

## Electronic Supplementary Information

### Photoluminescence Mechanism of CsPb<sub>2</sub>Br<sub>5</sub> Microplates

### Revealed by Spatially Resolved Single Particle Spectroscopy

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#### Part I. Chemicals and synthesis

All chemicals are purchased from Sigma-Aldrich and directly used without further purifications, including Cs<sub>2</sub>CO<sub>3</sub> (99.95%, trace metal basis), PbBr<sub>2</sub>(≥98%, Aldrich), oleylamine (OAm, technical grade, 70%), oleic acid (OA, technical grade, 90%), 1-octadecene (ODE, technical grade, 90%), and hexane (HPLC, 99.9%).

The CsPb<sub>2</sub>Br<sub>5</sub> microplates were prepared according to the following procedure. The Cs-oleate precursor was prepared first. Cs<sub>2</sub>CO<sub>3</sub> (202 mg) was mixed with OA (10 mL) in a 25 mL three neck flask, degassed, and then dried for 1 hour under vacuum at 120 °C before it was heated to 150 °C under N<sub>2</sub> protection and then kept for 1 hour to ensure complete reaction. The obtained product was stored in N<sub>2</sub> atmosphere after cooling to room temperature. Secondly, PbBr<sub>2</sub> (0.188 mmol, 69 mg), 5 mL of ODE, 0.5 mL of OA, and 1 mL of OAm were loaded into a 25 mL three neck flask, degassed 1 hour under vacuum at 120 °C, and then heated to 140 °C under N<sub>2</sub> protection. After keeping for another 1 hour, it was heated up to 190 °C until the temperature became stable. 0.5 mL of Cs-oleate precursor was extracted and injected into the above reaction solution quickly under stirring (> 650 rpm). The solution became yolk yellow immediately. The solution was subsequently kept for another 30 min for the growth of the microplates. The reaction solution was cooled using ice-cold water bath then transferred to centrifuge directly at 1000 rpm for 3 min. The sediments were collected, washed with hexane, centrifuged again at 1500 rpm for 3 min, and repeated 2~3 times. The sample can stay stable at room temperature for over one month.

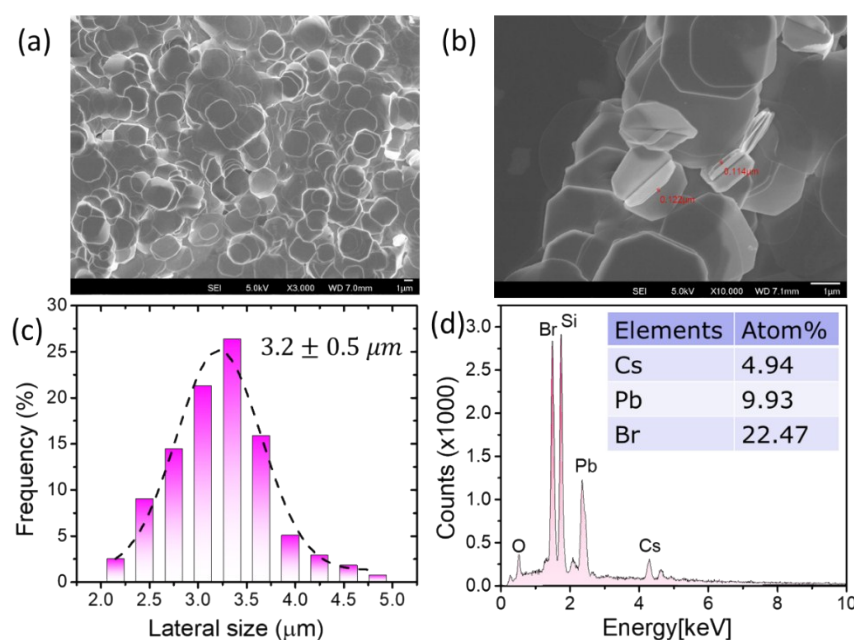
#### Part II. Instruments

FE-SEM images were taken by using JEOL JSM-6701F SEM, with accelerating voltage variable from 0.5 kV to 30 kV. EDX measurements were conducted by JEOL

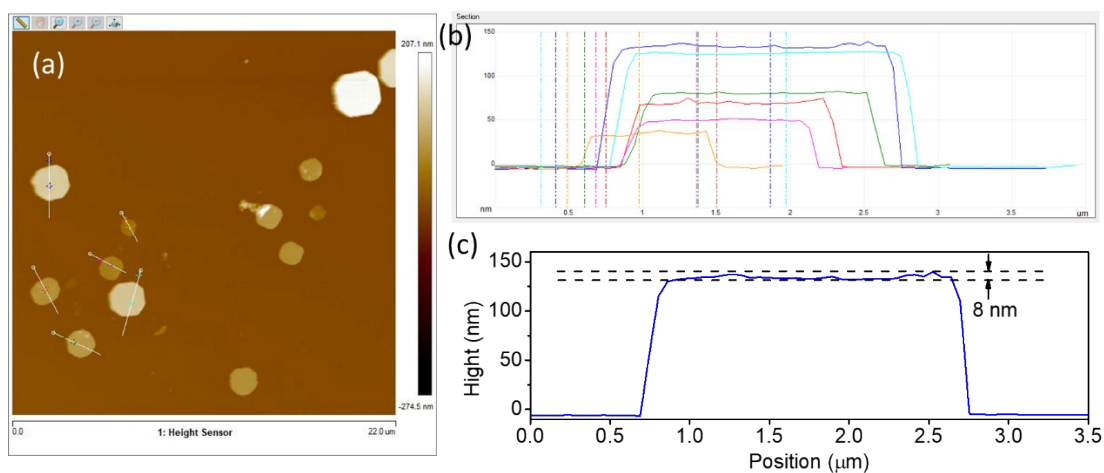
JED-2300 EDS system with resolution around 136 eV. TEM measurements were conducted using JEOL JEM-3010 with acceleration voltage of 300 kV; AFM measurements were performed using Bruker Dimension FastScan (Tapping mode). Powder XRD was conducted on a Bruker D8 Advance system, with Cu K $\alpha$  x-ray source at 1.547 Å; Absorption spectra were measured by using a Shimadzu UV-3600 spectrometer, the fluorescence and excitation spectra were measured using a commercial Horiba 320A Xeon lamp spectrometers.

The single particle experiments was conducted on a Nikon inverted microscope with x100 oil immersion objectives. A picosecond pulse diode laser (438 nm, <200ps, repetition rate of 10 kHz, average power of 6 $\mu$ W) was used as light source with a 440/10 nm band-pass used to narrow the spectral line width. PI XY Piezo translation stage was used for the PL and FLIM mapping, controlled by commercial software SymPho Time. The emission signals were collected by using Princeton Instruments, Acton Spectra Pro 2300i spectrometer (grating 150 g/mm), in combination with a PicoQuant PMA series PMT, and Acton Pixis 100 CCD and a 473 nm long pass filter.

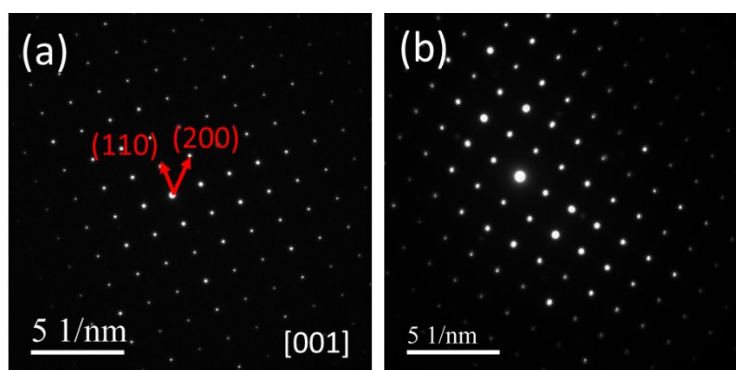
### Part III. Supporting data



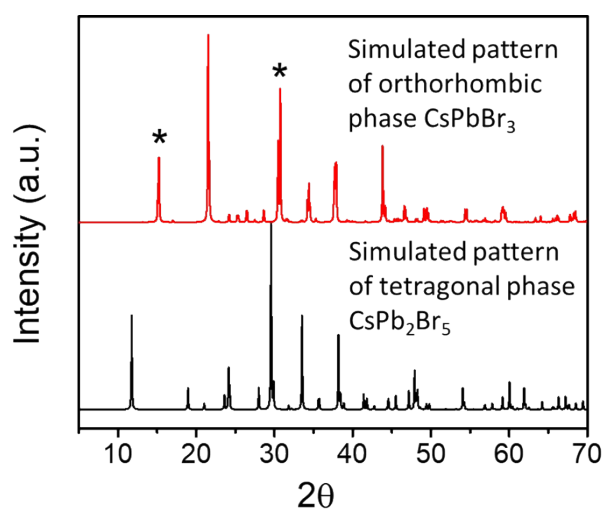
**Figure S1.** (a,b) SEM images of the obtained CsPb<sub>2</sub>Br<sub>5</sub> Microplates; (c) Size distribution obtained from the SEM images; (d) EDX elements analysis. The atomic ratio of Cs, Pb and Br is matched with standard stoichiometric composition.



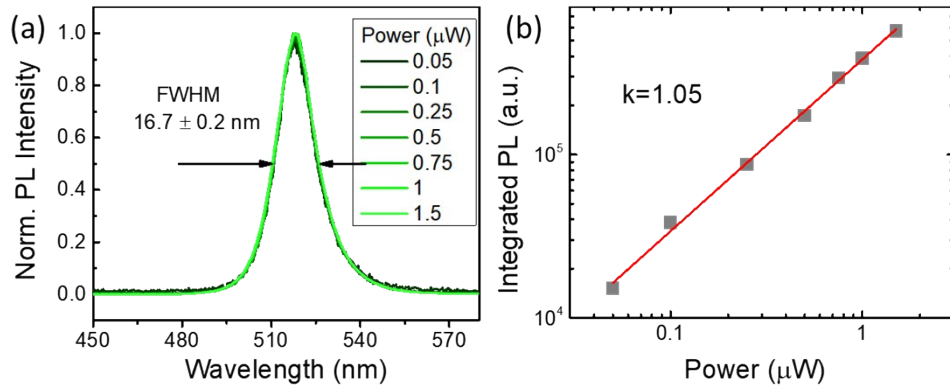
**Figure S2.** (a) AFM images of the prepared microplates; (b, c) Surface roughness of different microplates.



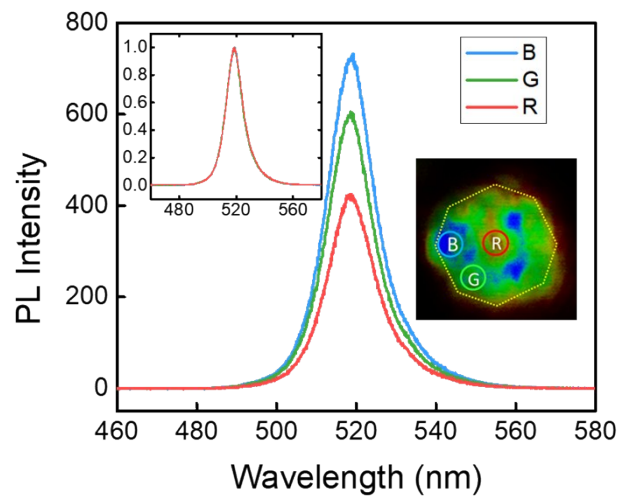
**Figure S3.** SEAD pattern of the original (a) and solvent treated (b) sample.



**Figure S4.** Simulated XRD pattern of orthorhombic phase CsPbBr<sub>3</sub> (red line, extracted from *Cryst. Growth Des.* 2013, 13, 2722–2727) and tetragonal phase CsPb<sub>2</sub>Br<sub>5</sub> (black line, simulated from PDF#25-0211).



**Figure S5.** (a) Steady state PL spectra of single microplates under different excitation powers. (b) The log-log plot of integrated PL intensity versus the excitation power.



**Figure S6.** Steady PL spectra of different regions under excitation power of 1  $\mu\text{W}$ , where B (blue), G (green) and R (red) refer to the different regions as illustrated in the inset image. The top left inset is the normalized PL spectra.