photoluminescence, rather than that of neutral excitons [17]. This means that many-body interactions play a key role in the electron transport and optical leaps in semiconductor materials [42]. As shown in Fig. 2(c), the Raman spectra of WSe<sub>2</sub> samples were measured on GNEC and undeposited GNEC on silicon substrates; and the right side is the state under the microscope. Compared with the Raman spectrum from WSe<sub>2</sub> on SiO<sub>2</sub>/Si substrate, two higher peaks of WSe<sub>2</sub>/TGNEC at 1359 and 1592 nm are observed, indicating the existence of graphene nanosheets.



**Fig. 2** (a) Measured PL spectra from WSe<sub>2</sub>/GNEC (purple line), WSe<sub>2</sub>/TGNEC (red line) and WSe<sub>2</sub>/SiO<sub>2</sub> (blue line) at 77 K. (b) Schematic representation of excitons and the electrons trapped by quantum wells (QWs) for TGNEC. The lower part is GNEC's conductive network. (c) Measured Raman spectra from WSe<sub>2</sub>/TGNEC, WSe<sub>2</sub>/GNEC and WSe<sub>2</sub>/SiO<sub>2</sub> (blue line). The right side is the state under the microscope.

We performed excitation power-dependent PL measurements at 77 K to further investigate the nature of the observed different multi-exciton peaks. The PL spectra [Fig. 3(a)] were fitted by Gaussian function using three peaks: orange (A peak), yellow (T peak) and

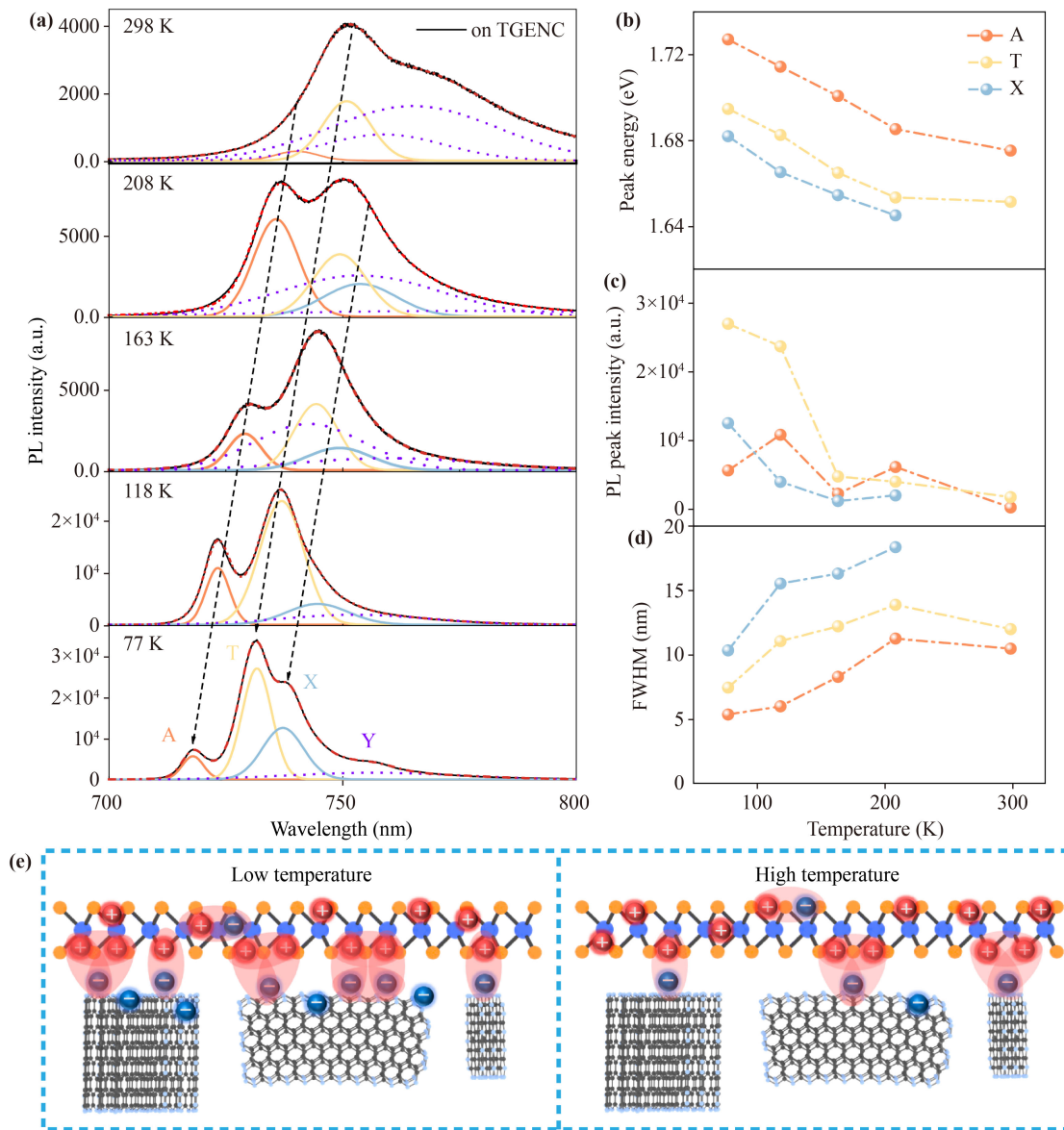
blue (X peak). As shown in Fig. 3(a), the purple dashed line is the fit of the Y peak, and the peak width is much larger than the other three peaks. It is caused by defects of substrate, so it has instability. To confirm the designation of each peak, the integrated PL intensities of both the



**Fig. 3** Power-dependent PL measurements. **(a)** Measured PL spectra from WSe<sub>2</sub>/TGNEC at various excitation powers of the 532 nm laser. Spectrum have been fitted by Gaussian function using three peaks: orange (A peak), yellow (T peak) and blue (X peak). Red line shows the cumulative fit. Purple line represents defect peak. **(b)** log-log plot showing the variation of integrated PL intensity of peak X as a function of peak A. From the fitting curve (orange solid line) the PL intensity of peak X grows superlinearly ( $\alpha \approx 1.35$ ), with the increase of the excitation power. **(c)** Variation of A, T and X peak positions with the increase in laser power. **(d)** Integrated PL intensity values plotted with the increase in laser power. **(e)** Full width at half maximum (FWHM) variation with laser power. **(f)** Representative formation of a trion (T) and neutral biexciton (X).

peaks were plotted with respect to each other on a log-log scale [Fig. 3(b)]; the line is fitted by a power-law relation of the form  $I_X \propto I_A^\alpha$ , where  $I_X$  is the integrated

PL intensity of X peak and  $I_A$  is the integrated PL intensity of A peak, it is found that peak X grows super linearly with the excitation power ( $\alpha \approx 1.35$ ). In the



**Fig. 4** Temperature-dependent PL measurements. **(a)** Measured PL spectra at various temperatures for WSe<sub>2</sub>/TGNEC. **(b–d)** Measured **(b)** peak position, **(c)** peak intensity and **(d)** FWHM of PL peaks A, T and X as a function of temperature in WSe<sub>2</sub>/TGNEC. **(e)** Schematic diagram of exciton binding at low and high temperatures.

ideal case, the PL emission of the exciton state tends to increase linearly (slope  $\alpha \approx 1$ ), and the value of  $\alpha$  for the biexciton emission is expected to be close to 2 [43]. However, the  $\alpha$  value of the exciton state in TMDs is generally less than 1, and the  $\alpha$  value of the biexciton is usually observed between 1.2 and 1.9 [44, 45]. The observed value is less than 2, which may be due to the lack of thermal equilibrium between the exciton states and the limited density of these multiple excitons [44]. According to the fitted  $\alpha$  values, peak A is attributed to exciton emission, peak T is attributed to trion emission, and peak X is attributed to biexciton emission. The shifts of the A and T exciton peak positions are small, while the X peak shows a slight red-shift with increasing

excitation intensity [Fig. 3(c)]. Figure 3(d) shows the absolute integral PL values plotted as a function of laser power. The full width at half maxima (FWHM) of the A peak does not change much overall after a slight decrease at increasing power, while the FWHM of the X peak becomes wider and the FWHM of T peak is almost constant [Fig. 3(e)]. Usually, the PL intensity growth rate with excitation power varies for excitons in different states due to its different physical mechanisms [46]. The way of exciton binding or recombination is shown in Fig. 3(f), electrons captured by the GNEC edge leap into WSe<sub>2</sub> under laser excitation; electrons bind tightly with the majority carrier-holes in WSe<sub>2</sub> to form excitons; some of the excitons recombine with active electrons to

form trions.

To study the thermal stability and activation energy of these different states of excitons, we also test the devices under the same excitation conditions for temperature-dependent PL. The PL spectra [Fig. 4(a)] were fitted by Gaussian function using three peaks: orange (A peak), yellow (T peak) and blue (X peak). At room temperature, we can also clearly observe the existence of trions, which is difficult to observe in general TMDs materials; but the X peak is difficult to be observed at room temperature. It may be attributed to the fact that the WSe<sub>2</sub> samples are heavily p-doped, which are more likely to combine with a small number of electrons at room temperature to form trions. As shown in Figs. 4(a) and (b), the peak position appears to be red-shifted with increasing temperature. When the temperature rises, the peaks of the excitons in all three states begin to decrease to different degrees. The PL intensity of the T and X peaks decreases sharply with increasing temperature [Fig. 4(c)], indicating that the charged exciton is greatly affected by temperature. Our experimental results are in agreement with the monotonic decrease in exciton emission intensity with increasing temperature as expected by other groups [47–49]. The intensity of PL decreases with increasing temperature because the exciton is not tightly bound to the electron in the edge trap [50]. This weaker mutual bonding is easily affected by temperature changes. With decreasing temperature, the peaks of A, T and X all increase significantly, meaning that more exciton complexes form or recombine in WSe<sub>2</sub>. The low temperature leads to a lack of thermal energy for the electrons, so the edge defects of the GNEC trap a large number of electrons [Fig. 4(e)]. It allows free excitons in the two-dimensional space of WSe<sub>2</sub> to locate/capture and radiate recombination, and then emit photons near 750 nm [51]. With increasing temperature, the thermal energy starts to increase and the electrons gain enough energy to avoid the edge trap [Fig. 4(e)]. The electron density at the edge of the carbon film starts to gradually decrease. However, the presence of T peaks can still be clearly observed at room temperature; it indicates that the edge trap of TGNEC has excellent electron trapping ability. The FWHM of the A, T and X peaks generally increase with increasing temperature [Fig. 4(d)]; the FWHM of the A and T peak decreases and then increases at 208 K, which may be caused by the internal

defects of WSe<sub>2</sub>. In conclusion, due to the increased probability of collision of excitons with longitudinal optical phonons, the exciton values in different states are red-shifted with increasing temperature, and the FWHM becomes wider with increasing temperature [51].

In order to explore the binding energies of different states of excitons and to confirm the correct designation of each peak, we calculated the charged trions binding energy ( $\approx 31.15$  meV) and the neutral biexcitons binding energy ( $\approx 48.22$  meV) at 77 K (see Table 1), which is consistent with the trions binding energy of 30 meV [52, 56] and biexcitons energy of 50–70 meV [43, 45, 55] in the monolayer TMDs. The biexciton binding energy is subtracted by the energy value between the exciton and the biexciton peak; it is assumed that the exciton is generated in the radiation decay of the biexciton [55]. The value of biexcitons binding energy ( $\approx 49.2$  meV) for the former exceeds that found in III–V quantum wells by almost two orders of magnitude [57]. Once again, it shows that we are correct in distinguishing the three characteristic peaks of PL. The biexcitons have a large binding energy, and the trions binding energy is much smaller than the observed biexcitons binding energy. In the supporting information, we measured the binding energies of trion and biexciton at different temperatures and excitation powers. The binding energies were found to be in the range of 24–36 meV for trions and 40–50 meV for biexcitons. The trions binding energy was found to be relatively stable with a small range of variation, while the biexcitons binding energy is more susceptible to the effects of temperature and laser power.

## 4 Conclusions

In conclusion, we introduced edge quantum wells (QWs) as electron capture centers through GNEC films. It allows free excitons in the two-dimensional space of WSe<sub>2</sub> to locate/capture and radiate recombination. In monolayer WSe<sub>2</sub>/TGNEC we observed a strong photoluminescence peak ( $\approx 730$  nm) from trions, and the PL intensity is significantly enhanced. We also performed photoluminescence measurements related to the incident power to confirm the emission characteristics of different exciton states. Meanwhile, we found that these many-body excitons have a high binding energy (charged trions  $\approx 31.15$  meV, neutral biexcitons  $\approx 48.22$  meV), which is consistent with experimental results of other subject groups, further indicating that we are correct in distinguishing the exciton states of each peak. Temperature-dependent PL measurements were also performed to explore the thermal stability of excitons in different bound states with increasing temperature. The geometric limits of monolayer TMDs cause enhanced Coulomb interactions and modulate the many-body exciton states of monolayer TMDs through GNEC edge traps, thereby

**Table 1** Comparison of experimental and theoretical TMD excitonic binding energies (meV)<sup>a</sup>.

|            | System | WSe <sub>2</sub>                |
|------------|--------|---------------------------------|
| Experiment | T      | 30 [41, 42], 31.15 <sup>b</sup> |
| Theory     | T      | 30 [43]                         |
| Experiment | X      | 52 [35], 48.22 <sup>b</sup>     |
| Theory     | X      | 59 [43]                         |

<sup>a</sup> Note: A is an exciton, T is a trion, X is a biexciton. <sup>b</sup> This work.

achieving enhanced optical properties and modulation of carrier transport properties. The introduction of carbon nanofilms to regulate the formation and recombination of exciton complexes, it provides a new idea in the field of studying optoelectronic devices of two-dimensional semiconductors.

**Electronic supplementary material** Supplementary materials are available in the online version of this article at <https://doi.org/10.1007/s11467-022-1232-8> and <https://journal.hep.com.cn/fop/EN/10.1007/s11467-022-1232-8>.

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