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111Equation Chapter 1 Section 1Optical and Electrical Properties of Two-Dimensional Palladium Diselenide

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Two-dimensional (2D) noble-metal dichalcogenides exhibit exceptionally strong thickness dependent-bandgaps, which can be leveraged in a wide variety of device applications. Detailed study of their optical (e.g. optical bandgaps) and electrical properties (e.g. mobilities) is important in determining potential future applications for these materials. In this work, we perform detailed optical and electrical characterization of 2D PdSe₂ nanoflakes mechanically exfoliated from a single-crystalline source. Layer-dependent bandgap analysis from optical absorption results indicates that this material is an indirect semiconductor with a bandgap of approximately 1.37 and 0.50 eV for monolayer and bulk, respectively. Spectral photoresponse measurements further confirm these bandgap values. Moreover, temperature-dependent electrical measurements of a 6.8-nm-thick PdSe₂ flake-based transistor shows effective electron mobilities of 130 and 520 cm² V⁻¹ s⁻¹ at 300 K and 77 K, respectively. Finally, we demonstrate that PdSe₂ can be utilized for short-wave infrared photodetectors. A room-temperature specific detectivity (D^*) of 1.8×10^{10} cm Hz^{1/2} W⁻¹ at 1 μ m with a band edge at 1.94 μ m is achieved on a 6.8-nm-thick PdSe₂ flake-based photodetector.

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I. INTRODUCTION

Two-dimensional (2D) transition metal dichalcogenides (TMDCs) have attracted considerable attention and have been explored for numerous applications including electronics, optoelectronics, catalysis, and sensors in the last decade.¹ Motivated by the attractive properties and wide applications of TMDCs, researchers have explored other 2D two-element systems including the 2D noble-metal dichalcogenides (NMDCs) with the general formula MX_2 (where $\text{M}=\text{Pt}$, Pd and $\text{X}=\text{S}$, Se , Te).² NMDCs have recently been synthesized and demonstrated to exhibit air stability and strongly thickness-dependent bandgaps. For example, PtSe_2 and PdS_2 exhibit a sharp thickness-modulated semiconductor-to-metal transition,³⁻⁵ whereas PtS_2 also shows strong thickness dependence but is a narrow-bandgap indirect semiconductor in bulk.⁶ PtTe_2 and PdTe_2 are both semimetals,^{7,8} with the latter exhibiting superconductivity.^{9,10}

Palladium diselenide (PdSe_2), a NMDC, has also been synthesized and explored in the last few years. Field-effect transistors based on PdSe_2 flakes show electron mobilities up to $\sim 200 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$.¹¹ PdSe_2 was experimentally reported to have a thickness-dependent bandgap, in which monolayer PdSe_2 has a bandgap of $\sim 1.4 \text{ eV}$ and bulk PdSe_2 is metallic with a bandgap approaching 0 eV .¹² More recently, PdSe_2 has been investigated for infrared detectors.¹³ In this work, we systematically study the optical and electrical properties of PdSe_2 with an emphasis on its thickness-dependent bandgap to determine future applications for this material. Our results show that PdSe_2 is an indirect semiconductor with a monolayer bandgap of 1.37 eV and a bulk bandgap of 0.5 eV (in contrast to previous works which have predicted metallic bulk), as shown by optical absorption and photoresponse measurements. Furthermore, the temperature-dependent electrical measurements

of a 6.8-nm-thick PdSe₂ flake-based transistor shows effective electron mobilities of 130 and 520 cm² V⁻¹ s⁻¹ at 300 K and 77 K, respectively. Owing to its relatively small bandgap, thick PdSe₂ can be utilized for short-wave infrared (SWIR) photodetectors. A room-temperature peak specific detectivity (D^*) of 6.4×10^{10} cm Hz^{1/2} W⁻¹ at 0.7 μm with a band edge at 1.94 μm is achieved on a 6.8-nm-thick PdSe₂ flake-based photodetector.

II. RESULTS AND DISCUSSION

The crystal structure of PdSe₂ is shown in Figures 1(a)–1(b). PdSe₂ is a layered 2D material with a theoretical monolayer thickness of ~4.8 Å. PdSe₂ also has pentagonal-structured layers,^{11,12} as shown in Figure 1(b). Pentagonal 2D materials have been theoretically proposed,^{14,15} though PdSe₂ has been synthesized and experimentally characterized as a 2D material. Figure 1(c) shows the measured X-ray diffraction (XRD) pattern of a bulk PdSe₂ crystal grown by chemical vapor transport (CVT), which is consistent with the simulated XRD pattern, confirming the synthesis of bulk single crystal PdSe₂. 2D PdSe₂ nanoflakes of varying thickness were mechanically exfoliated from a large single-crystalline source. Figure 1(d) shows the Raman spectra of PdSe₂ flakes of thicknesses ranging from monolayer to bulk. The locations of the Raman modes A_g^1 - B_{g1}^1 , A_g^2 , B_{g1}^2 , A_g^3 , and B_{g1}^3 and their shifts with changing thickness are consistent with previous reports on PdSe₂.¹² Figure 1(e) shows the high-resolution transmission electron microscope (HR-TEM) image of a 6.8-nm-thick PdSe₂ flake. The lattice constant is 0.29 nm, which can be assigned to the (200) planes of the PdSe₂ single crystal. The corresponding selected area electron diffraction (SAED) pattern shows a single set of bright diffraction dots (Figure 1(f)), further confirming the single crystalline nature.

To perform optical measurements, PdSe₂ flakes were exfoliated onto quartz by mechanical exfoliation and gold-mediated exfoliation,¹⁶ which can yield large-area mono- and few-layer PdSe₂ flakes. Optical transmittance and reflectance measurements were conducted in both a UV-Vis micro-absorption setup as well as a Fourier-transform infrared microscope. The combination of these techniques permits measurements over a wavelength range of 450 nm to 15 μm.¹⁷ UV-Vis measurements were performed with reference to a blank quartz substrate and a silver mirror (Figures 2(a)–2(b)), while FTIR measurements were performed with reference to a blank quartz or KBr substrate and a gold mirror (Figures 2(c)–2(d)). Quartz is optically transparent for visible to

SWIR wavelengths, whereas the transparency of KBr extends further a few micron beyond LWIR wavelengths. Note that the reflectance and transmittance of thick PdSe₂ flakes show out-of-phase oscillations due to multiple reflections occurring in the 2D film; this destructive and constructive interference behavior is accounted for when calculating the absorption coefficient, as shown more clearly in Figure S1 in the supplementary material. We did not observe differences in transmittance and reflectance for PdSe₂ flakes prepared by mechanical exfoliation and gold-mediated exfoliation. To account for thin-film interference in the PdSe₂ flake, we used the transfer matrix method. For transmission and reflection of light by a thin film on a substrate, we define the Fresnel coefficients:¹⁸

$$\begin{aligned} r_1 &= \frac{n_0 - n_1}{n_0 + n_1}, & t_1 &= \frac{2n_0}{n_0 + n_1}, \\ r_2 &= \frac{n_1 - n_2}{n_1 + n_2}, & t_2 &= \frac{2n_1}{n_1 + n_2} \end{aligned} \quad (1)$$

where $n_0 = 1.00$, $n_1 = n - ik$, and $n_2 = 1.46$ are the refractive indices of air, the PdSe₂ flake, and quartz, respectively. The reflected and transmitted amplitudes are:

$$R = \frac{r_1 + r_2 e^{-2i\phi_1}}{1 + r_1 r_2 e^{-2i\phi_1}}, \quad T = \frac{t_1 t_2 e^{-i\phi_1}}{1 + r_1 r_2 e^{-2i\phi_1}} \quad (2)$$

where $\phi_1 = (2\pi/\lambda)n_1 d_1$ and d_1 is the thickness of the PdSe₂ flake.¹⁸ The reflectance and transmittance as ratios of the energy reflected and transmitted to the energy incident are:¹⁸

$$\mathbf{R}_{\text{sim}} = \mathbf{R}\mathbf{R}^*, \quad \mathbf{T}_{\text{sim}} = \frac{n_2}{n_0} \mathbf{T}\mathbf{T}^* \quad (3)$$

We then used the Nelder-Mead algorithm to minimize $(\mathbf{R}_{\text{meas}} - \mathbf{R}_{\text{sim}})^2 + (\mathbf{T}_{\text{meas}} - \mathbf{T}_{\text{sim}})^2$ to estimate the complex refractive index, $n_1 = n - ik$, of the PdSe₂ flake.^{19,20} Finally, we extracted the optical bandgap of the PdSe₂ flake from a Cody plot of $(\epsilon''/h\nu)^{1/2}$ versus $h\nu$, where $\epsilon'' = 4\pi k/\omega$ is the

absorption coefficient of the PdSe₂ flake,¹⁹ as shown in Figure 2(e) for PdSe₂ flakes of varying thickness. The optical microscope images of these flakes along with their thicknesses measured via atomic force microscopy (AFM) are shown in Figure S2 in the supplementary material. Figure 2(f) shows the measured optical indirect bandgap of PdSe₂ as a function of its thickness, which agrees with theoretical calculations in terms of the monolayer bandgap, but deviates from previously reported experimental data in terms of the bulk bandgap.¹² This experimental deviation is likely due to both accounting for thickness-dependent reflectance (in addition to thickness-dependent transmittance) as well as the broader spectral range (~0.35–3.1 eV) measured in this work, which fully encompasses the bandgap range of PdSe₂. A previous work on PdSe₂ extrapolated optical bandgap values from a measured spectral range of ~1.3–3.25 eV, which is nearly a whole eV beyond the bulk bandgap of PdSe₂.¹² Accounting for thin-film interference only changes the bandgap by ~0.1 eV (supplementary material Figure S1),¹² whereas neglecting reflectance and extrapolating far beyond the bandgap can drastically alter the bandgap value (supplementary material Figure S3). This can be clearly seen for reflection and transmission measurements performed on a 134-nm-thick PdSe₂ flake which shows an absorption edge around 2 μm (supplementary material Figure S4).

Using electron-beam lithography, back-gated PdSe₂ field-effect transistors (FETs) were fabricated on 90 nm SiO₂/Si with 100-nm-thick nickel (Ni) contacts. The device structure and optical microscopy images of fabricated devices are shown in Figure 3(a). Figure 3(b) shows the temperature-dependent I_d - V_g characteristics of the 6.8-nm-thick PdSe₂ device measured at a low drain voltage V_d of 10 mV, indicating that the on/off ratio increases as temperature decreases. The inset shows a plot of the same data in linear- I_d scale, clearly showing that the drain-source current $I_{d,max}$ increases with decreasing temperature. We can therefore expect the mobility of this 6.8-nm-thick PdSe₂ flake to reach its maximum at low temperatures. The I_d - V_g shows ambipolar conduction since the onset of n -type and p -type conduction is roughly centered around $V_g = 0$ V, suggesting that the material is roughly intrinsic (*i.e.* low background doping). The ambipolar characteristics of PdSe₂ can be drastically tuned by introducing molecular dopants to PdSe₂ while annealed under vacuum.¹¹ The same temperature-dependent I_d - V_g plot is shown in Figure S5 in the supplementary material but with a second measurement of the device at $T = 300$ K taken after 9 months of

storage in nitrogen and ambient air, demonstrating the air stability of PdSe₂. From linear- I_d - V_g plots, we extrapolate room-temperature threshold voltages V_t of 2.25 V and 4.45 V for the 6.8-nm- and 116-nm-thick PdSe₂ devices, respectively.

Figure 3(c) shows the I_d - V_d characteristics of the 6.8-nm-thick PdSe₂ device inset with those of the 116-nm-thick PdSe₂ device. Since the drain saturation voltage $V_{d,sat} = V_g - V_t$ exceeds the range of measurement, we do not observe saturation. Additionally, the linear behavior of the I_d - V_d characteristics at low values of V_d suggests minimal contact resistance with small Schottky barrier heights. From these transfer characteristics, we calculated the effective electron mobility, $\mu_{eff} = (dI_d/dV_d)(L_g/w_c)[C_{ox}(V_g - V_t - 0.5V_d)]^{-1}$ and field-effect electron mobility $\mu_{fe} = (dI_d/dV_g)(L_g/w_c)(C_{ox}V_d)^{-1}$ of the 6.8-nm-thick PdSe₂ device versus temperature, as shown in Figure 3(d). The electron mobility of the 6.8-nm-thick PdSe₂ device increases with decreasing temperature. The 6.8-nm-thick PdSe₂ device exhibits effective electron mobilities of 130 and 520 cm² V⁻¹ s⁻¹ at 300 K and 77 K, respectively. We can fit the temperature-dependent mobility of this device with a power law, $\mu_{eff} \propto T^{-\square}$ where $\square = 1.08$, suggesting that the mobility is limited by phonon scattering.^{21,22} Finally, we extracted a lower bound on the bandgap of a 6.8-nm-thick PdSe₂ flake from an Arrhenius plot of the minimum drain-source current $I_{d,min}$ versus the inverse of temperature T by using:

$$I_{d,min} \propto \exp(-E_g / 2kT) \quad (4)$$

where E_g is the transport bandgap and k is the Boltzmann constant (Figure 3(e)). We extract a bandgap of 0.57 eV for a 6.8-nm-thick PdSe₂ flake. Due to the contribution from trap states, this method is expected to underestimate the bandgap.²³ Finally, to further confirm the optical measurements shown in Figure 2(f), we performed temperature-dependent measurements on a PdSe₂ transistor fabricated using a bulk (116-nm-thick) crystal, as shown in Figure 3(f). Since the thickness of this device is significantly greater than the Debye screening length, the device cannot be fully turned off. Nevertheless, we still observe moderate gate-dependent transport due to the low background doping in PdSe₂. This indicates that bulk PdSe₂ is semiconducting and is consistent with the absorption measurements shown above.

We next investigate the performance of PdSe₂ as a photoconductor utilizing the devices discussed in Figure 3. Figure 4(a) shows the room-temperature spectral responsivities of the 6.8-nm- and 116-nm-thick PdSe₂ devices shown in Figure 3(a). We measured the spectral responsivity of these devices using an FTIR by replacing the internal detector with the PdSe₂ devices and focusing its light source on the devices with a CaF₂ lens. The internal deuterated triglycine sulfate (DTGS) in the FTIR was used to find the relative intensity of the light source and a NIST-traceable Ge photodiode to calibrate the illumination intensity of the light source.²⁴ The responsivity is calculated as $R(\lambda) = I_{ph}(\lambda)/P_{in}(\lambda)$, where I_{ph} is the photocurrent, and P_{in} is the incident power on the device. The specific detectivity (D^*) is then calculated using:

$$D^* = \frac{\sqrt{A\Delta f}}{NEP} = \frac{R\sqrt{A\Delta f}}{i_n} \quad (5)$$

where A is the device area, Δf is the integration time, NEP is the noise equivalent power, and $i_n^2 = 2qI_d\Delta f$ is the squared noise current, where I_d is the dark current. Figure 4(b) shows the room-temperature spectral D^* of the 6.8-nm- and 116-nm-thick PdSe₂ devices. We find a room-temperature D^* of 1.8×10^{10} cm Hz^{1/2} W⁻¹ at a wavelength of 1 μ m for the 6.8-nm-thick PdSe₂ device. From a plot of $(EQE/h\nu)^{1/2}$ versus $h\nu$, we extracted 0.59 eV and 0.49 eV as the optical bandgaps for a 6.8-nm- and 116-nm-thick PdSe₂ flake, respectively (Figures 4(c)–4(d)). It is important to note that the photoresponse measurements shown were taken at $V_g = 0$. This was found to give the highest photoresponse and is consistent with the I_d - V_g characteristics which show the lowest dark current for V_g near zero.

III. CONCLUSIONS

In conclusion, we have performed a systematic study on the optical and electrical properties of PdSe₂, a layered two-dimensional pentagonal semiconductor with an indirect bandgap ranging from ~0.5–1.37 eV. We found that a 6.8-nm-thick PdSe₂ flake-based transistor has effective electron mobilities of 130 and 520 cm² V⁻¹ s⁻¹ at 300K and 77K, respectively. The same transistor can serve as a SWIR photoconductive detector with a D^* of 1.88×10^{10} cm Hz^{1/2} W⁻¹ at 1 μ m with a cutoff wavelength of 1.94 μ m. At 1 μ m, PdSe₂ has a D^* on the same order of magnitude compared to

those of 2D tellurium (Te)²³ and black phosphorous (bP).²⁴ Te and bP, however, have optimized cutoff wavelengths of 3.4 μm and 4.6 μm , respectively, allowing more coverage of the SWIR band compared to PdSe₂. Nonetheless, given the promising preliminary values of D^* for our simple PdSe₂ device structure, further optimization, e.g. optical cavity substrate engineering to shift the peak D^* and cutoff wavelength, may allow the use of PdSe₂ for high-performance SWIR photodetectors.²³ Most importantly, through different measurements, we have determined that PdSe₂ does not undergo a semiconductor to metal transition with increasing thickness. This finding changes the outlook for the potential application space in which PdSe₂ may be used without further manipulating the material's band structure, e.g. by strain^{25,26} or defect²⁷ engineering.

SUPPLEMENTARY MATERIAL

See supplementary material for additional material characterization.

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“Word-count-equivalents”:

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- Main text (excluding title, author list, abstract, acknowledgments, and references): 2030
- Figures: $3 \times 400 + 1 \times 200 = 1400$
- Equations: $4 \times 7 + 1 \times 13 = 41$

FIGURES

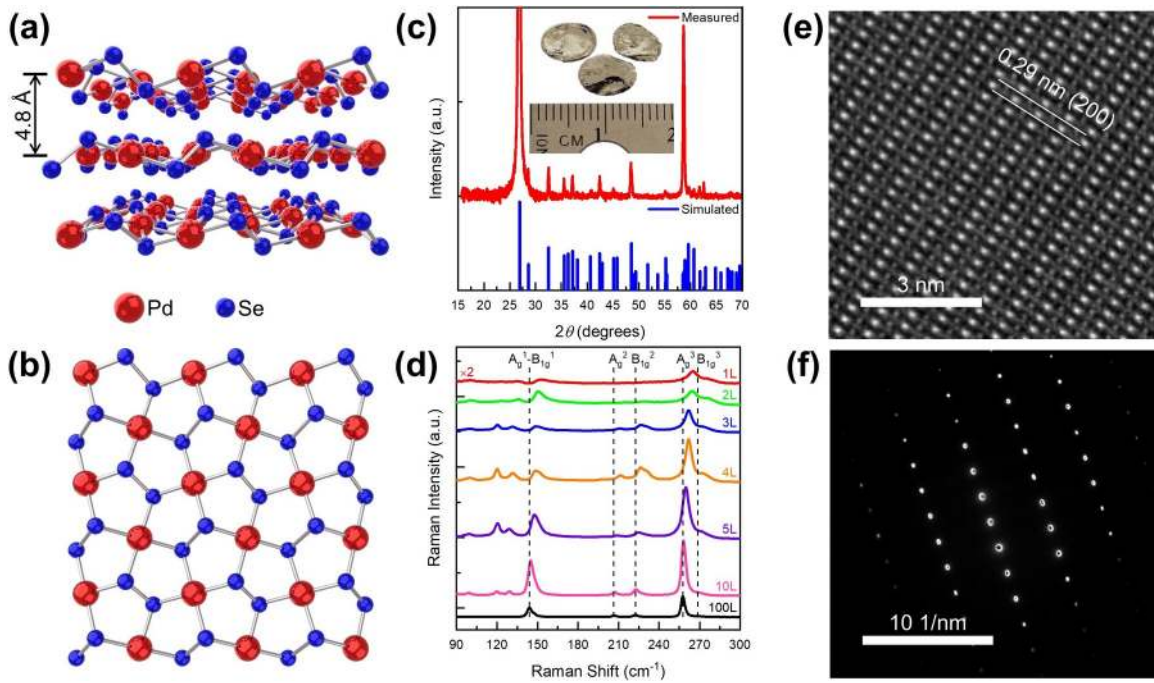


FIG. 1. Crystal structure of PdSe₂: (a) lateral and (b) top-down view. (c) Measured and simulated XRD spectra of a bulk PdSe₂ crystal. (d) Raman spectra of PdSe₂ for various thicknesses. (e) HR-TEM image and (f) SAED pattern of a 6.8-nm-thick PdSe₂ flake.

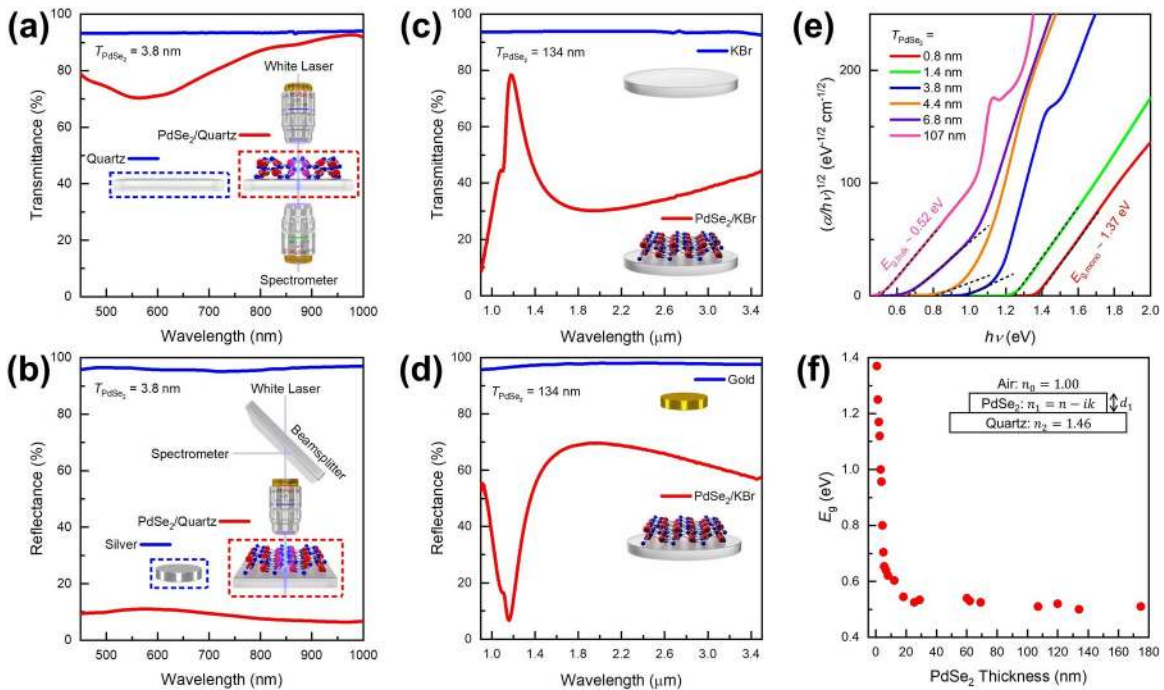


FIG. 2. (a) Transmission and (b) reflection measurements of a 3.8-nm-thick PdSe₂ flake on a quartz substrate in the visible-wavelength range. (c) Transmission and (d) reflection measurements of a 134-nm-thick PdSe₂ flake on a KBr substrate in the infrared-wavelength range. (e) Optical indirect bandgap extraction of PdSe₂ for various thicknesses. (f) Optical indirect bandgap of PdSe₂ as a function of its thickness.

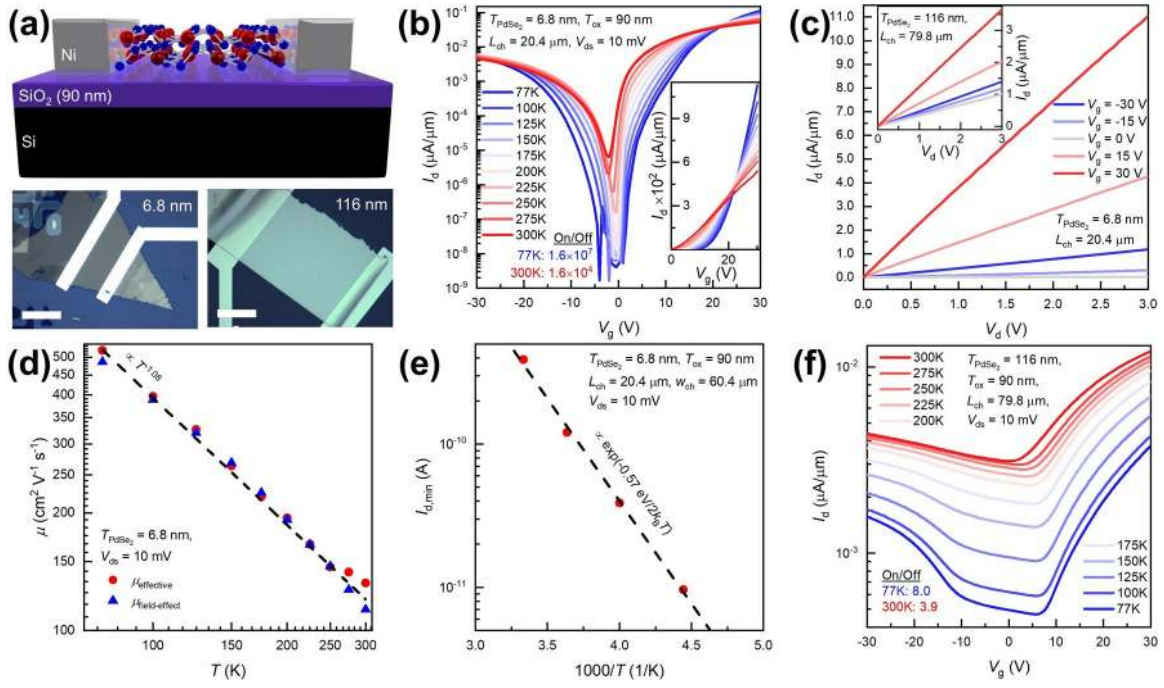


FIG. 3. (a) Back-gated PdSe₂ field-effect transistor (FET) structure and optical microscope images of devices with 6.8-nm- and 116-nm-thick PdSe₂ flakes. Scale bars are 30 μm. (b) Temperature-dependent I_d - V_g characteristics of the 6.8-nm-thick PdSe₂ device measured at a low V_d of 10 mV. The inset shows a plot of the same data in linear- I_d scale. (c) I_d - V_d characteristics of the 6.8-nm-thick PdSe₂ device inset with those of the 116-nm-thick PdSe₂ device. (d) Effective and field-effect mobility of the 6.8-nm-thick PdSe₂ device. The dashed line shows a power law fit $\mu_{\text{effective}} \propto T^{-\square}$ where $\square = 1.08$. (e) Arrhenius plot showing the minimum drain-source current ($I_{d,\text{min}}$) of the 6.8-nm-thick PdSe₂ device as a function of temperature to extract the transport bandgap $E_g = 0.57$ eV. (f) Temperature-dependent I_d - V_g of the 116-nm-thick PdSe₂ device measured at $V_d = 10$ mV, showing bulk semiconducting behavior.

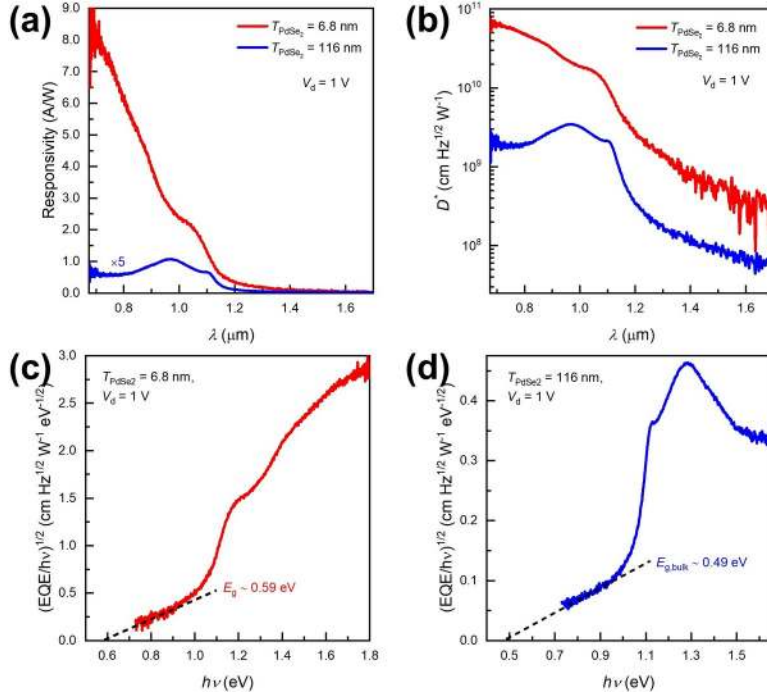


FIG. 4. (a) Room-temperature spectral responsivities and (b) specific detectivities D^* of the 6.8-nm- and 116-nm-thick PdSe₂ devices shown in Figure 3(a) measured at a drain voltage V_d of 1 V with zero gate voltage V_g applied. (c), (d) Indirect bandgap extraction of a 6.8-nm-thick and a 116-nm-thick PdSe₂ flake.

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