Supporting information

sp-sp^2 Hybrid Conjugated Microporous Polymers Derived Pd Encapsulated Porous Carbon Materials for Lithium-sulfur Batteries

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2. Experimental Section

2.1 Materials preparation

1,4-diiodobenzene (DIB, 0.33g, 1 mmol), 1,3,5-triehynylbenzene (TEB, 0.15g, 1 mmol), $Pd(PPh_3)_4$ (0.1g, Pd 4 mol%), and a magnetic stirring bar was loaded into a 200mL Schlenk bottle in Ar filled glove box. Then toluene (20 mL) and NEt_3 (20 mL) were injected into the bottle in the Schlenk line. The reaction was stirred at 100 °C for 72h with the protection of Ar. The precipitate was filtered and washed by Soxhlet extraction with a mixture of $CHCl_3$/MeOH (1:1 v/v) for 24h, and then dried under vacuum to obtain Pd-CMP.

Pd-PCMs were prepared by pyrolysis of the obtained Pd-CMP under Ar + H_2 atmosphere. Different sizes of Pd nanoparticles were obtained in Pd-PCMs with pyrolysis temperature and time varies (Fig. S6). Considering the conductivity of carbon matrix, Pd-PCMs obtained at 800 °C were selected as the electrode for infiltrating sulfur.

In order to illustration the enhancement effect of Pd nanoparticles, PCMs were prepared by acidic treatment of Pd-PCMs. Pd-PCMs were dispersed in conc. HNO_3 with magnetic stirring for 24h, the precipitate was filtered and washed with amounts of water till the pH = 7, and then frozen dry to obtain PCMs.

2.2 Materials characterization

Phase analysis was characterized by wide angle X-ray diffraction (XRD). Images of morphology were obtained from scanning electron microscopy (SEM, HITACHI-4800). Images, HRTEM, and STEM mapping were carried out on transmission electron microscopy (TEM, FEI TECNAIG2 F30). Binding of energy was launched on X-ray photoelectron spectroscopy (XPS, PHI5802). The loading of sulfur in carbon matrix was tested by thermogravimetric analysis (TG-DSC, Netzsch, STA449). Raman spectrometer (HORIBA LabRAM HR800) was used to characterize samples' Raman peaks using 532 nm incident radiation. The surface area and pore volume of the samples were determined by nitrogen gas adsorption at 77 K with an automated adsorption apparatus (Micrometrics, ASAP 2020).

2.3 Electrochemical measurements

The carbon/sulfur electrode was prepared by mixing the carbon/sulfur hybrids, carbon black, and polyvinylidene difluoride (PVDF) with a weight ratio of 80:10:10 in NMP solvent to form slurry. The slurry was coated onto a carbon-coated aluminum foil using a doctor blade and dried at 60 °C under vacuum for 24h. The coin cell with a metallic lithium anode was assembled in glove-box (Mbraun) to evaluate the electrochemical performance of the as-obtained samples. The mass loading of sulfur in the obtained cathode is ~1.2 mg/cm^2 and microporous polypropylene sheet (Celgard 2500) was used as the separator. The electrolyte was 1 M bis(trifluoromethane) sulfonamide lithium salt (LiTFSI) dissolved in a mixture of 1,2-dioxolane (DOL) and dimethoxymethane (DME) (1:1 by volume) with adding 1 wt% $LiNO_3$. Cyclic voltammetry (CV) experiment was carried out over the potential range of 1.5-3 V (versus Li metal) at a scanning rate of 0.1 mV/s using VMP3 electrochemical workstation (Bio Logic Science Instruments). The galvanostatic charge/discharge performance test and the rate capability at different rates were performed using a Land 2001A cell test system (Wuhan, China) at 298K.

II. Supplementary Figures:

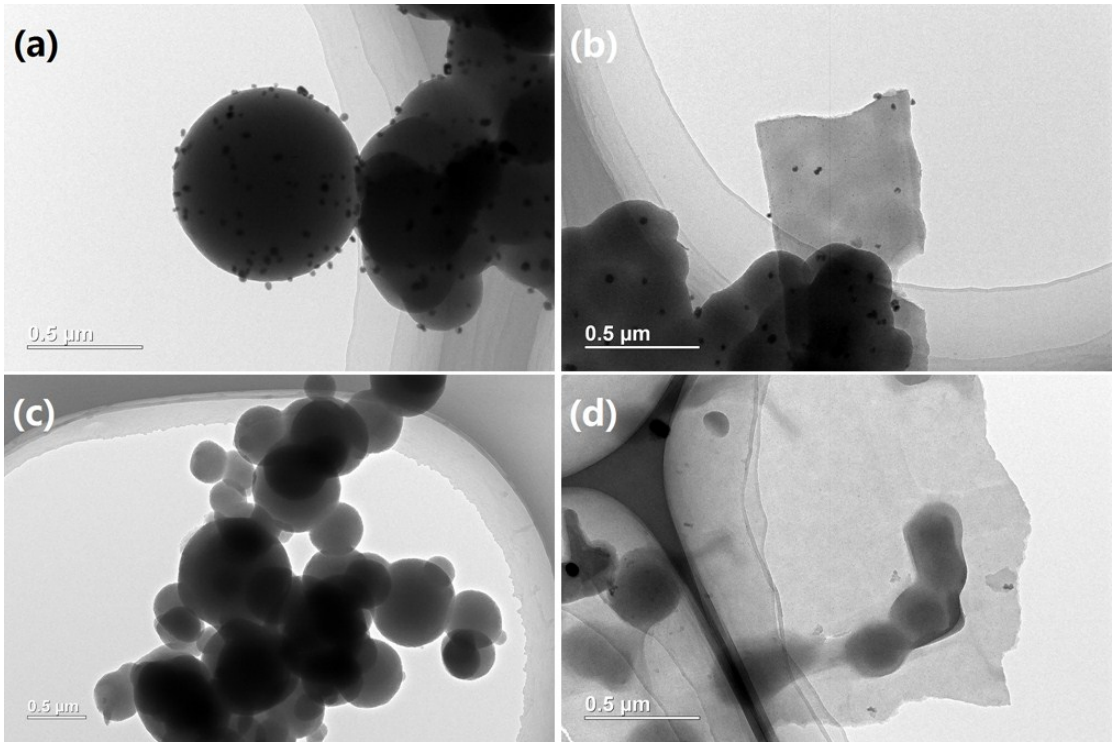

Fig. S1 TEM of (a) , (b) Pd-PCMs and (c), (d) PCMs.

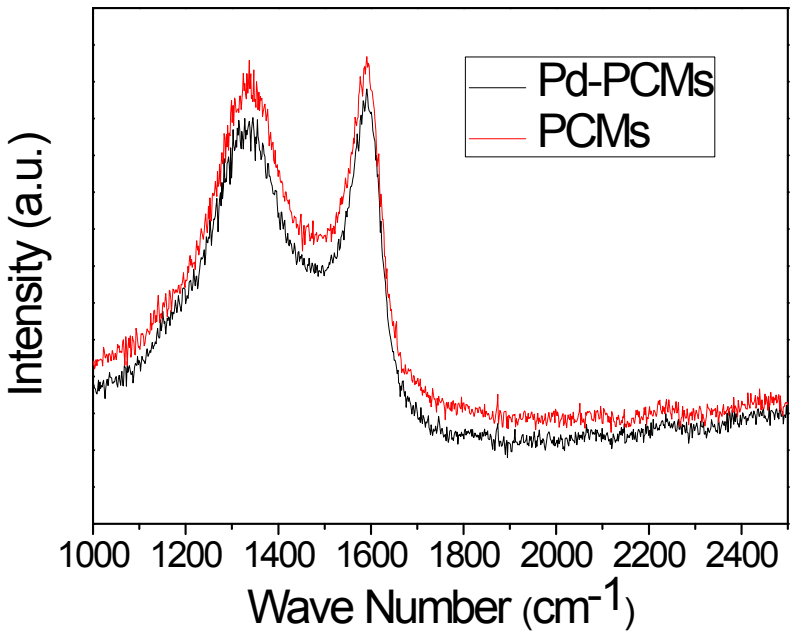

Fig. S2 Raman spectrum of Pd-PCMs and PCMs.

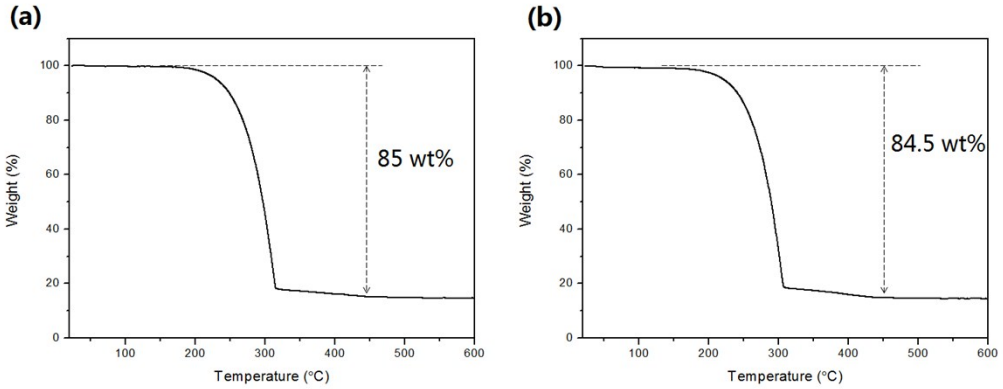

Fig. S3 TG curves of the (a) Pd-PCMs/S and (b) PCMs/S

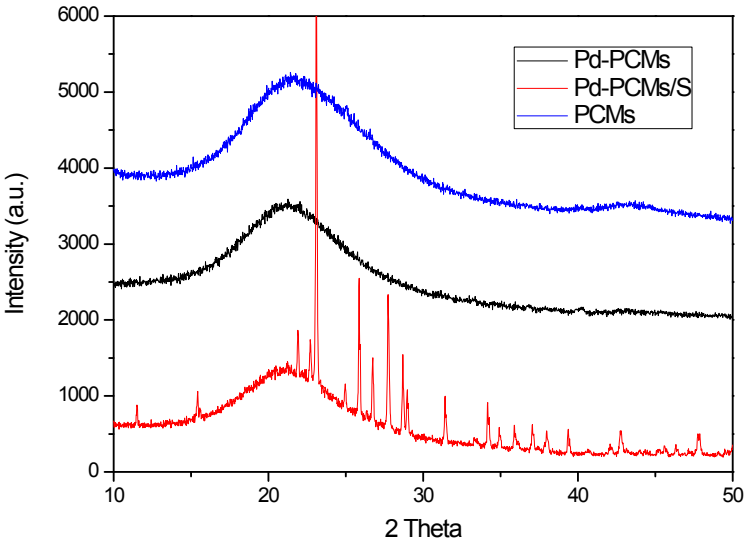

Fig. S4 XRD patterns of Pd-PCMs, PCMs and Pd-PCMs/S.

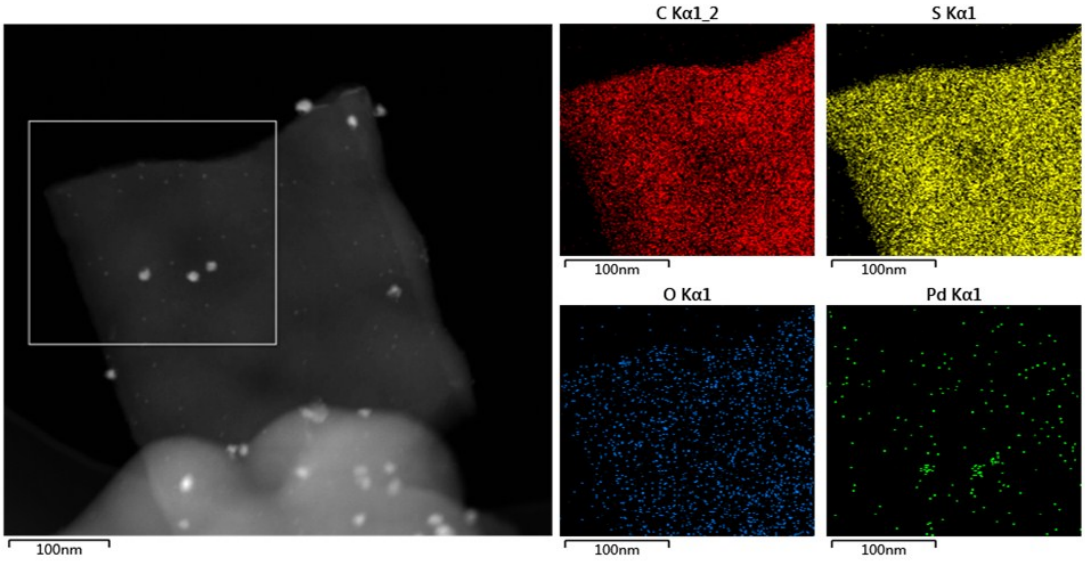

Fig. S5 STEM image of Pd-PCMs/S and the corresponding elemental mapping.

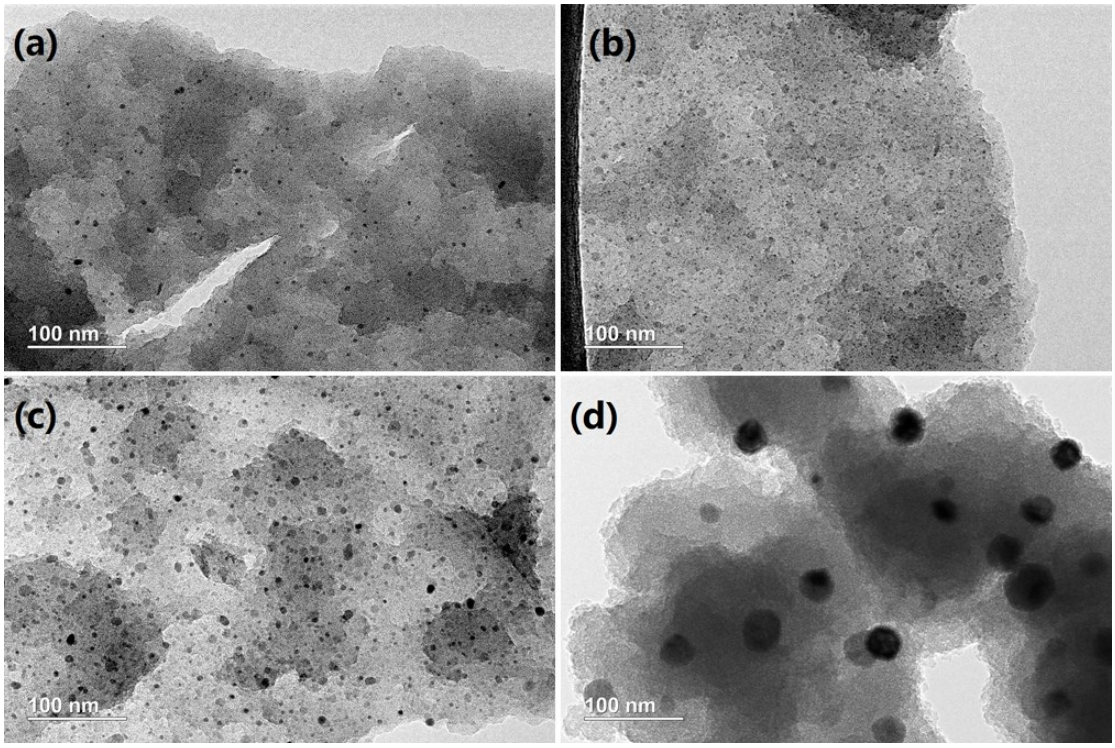

Fig. S6 TEM images of Pd-PCMs pyrolysis at different temperature and time. (a) 250°C, 4h; (b) 400°C, 4h; (c) 600°C, 3h, (d) 800°C, 2h.