In-situ Synthesis of Olivine LiFePO₄/C Composite by Emulsion Drying Method and Its Electrochemical Properties as Lithium Intercalation Material

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The electroactive LiFePO₄/C nano-composite has been synthesized by an emulsion drying method. During burning out the oily emulsion precipitates in air-limited atmosphere at 300 °C, CO, CO₂, and amorphous or low crystalline carbon are generated, and trivalent iron as a cheap starting material is immediately reduced to divalent one at this stage, leading to a low crystalline LiFePO₄/C compound. Heat-treatment of the low crystalline LiFePO₄/C in an Ar atmosphere resulted in a well-ordered olivine structure, as refined by Rietveld refinement of X-ray diffraction pattern. From secondary electron microscopic and scanning transmission electron microscopic with corresponding elemental mapping images of iron and phosphor, it was found that the fine LiFePO4powders are modified by carbon. The in situ formation of nano-sized carbon during crystallization of LiFePO₄ brought about two advantages; prohibition of crystal growth of LiFePO4 particle and a great enhancement of electric conductivity as high as 10⁻⁴ S cm⁻¹ at room temperature. These effects of carbon on LiFePO4/C composite led to high capacity retention upon cycling at 25 °C and 50 °C and rate capability. That is, the obtained capacity was higher than 90 mAh (g-phosphate)⁰⁻¹ by applying a higher current density of about 1000 mA g⁻¹ (11C) at 50 °C.