Supplementary Information

Research on the mechanism and reaction conditions of electrochemical preparation of persulfate in a split-cell reactor using BDD anode

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Catalogues

1. Morphology and electrochemical performance of BDD

1.1 XRD pattern of BDD/Ta electrode

Fig. S1 shows the XRD pattern of BDD/Ta thin film electrodes. As can be seen in the figure, the diffraction peaks of Ta appear at 2θ = 38.6°, 55.5°, 69.6°, 82.2°, 94.9° and 107.5°. The diffraction peak at 2θ = 43.9° corresponds to the diamond phase. Besides, the diffraction peaks of Ta_2C and TaC appear in the XRD spectrum of the BDD/Ta electrode film [1]. But, the diffraction peaks of Ta are significantly stronger, and the main phase of the BDD/Ta electrode film is still Ta. This not only gives the electrode excellent conductivity of metal electrodes, but also electrochemical active adsorption sites during the electrolysis, and the electrode is not easy to corrode.

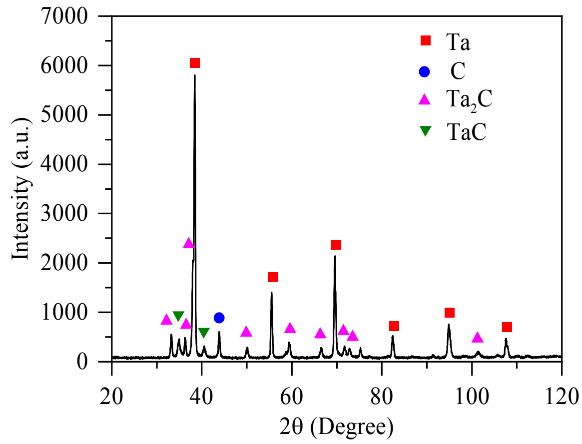


Fig. S1 XRD pattern of BDD thin film electrodes deposited on Ta substrate[2]

1.2 SEM image of the BDD/Ta electrode

The SEM image of Fig. S2 shows the surface morphology of the BDD/Ta electrode. The electrochemical performance of the BDD electrode surface depends on the control the diamond polycrystals growth, the level of boron doping and the electrode pre-treatments [3]。 In this figure, the BDD thin film is about 5 μm thick and is composed of randomly oriented microcrystals. The crystals are clear in shape and are complete. The crystal structure on the surface of the BDD electrode dramatically increases the contact area between the electrolyte and the electrode surface, which facilitates the adsorption of electrolyte at the active site [4]. As is known, the boron-doping concentration of the BDD/Ta electrode is 2500 ppm; and boron is doped in the interstitial state in substrate materials, so that the conductivity of transition metal Ta is slightly reduced.

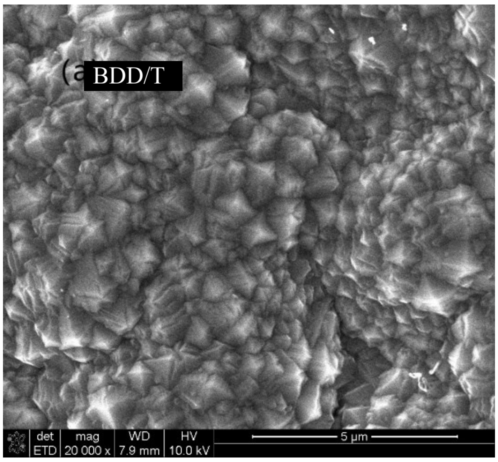


Fig. S2 SEM image of the BDD film on a Ta substrate[2].

1.3 Raman spectrum of the BDD/Ta electrode

Fig. S3 shows the Raman spectrum of the BDD/Ta electrode. A peak with obvious diamond characteristics (sp^3) appears at 1327 cm^{-1}, and the appearance of the peak at 1483 cm^{-1} can be attributed to the sp^2 hybrid structure of graphite-like non-diamond phases. This indicates that the graphite structure formed on the substrate of Ta is structurally complete and has the remarkable features of a high quality polycrystalline diamond thin film [5]. In the process of preparing the PDS by electrolyzing sulphate solution, the weakly adsorbed diamond thin film on the electrode surface can effectively generate •OH instead of directly oxidizing it to O_2.

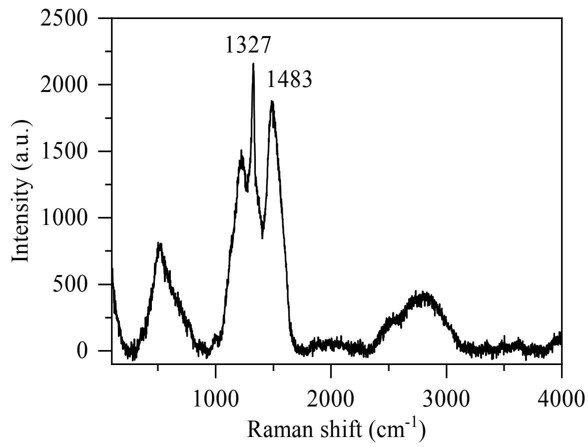
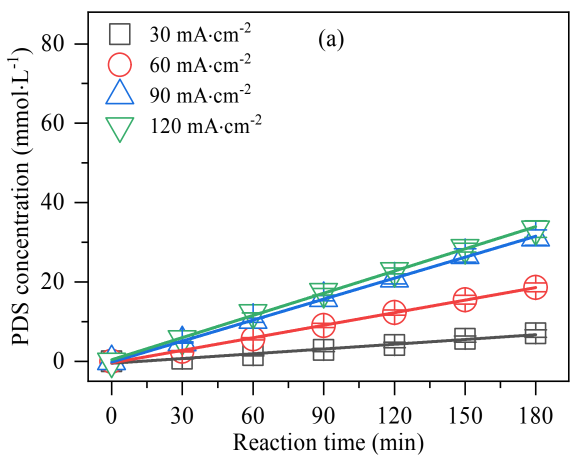


Fig. S3 Raman spectrum of the BDD/Ta electrode

2. Effect of current density on the synthesis of PDS under different concentration conditions



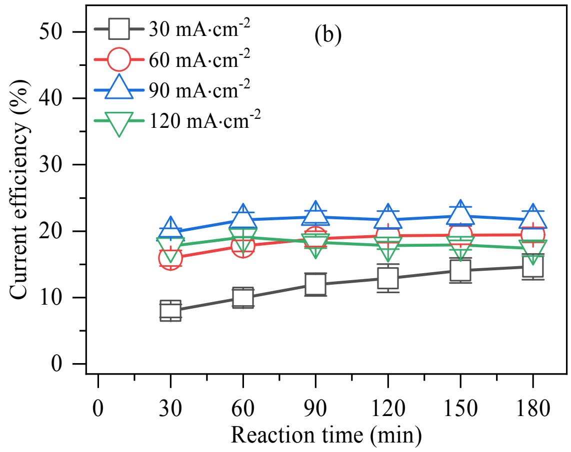


Fig. S4 Effect of current density on the synthesis of PDS by 0.2 mol L^{-1} Na_2SO_4

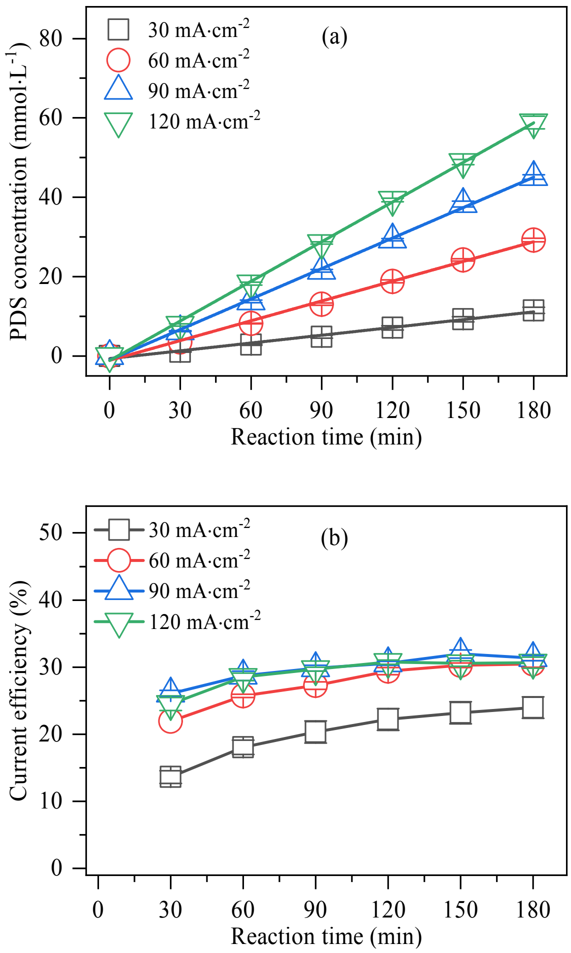


Fig. S5 Effect of current density on the synthesis of PDS by 0.4 mol L^{-1} Na_2SO_4

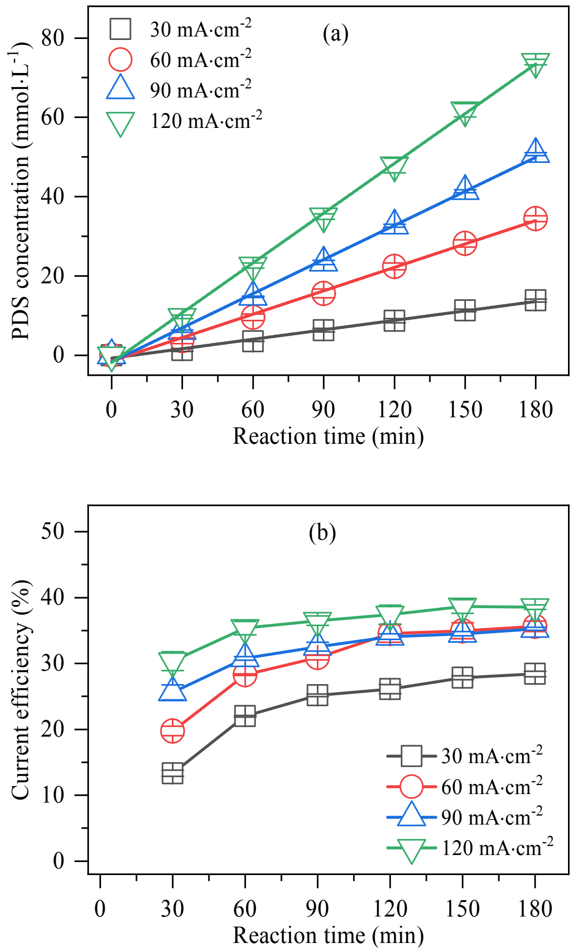
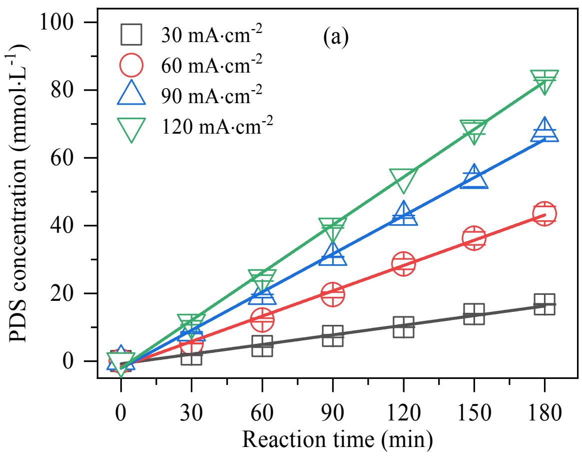


Fig. S6 Effect of current density on the synthesis of PDS by 0.6 mol L^{-1} Na_2SO_4



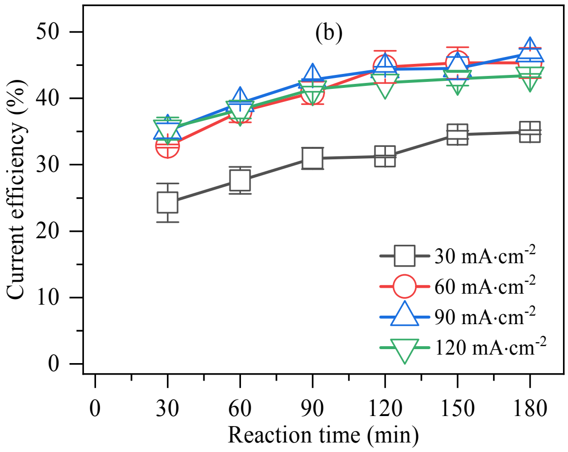


Fig. S7 Effect of current density on the synthesis of PDS by 0.8 mol L^{-1} Na_2SO_4

References

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