

1 Supporting Information

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4 **Plasma-enhanced Microwave Solid-state Synthesis of Cadmium** 5 **Sulfide: Reaction Mechanism and Optical Properties**

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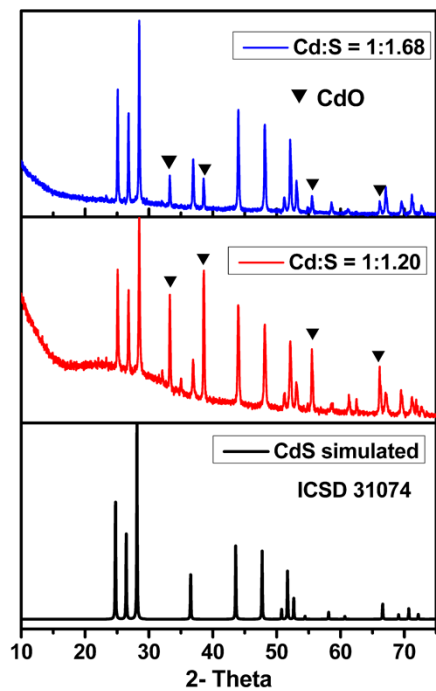
14 **Chemical:** The Cadmium powder (-200 mesh, 99.99%), sulfur pieces (Puratronic, 99.9995%),
15 selenium powder (200 -mesh, 99.999%), tellurium powder (200 -mesh, 99.5%), zinc flake (-325
16 mesh, 99.9%) and iodine spheres (ultra dry, 10 -mesh, 99.998%) were all purchased from the
17 Alfa Aesar company. All elements were purchased in form of powders delivered and stored in
18 glass bottles. Therefore, powders could be slightly surface oxidized. The content of oxygen in
19 the powders, used for reactions, is not defined by vendors and was not analyzed by us. However,
20 we detected some traces of CdO and Cd(OH)₂ in powder diffraction spectra.

21 **PMPVT experiments:** All of the reagents were used without further purification. Fine powders
22 of respective metals and chalcogens were mixed and sealed in a quartz ampoule (approximately
23 100~200 mg of mixture). The length of the quartz ampoule is about 8~9 cm with the 13 mm
24 external diameter. The ampoule was filled with Ar gas and the pressure inside could be
25 controlled from 1×10^{-4} Torr to 750 Torr. The optimized pressure is 10 Torr. Then, the ampoules
26 were placed in a quartz tube that was located in the center of a modified domestic microwave
27 oven (2450 MHz, 1 kW) without any additional conventional heating. The argon gas with about
28 5 ~ 10 Torr pressure flowed through the evacuated quartz tube. When the microwave oven was

29 running, the argon plasma in the quartz tube was ignited and could be seen. After 2~4 hours,
30 most of the reactions were complete.

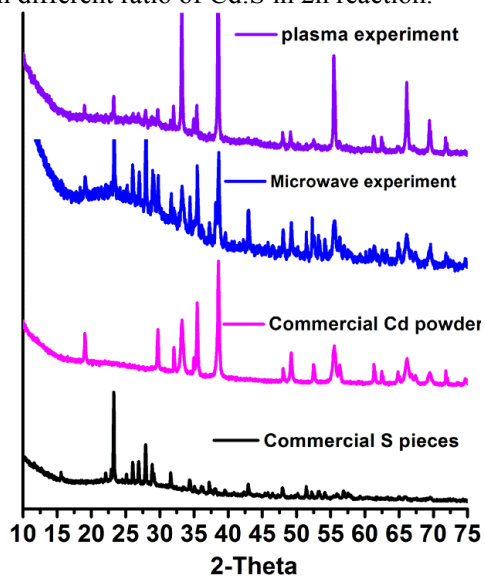
31 **PMCVT experiment:** The PMCVT experiment was very similar with PMPVT. The only
32 different was that the iodine (about 8 mg) was added as transport chemical in the starting mixture.
33 The Cd:S:I ratio is about 16: 1. We kept the PMCVT for 4 hours to get the fully reacted product.

34 **Physical measurement:** PXRD patterns were collected by a Bruker D8 Advance diffractometer
35 using $\text{CuK}\alpha$ (1.54178 Å) radiation in the angular range of $2\theta = 5-75^\circ$ at room temperature. SEM
36 images and EDX were obtained from a JEOL JSM 7600F scanning electron microscope. Both
37 the laser beams (solid-state laser, 473 nm and Nd:YAG solid-state laser, 532 nm) were
38 collimated and focused thorough a 100× objective onto the sample surface. All of the spectra
39 were collected using a confocal triple-grating spectrometer (Horiba-JY T64000). The reflectance
40 was collected by CRAIC 20 Microspectrophotometer and converted to absorbance using the
41 Kubelka - Munk equation.¹ The PL spectrum was fitted by a Gaussian function in Origin
42 software, whereas the baseline-subtracted Raman peaks were fitted by a Lorentzian function. The
43 Raman baseline was fitted by a line function. The simulated PXRD pattern through single crystal
44 data was generated by Mercury 2.3 software.



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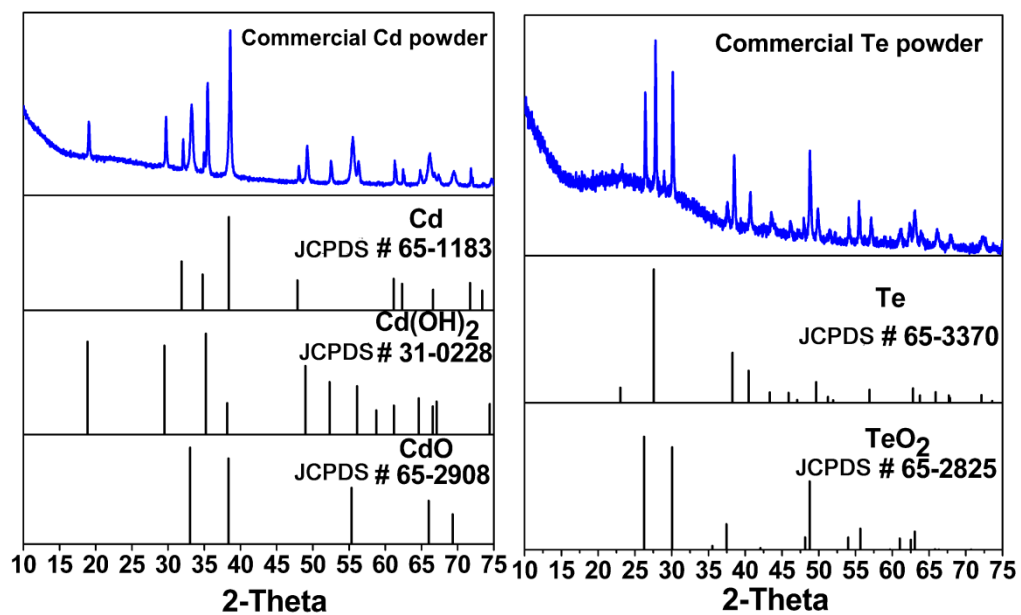
46 **Fig. S1.** XRD of the products with different ratio of Cd:S in 2h reaction.



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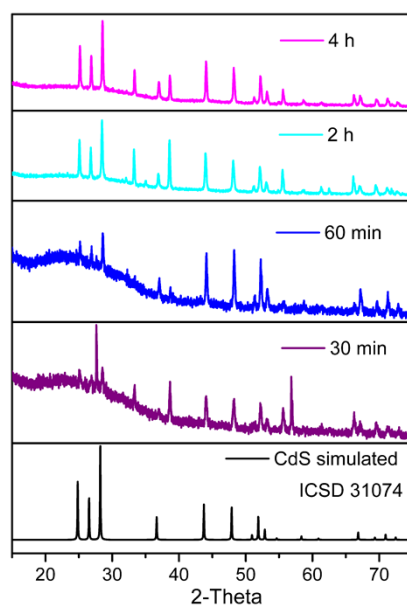
48 **Fig. S2.** The XRD of the products under different conditions.

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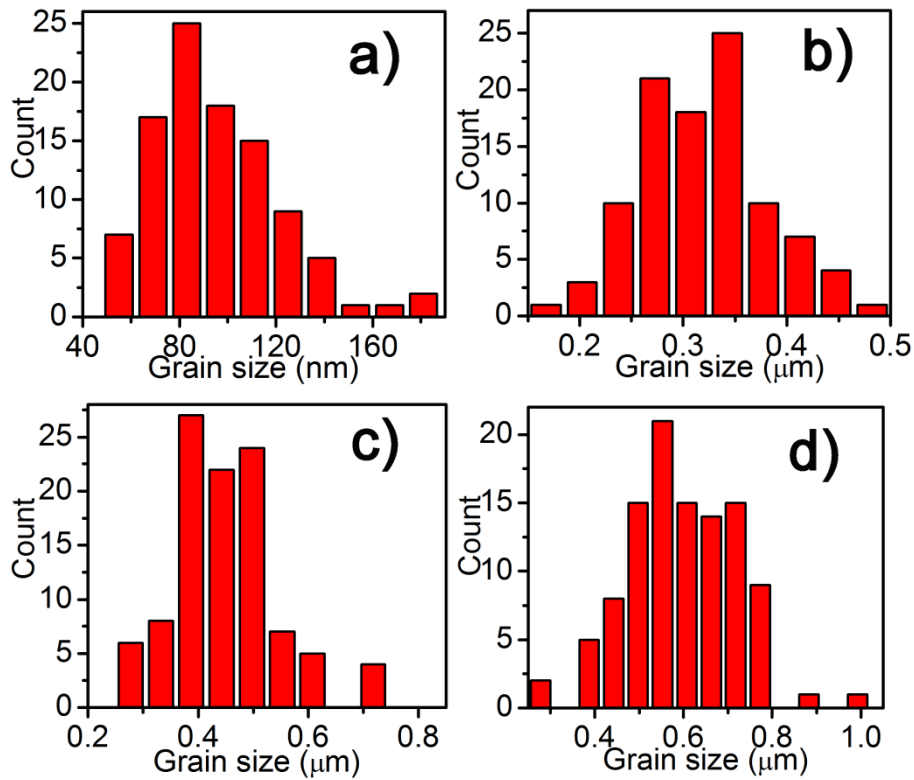
51 **Fig. S3.** The PXRD of commercial Cd and Te powder.



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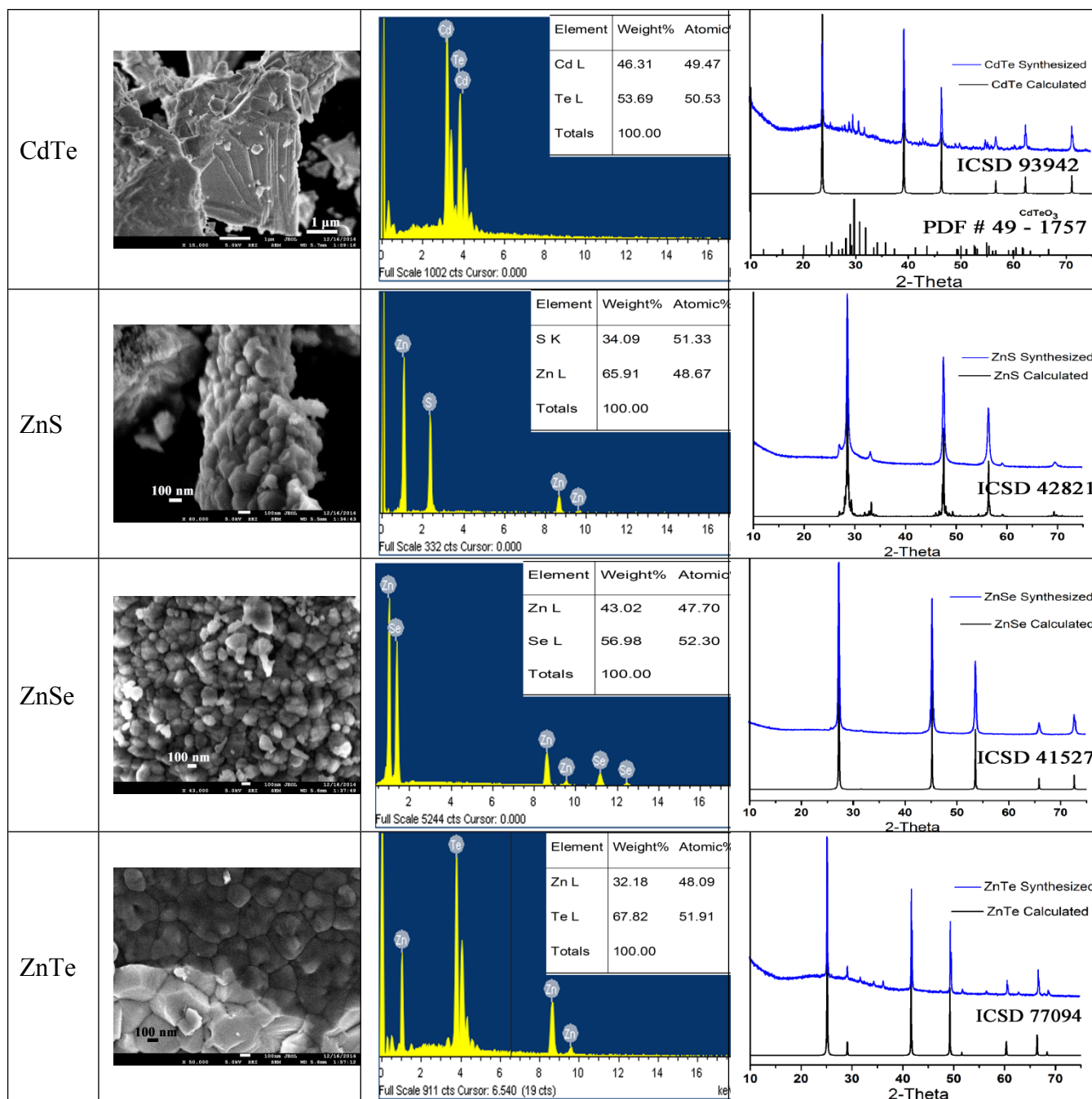
54 **Fig. S4.** The effect of reaction time.



58 **Fig. S5.** Time dependent grain size distribution of CdS synthesized by PMPVT, a) 30 min; b) 60 min; c) 120
 59 min and d) 240 min.

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 61 **Table S1** SEM images, EDX and PXRD patterns of other II-VI chalcogenides synthesized by PMPVT. The
 62 blue curve in the XRD results is the experimental pattern, whereas the black one is the related theoretical one.

	SEM	EDX	PXRD												
CdSe		<table border="1"> <thead> <tr> <th>Element</th> <th>Weight%</th> <th>Atomic%</th> </tr> </thead> <tbody> <tr> <td>Se L</td> <td>38.54</td> <td>47.16</td> </tr> <tr> <td>Cd L</td> <td>61.46</td> <td>52.84</td> </tr> <tr> <td>Totals</td> <td>100.00</td> <td></td> </tr> </tbody> </table> <p>Full Scale 2111 cts Cursor: 6.444 (38 cts)</p>	Element	Weight%	Atomic%	Se L	38.54	47.16	Cd L	61.46	52.84	Totals	100.00		<p>— CdSe Synthesized — CdSe Calculated ICSD 60630</p> <p>2-Theta</p>
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Totals	100.00														



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64 1. Nobbs, J. H., Kubelka—Munk Theory and the Prediction of Reflectance. *Color. Technol.*

65 **1985**, 15 (1), 66-75.

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