



four Se atoms in the same layer and two Pd atoms and three S atoms can form a wrinkled pentagon, which is rather rare in the current materials. As one of the most air-stable TMDCs, PdSe<sub>2</sub> shows a high charge carrier mobility and tunable thermal property by external fields, making extensive applications in field-effect transistors, optoelectronic devices [8, 10–13], thermal switches and TE devices [14, 15]. Moreover, the bandgap of PdSe<sub>2</sub> can be changed by engineering its thickness and from bulk to monolayer, its bandgap changes from 0 eV to 1.3 eV [13]. Although PdSe<sub>2</sub> has shown these interesting physical properties, its phonon thermal transport was studied until recently.

Due to its strong interlayer coupling, the thermal conductivity of PdSe<sub>2</sub> shows a thickness-dependent behavior [16–19]. The thermal conductivity of bilayer of PdSe<sub>2</sub> grown by CVD is 36.8 W·m<sup>-1</sup>·K<sup>-1</sup> at room temperature measured by Raman characterization, while the thermal conductivity value of few-layer PdSe<sub>2</sub> is only 10.1 W·m<sup>-1</sup>·K<sup>-1</sup> [18]. Zhang *et al.* [17] used first-principles calculation to study the acoustic velocity, bandgap and density of states of PdSe<sub>2</sub> and demonstrated that the optical modes with low frequencies are increasingly involved in the scattering of acoustic phonons as the number of layers decreases, leading to the decrease of thermal conductivity. Qin *et al.* [19] discovered that monolayer PdSe<sub>2</sub> has a low lattice thermal conductivity around 3 W·m<sup>-1</sup>·K<sup>-1</sup> along the *x* direction at room temperature by the first-principles calculation. Wang *et al.* [20] proposed that PdSe<sub>2</sub> can achieve a continuously switchable thermal conductivity through strain-driven structural phase transition. Moreover, due to the low symmetrical pentagonal fold structure, PdSe<sub>2</sub> has an anisotropy in lattice structure and thus an anisotropic transport properties [21], and the thermal transport anisotropy ratio as high as 1.42 is demonstrated by micro-Raman thermometry [16].

In semiconductor materials, phonon is the main carriers of heat and its behaviors have a great effect on the thermal conductivity [22–25]. Defects, like vacancies, impurities and dislocations are inevitably introduced in the sample preparation process, and the defect engineering could be further used to modulate the phonon transport. Many studies have used “defect engineering” to regulate the thermal conductivity of 2D materials and investigated the influence of defects on heat transport [26–28]. By controlling the oxygen plasma exposure time, Aiyiti *et al.* [29] tuned the thermal conductivity of MoS<sub>2</sub> to a desired value and observed the transition from crystal to amorphous. Zhao *et al.* [30] studied the change of thermal conductivity of MoS<sub>2</sub> flakes with different defect concentrations under helium ion (He<sup>+</sup>) irradiation, and it showed that the Mo vacancy could significantly impede phonon transport compared to that of S vacancy. Wu *et al.* [27] used extensive molecular dynamics simulations to calculate the effects of different types and doses of defects on thermal conductivity of BN and the relationship

between phonon-phonon scattering term and phonon-defect scattering term was proposed. For PdSe<sub>2</sub>, there exist a wide range of binary phases like Pd<sub>17</sub>Se<sub>15</sub>, Pd<sub>7</sub>Se<sub>4</sub> and Pd<sub>4</sub>Se, and by laser irradiation, electron beam irradiation, electric field and so on, PdSe<sub>2</sub> is expected to undergo phase transition [14, 15]. Among these phases, it has been shown that Pd<sub>17</sub>Se<sub>15</sub> is metallic phase and has superconducting behavior, which could be obtained by defect engineering and therefore provides an appropriate approach for engineering phonon transport in PdSe<sub>2</sub> [13, 31].

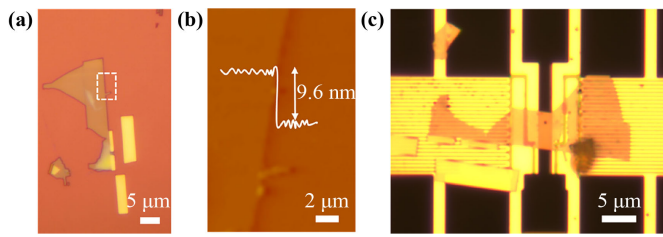
In this work, we study the phonon thermal transport in both intrinsic and defected PdSe<sub>2</sub>. By wet transfer method, few-layer PdSe<sub>2</sub> flake was placed onto a micro-electro-thermal systems (METS) device and the thermal bridge method was employed to measure the thermal conductivity of PdSe<sub>2</sub>. The Ar<sup>+</sup> was further employed to introduce defects into PdSe<sub>2</sub>. With increasing the Ar<sup>+</sup> dose, the thermal conductance of PdSe<sub>2</sub> kept decreasing until to a critical dose, where the thermal conductance dropped dramatically by 50%. During the Ar<sup>+</sup> irradiation, Raman characterization was carried out as well to record the lattice structure evolution of defected PdSe<sub>2</sub>, where a possible phase change at the critical dose was observed, corresponding to the change observed in the thermal conductivity. Our work provides a novel approach to tuning the thermal transport in novel 2D materials, enabling further thermal management at nanoscale.

## 2 Methods

### 2.1 Preparation of suspended-pad micro-devices

Bulk PdSe<sub>2</sub> single crystals were synthesized by a self-flux method as previous reported [32]. Unlike graphene and boron nitride, whose layers are connected by weak van der Waals forces [33, 34], it is challenging to acquire few-layer PdSe<sub>2</sub> by mechanical exfoliation due to its strong layer to layer coupling [35]. By using Ar<sup>+</sup> or O<sup>2+</sup> to bombard clean SiO<sub>2</sub>/Si substrates, the interaction force between PdSe<sub>2</sub> flakes and substrates was enhanced, and the flake with desired thickness was further obtained. Before transferring onto the pre-patterned suspended thermal bridge device, the atomic force microscope (AFM) is employed to measure the thickness of samples. The thickness of measured PdSe<sub>2</sub> sample is 9.6 nm, as shown in Figs. 1(a) and (b).

For the thermal bridge method measurement, there are typically three methods to transfer samples to the devices, including drop-cast method [36], dry/wet transfer method [37] and nano-manipulation method [38]. Considering the operative difficulty and yield, the wet transfer method was used in this study. The substrates with targeted samples were spin-coated with PMMA at



**Fig. 1** (a) Optical image and (b) AFM image of PdSe<sub>2</sub>. The thickness of the sample is 9.6 nm. (c) Optical image of transferred METS sample. Scale bars have been shown in each figure.

the rate of 2000 raps/s with duration of 30 s. Then, the substrates were heated on the hot plates at 100 °C to accelerate the solidification process. The substrates coated with PMMA were then immersed into 20% KOH solution. Under an optical microscope, the marked sample on the suspended PMMA film region was placed on top of the middle of the thermal bridge. Before using acetone to dissolve PMMA, the devices were heated to 120 °C for twenty minutes to well stick the flake with the thermal bridge electrodes. Finally, the critical point dryer (CPD) was used to remove the acetone and the final METS device is shown in Fig. 1(c).

## 2.2 Thermal bridge measurement

In 2001, the thermal bridge method was firstly used to measure the thermal conductivity of micro- and nano-scale samples [39]. This method can measure not only the thermal conductivity, but also the electric conductivity of nanosheets and nanowires [40–42]. A METS device has two suspended SiN<sub>x</sub> membranes with integrated Pt heaters/resistance-thermometers and extra electrodes. One pad is treated as heater and the other sensor. Each pad is connected with six suspended cantilevers to avoid the influence of substrate. During the measurement, a direct current (DC) produced by Keithely 6221 current source meter flows into heater pad to act as a heat source. One part of the heat ( $Q_1$ ) flows through the suspended cantilevers to the substrate, and the other part ( $Q_2$ ) flows through the sample to the sensor.  $Q_1$  and  $Q_2$  can be expressed as

$$Q_1 = G_b \times \Delta T_h, \quad (1)$$

$$Q_2 = G_s \times (\Delta T_h - \Delta T_s) = G_b \times \Delta T_s, \quad (2)$$

where  $G_b$  is the thermal conductance of heater membrane.  $G_s$  is the thermal conductance of the sample.  $\Delta T_h$  and  $\Delta T_s$  is temperature rise of heater and sensor. Under a thermal steady state,  $G_b$  and  $G_s$  are derived as

$$G_b = \frac{I^2 \times R_h + \frac{1}{2} I^2 \times R_L}{\Delta T_h + \Delta T_s}, \quad (3)$$

$$G_s = \frac{\left( I^2 \times R_h + \frac{1}{2} I^2 \times R_L \right) \times \Delta T_s}{\Delta T_h^2 - \Delta T_s^2}, \quad (4)$$

where  $R_h$  and  $R_L$  respectively represent the resistance of heater pad and suspended cantilevers. When applying a direct current (DC), the current source meter Keithely 6221 produces a tiny alternating current (AC) at the same time and the lock-in amplifier SR 830 is employed to measure the change of resistance of heater and sensor. Thus, the temperature change is obtained and the thermal conductance of the sample is calculated. During measurement, the thermal bridge device is placed in a high vacuum thermostat with a pressure less than  $10^{-5}$  mbar, which avoids the effect of thermal convection on the measurement. Two Cu covers were employed to reduce the possible thermal radiation inside the thermostat. As shown in Fig. 2(a), the resistance of Pt loops and the temperature coefficient of resistance (TCR) of the measured device can be acquired. Combining Eqs. (3) and (4),  $G_s$  and  $G_b$  can be obtained, and the thermal conductivity of PdSe<sub>2</sub> is further calculated by the following equation:

$$\kappa = G_s \frac{L}{S} = G_s \frac{L}{w \times t}, \quad (5)$$

where  $L$ ,  $w$ , and  $t$  are the length, width, and thickness of the suspended part of sample, which are 2 μm, 6.5 μm, and 9.6 nm, respectively.

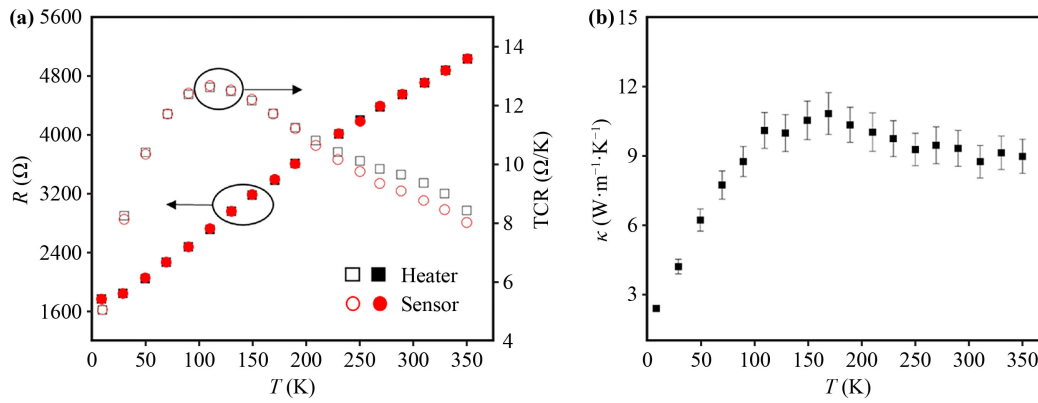
The measurement errors include the temperature drift in the thermostat, the error of sample dimensions, the error in the contact thermal resistance and so on, which can be expressed as

$$\frac{\delta \kappa}{\kappa} = \sqrt{\left( \frac{\delta G}{G} \right)^2 + \left( \frac{\delta L}{L} \right)^2 + \left( \frac{\delta W}{W} \right)^2 + \left( \frac{\delta t}{t} \right)^2 + \left( \frac{\Delta R_c}{R_c} \right)^2}, \quad (6)$$

where  $R_c$  is the thermal resistance between samples and thermal bridge devices. Combining the above factors, the uncertainty of the thermal conductivity is obtained accordingly.

## 3 Results and discussion

By using the thermal bridge method, the temperature dependent thermal conductance was obtained as shown in Fig. 2(b). The thermal conductivity of PdSe<sub>2</sub> at room temperature is  $9.4 \pm 1.1 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ , consistent with the theoretical calculation [17]. At low temperature, the phonon Umklapp scattering is weak, the mean free path of phonons (MFP) of PdSe<sub>2</sub> is mainly limited by defects, boundaries, and so on [43, 44]. Impurities like PMMA residues during the transfer process could contribute as well. As the temperature rises, more and more phonons



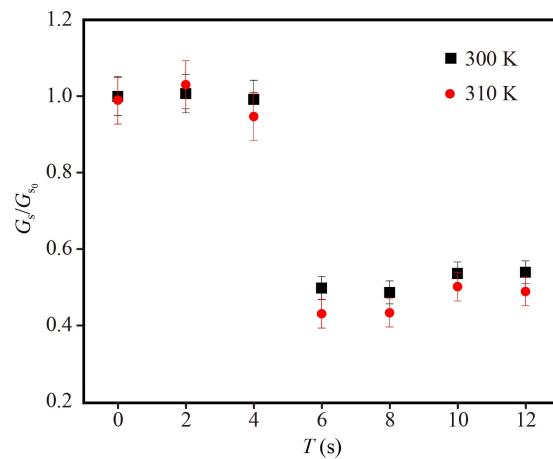
**Fig. 2** (a) Measured temperature-dependent resistance of heater and sensor and the calculated TCR. (b) Intrinsic thermal conductivity of PdSe<sub>2</sub>. The extracted thermal conductivity ranges from  $(9.4 \pm 1.1) W \cdot m^{-1} \cdot K^{-1}$  at room temperature.

are excited and the thermal conductivity of PdSe<sub>2</sub> rises. With further increasing the temperature, the thermal conductivity of PdSe<sub>2</sub> reaches a maximum value, which is  $(10.8 \pm 0.9) W \cdot m^{-1} \cdot K^{-1}$  at a temperature of 170 K as shown in Fig. 2(b). At even high temperature, the phonon Umklapp scattering is enhanced and MFP of phonons is reduced, resulting in decreasing thermal conductivity [37, 45].

In order to tune the thermal conductivity of few-layer PdSe<sub>2</sub>, the Plasma etching machine was used to introduce defects into PdSe<sub>2</sub>. Here, we chose Ar<sup>+</sup> with a radio-frequency (RF) power  $\sim 4$  W to well control the concentration of defects. We followed the assumption that per unit time generates the same amount of plasma and the ionized ions uniformly bombard the sample [29]. The measured thermal conductance change with the irradiation time is summarized in Fig. 3, which can be divided into the following parts, as discussed below. Here, we focus on the thermal conductance measured at room temperature.

1)  $t < 4$  s: Due to the low Se-Se intralayer bonding energy, PdSe<sub>2</sub> is sensitive to defects [13]. As shown in Fig. 3, when the irradiation time was less than 4 s, there was a slight fluctuation in the thermal conductance with increasing the dose. In the meantime, the morphology of the samples did not change under the optical microscope, implying that the thickness of the sample keeps unchanged. The fluctuation of thermal conductance was possibly attributed to both the introduced defects and the removal of impurities. During irradiation, the enhanced phonon-defect scattering resulted in the reduction of thermal conductivity. While some organic impurities may remain on the sample in the transfer process, which can be removed by ionized particle during the bombardment. Thus, it is reasonable that the thermal conductance shows a tiny fluctuation.

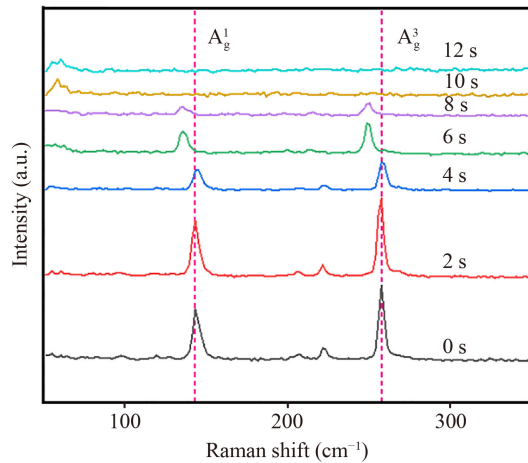
2)  $t > 4$  s: When the irradiation time was increased to 6 s, the thermal conductance of the sample had a sharp drop around 50%. With further increasing the irradiation



**Fig. 3** The thermal conductance of PdSe<sub>2</sub> versus the Ar<sup>+</sup> irradiation time at 300 K and 310 K. The thermal conductance of PdSe<sub>2</sub> decreases with an increase in the defect concentration.

time, more Se vacancies were created and got concentrated. In this process, the defected PdSe<sub>2</sub> would collapse into the other stable phase. According to paper [13], for PdSe<sub>2</sub> under Ar<sup>+</sup> irradiation, there exists a phase transition from PdSe<sub>2</sub> to Pd<sub>17</sub>Se<sub>15</sub>. For the metallic phase Pd<sub>17</sub>Se<sub>15</sub>, the thermal conductance would reach a saturated value as the irradiation time increased further.

In order to further understand the effect of defects on phonon transport, the Raman spectrometer is employed to explore the lattice structure changes of PdSe<sub>2</sub> under the introduction of defects. After each thermal measurement under different Ar<sup>+</sup> irradiation, the Raman characterization was performed. The evolutions of Raman spectrum are summarized in Fig. 4. The PdSe<sub>2</sub> with a thickness of 9.6 nm showed two sensitive peaks, A<sub>g</sub><sup>1</sup> and A<sub>g</sub><sup>3</sup>, which were  $\sim 145$  and  $\sim 255$  cm<sup>-1</sup>, respectively, consistent with the previous experiments [46]. A<sub>g</sub><sup>1</sup> is connected with the in-plane vibration of covalent bond between Pd and Se atoms, and A<sub>g</sub><sup>3</sup> is related to the in-plane vibration of



**Fig. 4** The evolution of Raman sensitive peaks,  $A_g^1$  and  $A_g^3$ , for few layer PdSe<sub>2</sub> flakes under various irradiation doses.

two neighboring covalently bonded Se atoms. As the irradiation time increased on a time scale of 0 to 4 seconds, the intensity of these two Raman peaks gradually decreased. The positions of the two modes did not change, indicating that the crystal structure of the sample kept constant. This change coincided with a slight fluctuation in the thermal conductivity. When the time exceeds 4 s, the modes of  $A_g^1$  and  $A_g^3$  showed a slight red-shift and with further increasing the irradiation time, these two peaks almost vanished and at the same time, the thermal conductance reaches a saturated value. The disappearance of Raman peaks suggested the possible phase transition from PdSe<sub>2</sub> to Pd<sub>17</sub>Se<sub>15</sub>. As shown in the work conducted by Akinola *et al.* [13], under the Ar<sup>+</sup> irradiation, semiconductor PdSe<sub>2</sub> could change into stable metallic Pd<sub>17</sub>Se<sub>15</sub>, which could be characterized by low-frequency anti-Stokes peaks (below 50 cm<sup>-1</sup>). Further study would be carried out to perform this Raman characterizations for the defected PdSe<sub>2</sub>.

## 4 Conclusion

In summary, we experimentally studied the effect of Ar<sup>+</sup> on phonon transport in few-layer PdSe<sub>2</sub>. By using a wet transfer method, the exfoliated few-layer PdSe<sub>2</sub> flakes were transferred onto the pre-patterned suspended thermal bridge devices and the intrinsic thermal conductivity of PdSe<sub>2</sub> was measured by the thermal bridge method. By Ar<sup>+</sup> irradiation, defects with different concentrations were introduced into the PdSe<sub>2</sub> sample. When the irradiation time is 6s, the thermal conductance of the sample had a sharp drop. Raman spectroscopy was employed to characterize the structure of defected PdSe<sub>2</sub>, where a phase transition was observed. Our work provides a convenient approach to engineering the thermal transport in 2D layered materials by defects engineering, which can be applied to a broad class of emerging 2D materials

for engineering their thermal properties. By introducing defects, the thermal conductivity of PdSe<sub>2</sub> is modified, shedding light on the applications of PdSe<sub>2</sub> in thermal management, TE energy conversion and so on.

**Declarations** The authors declare that they have no competing interests and there are no conflicts.

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