

Research Article

Comparing the Electrochemical Performance of LiFePO₄/C Modified by Mg Doping and MgO Coating

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Supervalent cation doping and metal oxide coating are the most efficacious and popular methods to optimize the property of LiFePO₄ lithium battery material. Mg-doped and MgO-coated LiFePO₄/C were synthesized to analyze their individual influence on the electrochemical performance of active material. The specific capacity and rate capability of LiFePO₄/C are improved by both MgO coating and Mg doping, especially the Mg-doped sample—Li_{0.985}Mg_{0.015}FePO₄/C, whose discharge capacity is up to 163 mAh g⁻¹, 145.5 mAh g⁻¹, 128.3 mAh g⁻¹, and 103.7 mAh g⁻¹ at 1 C, 2 C, 5 C, and 10 C, respectively. The cyclic life of electrode is obviously increased by MgO surface modification, and the discharge capacity retention rate of sample LiFePO₄/C-MgO_{2.5} is up to 104.2% after 100 cycles. Comparing samples modified by these two methods, Mg doping is more prominent on prompting the capacity and rate capability of LiFePO₄, while MgO coating is superior in terms of improving cyclic performance.

1. Introduction

As a cathode material for lithium-ion batteries, olivine-type lithium iron phosphate (LiFePO₄) proposed by Padhi et al. [1] has attracted extensive attention. And its nontoxicity, flat voltage profile, high theory capacity, high safety, and abundance of raw materials have promoted LiFePO₄ to become one of the most outstanding lithium cathode materials. But the low electrical (electronic and ionic) conductivity limits its electrochemical properties according to the charge/discharge rate capability and operating temperature [2]. Decreasing particle size [3, 4], doping supervalence metal ion [5–7], and surface modification [8–17] are normal and efficient ways to overcome the weakness as well as enhance the electrochemical performance of LiFePO₄.

Lots of work were carried out on supervalent cation doping since Chung et al. [7] claimed that they raised the electronic conductivity of bulk LiFePO₄ by 8 orders of magnitude to 10⁻² S cm⁻¹ through low-level doping with some super-valent cations (Nb⁵⁺, Ti⁴⁺, and W⁶⁺). Although the origin of the increased conductivity by doping is still under

controversy, many groups have prepared LiFePO₄ positive material with excellent property through super-valence metal ion doping. Surface modification is also extensively applied to improve the electronic conductivity of LiFePO₄, especially carbon coating. But the tap density of active material dramatically decreases after only a little amount of carbon coating [10, 11]. Hence, researchers expect to find an appropriate coating to wholly or partly substitute for carbon, such as metal oxide. CeO₂ [7], V₂O₃ [12], MgO [13], SiO₂ [14], TiO₂ [15], CuO [16] and so forth have been employed to modify LiFePO₄, and predominant performance was gained.

In conclusion, super-valent cation doping and metal oxide surface modification are two efficient ways to improve the electrochemical property of lithium cathode electrode. The mechanism of enhancing electrode performance is dissimilar for doping and coating as well as their promoted aspects. Up to now, the different influence on LiFePO₄ material by these two methods using the same metal element was not reported.

Therefore, we take magnesium modification, for example, to discuss the different effects of metal ion doping and metal

oxide coating on electrode material. Mg doping or MgO coating has been carried out to ameliorate the electrochemical performance of LiCoO₂ [17], LiNiO₂ [18], LiNi_{0.8}Co_{0.2}O₂ [19], Li₃V₂(PO₄)₃ [20], and LiFePO₄ [13], and excellent rate capability or cyclic performance has been gained. It indicates that magnesium modification is an efficient and popular method to improve the property of lithium positive material. Mg-doped and MgO-coated LiFePO₄/C were prepared in our paper, and their individual effect on electrode material performance was, respectively, discussed to analyze how Mg doping and MgO coating enhance the electrochemical property of LiFePO₄ and where their protrudent part is.

2. Experiment

2.1. Synthesis of Material. Iron oxide (Fe₂O₃) and lithium dihydrogen phosphate (LiH₂PO₄) with Li : Fe : P molar ratios of 1 : 1 : 1 were mixed with citric acid through a balling process for 5 h, followed by calcining at 700°C for 12 h under pure nitrogen to obtain LiFePO₄ powders. Different amounts of magnesia (with LiFePO₄ : MgO molar ratios of 1 : 0.025, 1 : 0.05) were dissolved in 20 mL citric acid solution to prepare magnesium citrate complex and mixed with LiFePO₄ powders through balling for 5 h after drying at 150°C. Finally, all of them were calcined at 700°C for 4 h under inert atmosphere to obtain MgO-coated LiFePO₄/C, referred to as LFP/C-M_x ($x = 2.5, 5$).

Mg-doped samples are prepared by the same steps except the order of adding MgO. Iron oxide (Fe₂O₃), lithium dihydrogen phosphate (LiH₂PO₄), and magnesia (MgO) were mixed with Li : Mg : Fe : P molar ratios of 1 - y : y : 1 : 1 ($y = 0.015, 0.03$), and they were prepared through the above procedures to gain Mg-doped LiFePO₄/C, referred to as L_(1-y)M_yFP/C.

2.2. Characterization of the Samples. The crystallographic structural characterization of samples was analyzed by X-ray diffraction (XRD, Rigaku D/max-2500/pc, CuK radiation). The particle morphology and surface texture of samples were observed with field-emission scanning electron microscope (FE-SEM, S-4800) and transmission electron microscope (TEM, JEOL, JEM-2010).

2.3. Electrochemical Measurements. The electrochemical performance of magnesium modified LiFePO₄/C and pristine cathode materials was evaluated by using columnar cells. Active material powder (80 wt%) was mixed with acetylene black (10 wt%) and poly(vinylidene fluoride) binder (10 wt%) in N-methyl-2-pyrrolidone (NMP) to obtain slurry. The slurry was coated onto an aluminum foil and dried under vacuum at 120°C for 12 h. Finally, the laminate was cut into round dics (1.0 cm in diameter) to be used as working electrode. The electrolyte was 1 mol L⁻¹ LiPF₆ dissolved in the mixture of ethylene carbonate (EC) and diethyl carbonate (DMC) with volume ratio of 1:1. Histogram cells were assembled in the glove box filled with argon gas. Charge-discharge tests were conducted on a battery test system (CT2001A, LAND, China) with cutoff voltages of 2.4 V and 4.2 V (versus

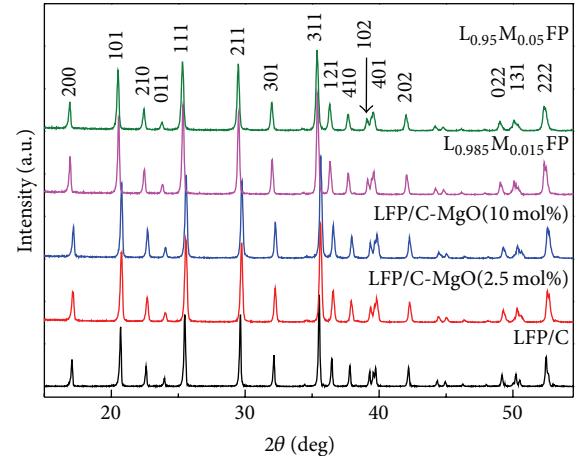


FIGURE 1: XRD patterns of pristine and modified LiFePO₄/C.

TABLE 1: Lattice parameters of pristine and Mg-doped LiFePO₄/C.

Sample	a (Å)	b (Å)	c (Å)	V (Å ³)
LFP/C	10.349	6.015	4.697	292.36
Li _{0.985} Mg _{0.015} FP/C	10.379	6.031	4.713	295.06
Li _{0.95} Mg _{0.05} FP/C	10.387	6.033	4.723	295.94

Li/Li⁺) at different current rates at room temperature. Cyclic voltammetry (CV) measurements were performed on an electrochemical working station (LK2005, LANLIKE, China) at a slow scanning rate of 0.1 mV s⁻¹ within a voltage range of 2.4–4.2 V. And electrochemical impedance spectroscopy (EIS) was employed to characterize the interfacial resistance of cathode using a Chenhua CHI660A electrochemical workstation over the frequency range from 1 MHz to 0.01 Hz with amplitude of 10 mV ms⁻¹ at room temperature.

3. Results and Discussion

3.1. Structural Analyses. XRD patterns of selected samples are shown in Figure 1. Obviously, the crystal phases of all the samples are to be an ordered olivine structure indexed orthorhombic Pnmb, and no extra reflection peak from impurity is observed, indicating that a small amount of magnesia or magnesium does not destruct the lattice structure of LiFePO₄. Table 1 shows the corresponding lattice parameters of the pristine and Mg-doped LiFePO₄/C. The lattice parameters and unit cell volume slightly increase after doping Mg. This result is attributed to the larger ionic radii of Mg²⁺ compared with those of Li⁺.

SEM images of LiFePO₄/C and modified LiFePO₄/C are shown in Figure 2. The LiFePO₄/C particles are spherical or elliptical, and the size ranges from nanometer to micrometer, which is beneficial to high tap density. Low level magnesium doping and coating do not make apparent changes on the morphology, as shown in Figures 2(b) and 2(d). But the morphology changes a lot when the magnesium content rises up to a high level (Figures 2(c) and 2(e)). Small particles aggregate with each other to decrease surface energy in

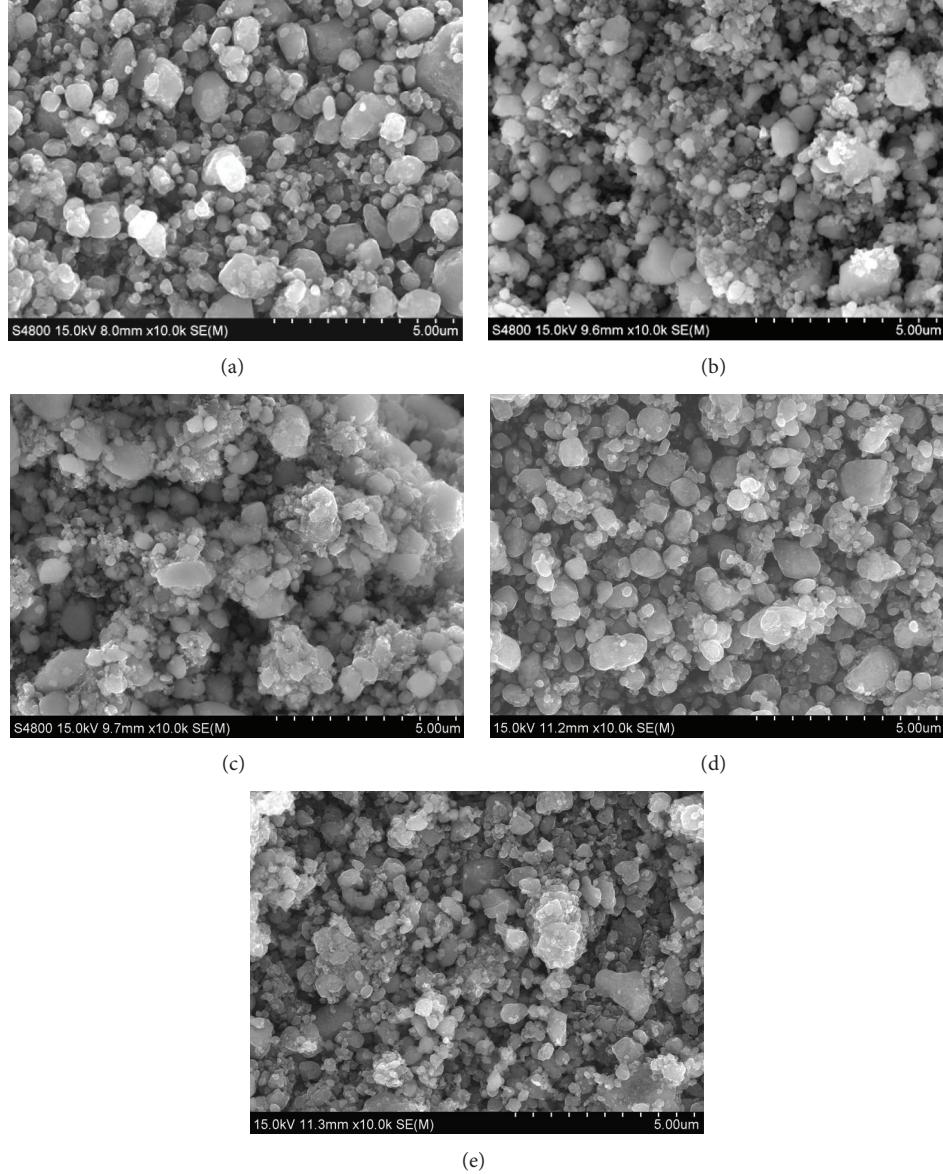


FIGURE 2: SEM images of LFP/C (a), LFP/C-M_{2.5}(b), LFP/C-M₅(c), L_{0.985}M_{0.015}FP/C (d), and L_{0.97}M_{0.03}FP/C (e).

sample LFP/C-M₅, which prevents electrolyte from penetrating through electrode and encumbers the Li-ion diffusion. And it is shown that high level magnesium doping destroys the spherical structure, and the shape of particles becomes irregular in Figure 2(e). Mg steps into LiFePO₄ lattice and substitutes Li site, which is expected to change its morphology more or less.

Figure 3 shows TEM images of sample LFP/C-M_{2.5} and L_{0.985}M_{0.015}FP/C, both presenting a typical core-shell structure with an amorphous carbon wrapping and connecting out of particles. As shown in Figure 3(b), several dark grains with well-crystallized structure are observed around the LiFePO₄ particles, and the gap between every two parallel fringes is measured to be 0.242 nm, which corresponds to the interplanar spacing distance of (111) planes of MgO crystals. MgO comes from the decomposition of magnesium citrate complex, distributing on the surface of LiFePO₄ particles

in magnesium-coated samples. As shown in Figure 3(d), the thickness of the carbon layer is about 2–3 nm in sample L_{0.985}M_{0.015}FP/C, which is favorable to enhance the conductivity but does not affect intercalation/deintercalation of Li⁺ because Li⁺ can readily penetrate through the thin carbon layer.

3.2. Electrochemical Performances. The first discharge curves of LiFePO₄/C and magnesium modified LiFePO₄/C electrodes at 1 C-rate are shown in Figure 4. According to results, proper magnesium modification efficiently enhances the specific capacity. Among all samples, sample L_{0.985}M_{0.015}FP/C shows the best discharge property, whose first special capacity is up to 163 mAh g⁻¹ much higher than the pristine one, only 126.5 mAh g⁻¹. And the superfluous electrons, supplied by Mg ion, increase the amount of electrons in electrode material and advance the electronic conductivity in a further

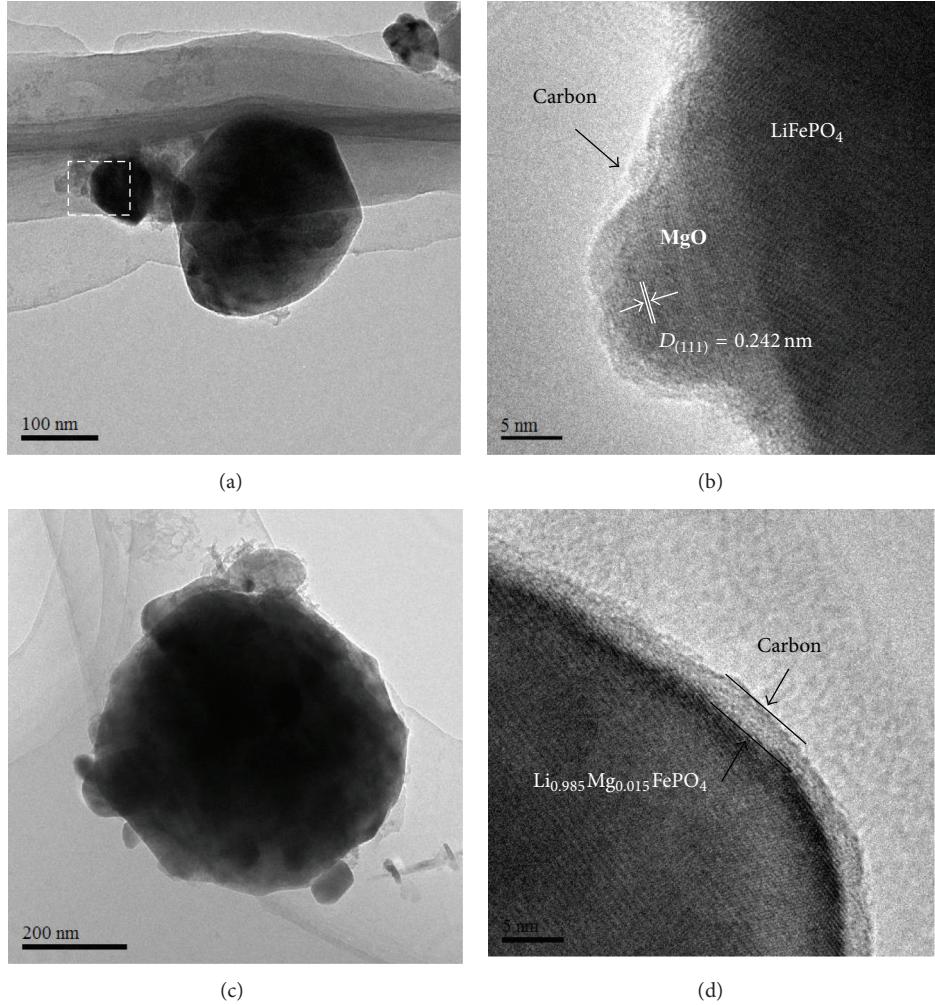


FIGURE 3: TEM images of LFP/C-M_{2.5} (a) and (b), and L_{0.985}Mg_{0.015}FP/C (c) and (d).

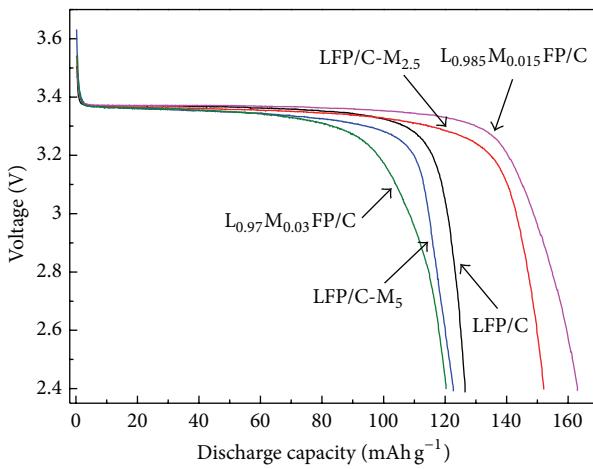


FIGURE 4: The first discharge curves of LiFePO₄/C and magnesium modified LiFePO₄/C electrodes at 1C.

step. Hence the electrochemical performance of LiFePO₄/C is improved by Mg substituting Li-site. But the negative influence is gained when the doping level is high. The first

discharge capacity of L_{0.97}M_{0.03}FP/C is only 120.3 mAh g⁻¹. It possibly owns to the replacement of Mg ion, which handicaps Li⁺ diffusion when superabundant Mg²⁺ occupied lithium channels [21].

The discharge capacity is also enhanced by a certain amount of MgO coating, and it reaches 152.1 mAh g⁻¹ at 1C when MgO content is 2.5 mol%. At the same time, MgO, distributing on the surface of LiFePO₄ particles, prevents electrolyte from corrupting active material and suppresses electrolyte-induced thermal decomposition [18] to improve the cyclic life of electrode, as shown in Figure 5. The discharge capacity of sample LFP/C-M_{2.5} does not fade after 100 cycles at 1C, whose retention ratio is up to 104.2%, while the retention ratios of pristine and sample L_{0.985}Mg_{0.015}FP/C are only 96.8% and 96.7%, respectively. But overmuch MgO coating also shows disadvantageous effect on electrochemistry performance. The first discharge capacity is 122.7 mAh g⁻¹ when Mg content reaches 5 mol%. It is conjectured that redundant MgO should decrease Li⁺ diffusion rate because Li⁺ has to get across the magnesia coating layer when it drills through the interface of electrode and electrolyte.

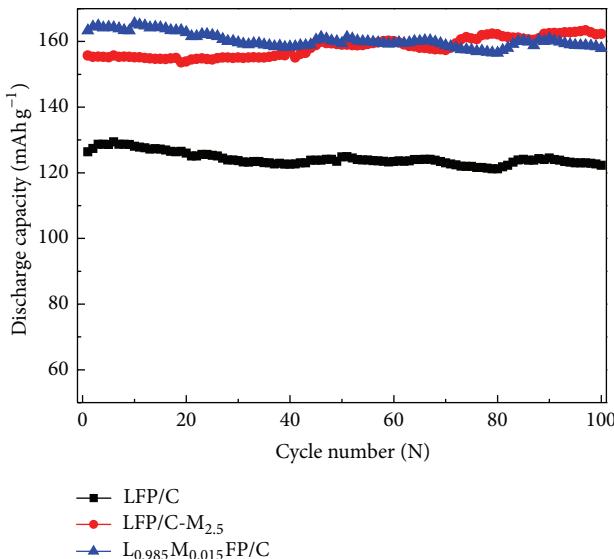


FIGURE 5: Cyclic performance of LiFePO_4/C and magnesium modified LiFePO_4/C electrode at 1C.

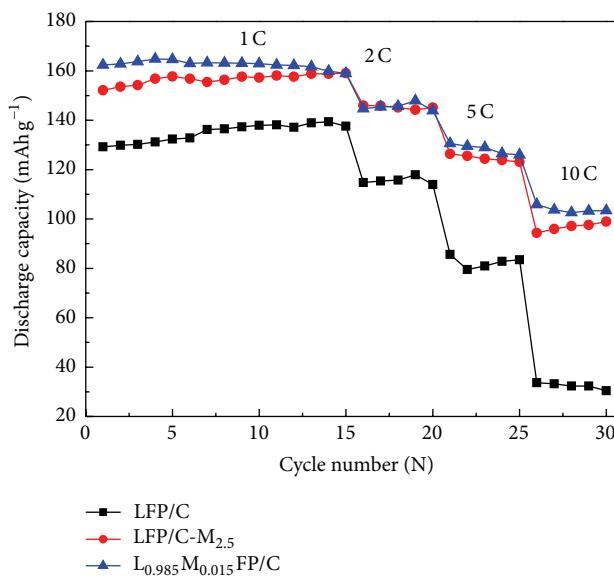


FIGURE 6: Rate capacity of LiFePO_4/C and magnesium modified LiFePO_4/C at 1C, 2C, 5C, and 10C.

Transport of electrons from the particle surface to current collector is the critical step, particularly at high current rate [22]. Therefore, the promotion effect by magnesium modification is obvious at high current. As Figure 6 shows, the average discharge capacity of pristine sample at 2C, 5C, and 10C is only 115.5 mAh g^{-1} , 82.5 mAh g^{-1} , and 32.4 mAh g^{-1} , respectively. Both Mg doping and MgO coating improve the rate capability of the electrode material. The discharge capacity of sample LFP/C-M_{2.5} is 145.2 mAh g^{-1} , 124.6 mAh g^{-1} , and 96.8 mAh g^{-1} at 2C, 5C, and 10C, respectively, while L_{0.985}M_{0.015}FP/C is 145.5 mAh g^{-1} , 128.3 mAh g^{-1} , and 103.7 mAh g^{-1} . It indicates that Mg doping has

TABLE 2: Impedance parameters of samples.

Sample	$R_{\text{ct}} (\Omega)$	$I_0 (\text{mAh} \cdot \text{g}^{-1})$
LFP/C	927.8	27.7
LFP/C-M _{2.5}	352.3	72.9
L _{0.985} M _{0.015} FP/C	289.3	88.8

advantages over MgO coating in terms of improving rate capability.

Constant current charge/discharge tests demonstrate that the electrochemical performance of LiFePO_4 is efficiently improved by both proper magnesium doping and magnesia coating. Moreover, Mg doping is more prominent at prompting the capacity and rate capability of LiFePO_4 , while MgO coating is good at improving cyclic performance.

Cycle voltammetry profiles reflect not only the electrochemical properties of active material, but also the activity of the entire electrode. CV curves of samples LFP/C, LFP/C-M_{2.5}, and L_{0.985}M_{0.015}FP/C at a scan rate from 0.25 to 1 mV s⁻¹ are shown in Figures 7(a), 7(b), and 7(c), respectively. The intensity and area of reduced and oxide peak increase with the scan rate. The linear relationship of the peak current as a function of square root of scan rate is illustrated in Figure 7(d). Thus, the apparent diffusion coefficient can be derived according to the following equation:

$$i_p = (2.6 \times 10^5) n^{2/3} A D_{Li}^{1/2} C_{Li} v^{1/2}, \quad (1)$$

where i_p is the peak current (A), n is the charge transfer number which is one for the electrode reaction, A is the contact area between active material and electrolyte (approximate to the surface area of electrode, 0.785 cm^2), and v is the scan rate (V s^{-1}).

According to the relationship of i_p and $v^{1/2}$ based on (1), biases are painted in Figure 7(d). Apparent diffusion coefficients of LFP/C, LFP/C-M_{2.5}, and L_{0.985}M_{0.015}FP/C are $2.99 \times 10^{-11} \text{ cm}^2 \text{ s}^{-1}$, $3.06 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$, and $4.23 \times 10^{-10} \text{ cm}^2 \text{ s}^{-1}$, respectively. Sample L_{0.985}M_{0.015}FP/C gains the highest value, consistent with the electrochemical performance measurements.

Figure 8 shows the electrochemical impedance spectra (EIS) of the LiFePO_4/C and magnesium modified LiFePO_4/C electrode material after the 5th cycle. The EIS curves are composed of a depressed semicircle in high-frequency region and a straight line in low-frequency region. An intercept at the Z_{re} axis in the high-frequency region corresponds to the ohmic resistance of the electrolyte, followed by a semicircle in the middle-frequency range, indicating the charge transfer resistance, and a straight line in the low-frequency region, related to the Warburg impedance due to the diffusion of the lithium ion in the bulk of the electrode material. The impedance spectra can be described by the equivalent circuit presented in the inset picture, where R_s represents ohmic resistance, R_{ct} represents the charge transfer resistance, Z_w represents the Warburg impedance, and the constant phase element CPE is placed to represent the double-layer capacitance and passivation film capacitance [23]. Fitting results were analyzed by Zview-Impedance 2.80 software, and R_{ct} values are listed in Table 2. The charge transfer

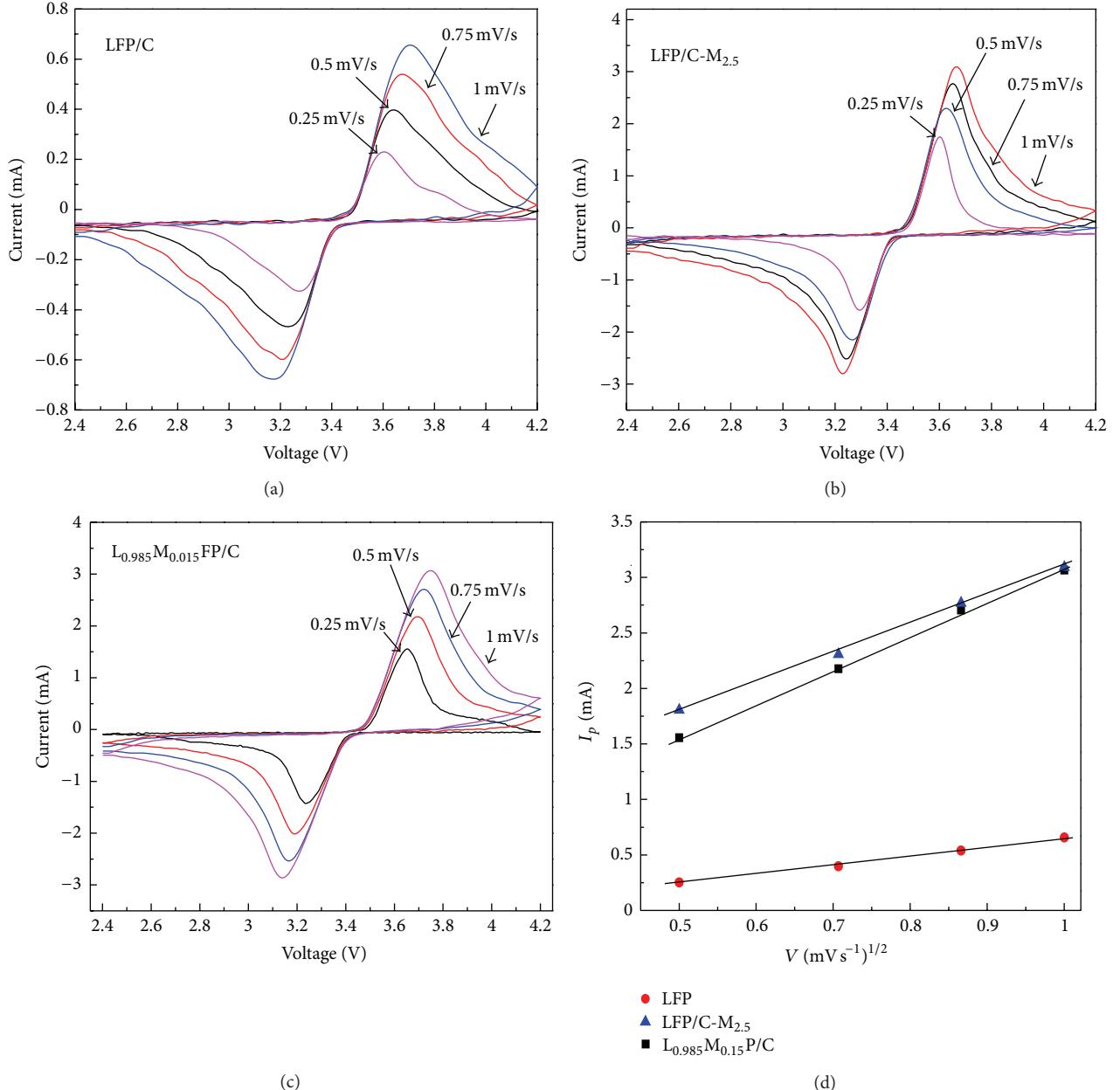


FIGURE 7: Cyclic voltammetry of LiFePO₄/C (a) and magnesium modified LiFePO₄/C (b), (c) in the voltage range of 2.4–4.2 V at scan rates of 0.25, 0.5, 0.75, and 1 mV s⁻¹; (d) the relationship of peak current (i_p) and the square root of scan rate ($v^{1/2}$).

resistance is related to complex reaction of charge transfer process between the electrolyte and the active materials [24]. Compared with the pristine, R_{ct} is obviously decreased by MgO coating and Mg doping. The smaller the charge transfer resistance is, the more feasible it is for lithium-ion and electron transformation. It indicates that magnesium modification is beneficial for active material to overcome the restriction of kinetics in the charge/discharge process and improve electrochemical activity. In addition, the promotion effect from Mg doping is more effectual than MgO coating.

The exchange current density (I_0) is an important parameter of kinetics for an electrochemical reaction and can

measure the catalytic activity of electrodes. It is calculated using the following formula:

$$I_0 = \frac{R \cdot T}{n \cdot m \cdot R_{ct} \cdot F}, \quad (2)$$

where R is the gas constant (8.314 J mol⁻¹ K⁻¹), T is the temperature (298 K), n is the charge transfer number per molecule during the intercalation which is 1 for LiFePO₄, m is the mass of active material on electrode, and F is the Faraday's constant (96,500 C mol⁻¹). The calculated results are listed in Table 2, and it can be clearly concluded that the magnesium modified LiFePO₄/C electrodes exhibit smaller R_{ct} , 289.3,

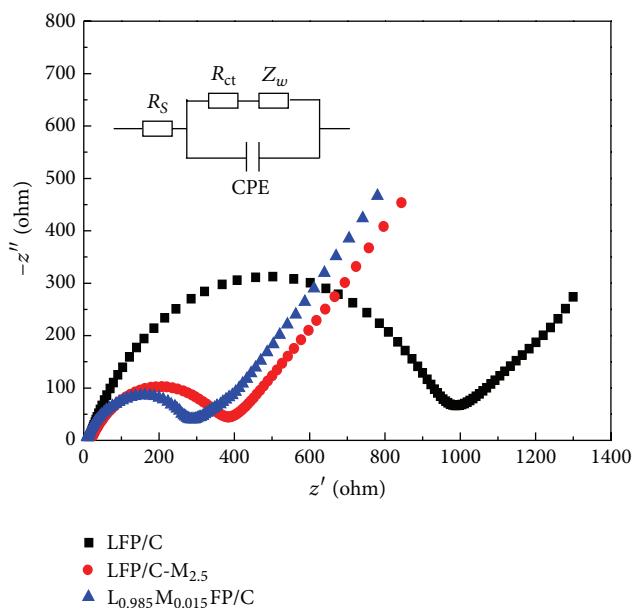


FIGURE 8: The electrochemical impedance spectra profile of samples and equivalent circuit (inset).

352.3 versus 927.8 Ω , than pure LiFePO₄/C. What is more, the L_{0.985}M_{0.015}FP/C sample gives the highest value of exchange current density (I_0), which is in perfect agreement with its excellent electrochemical performance. The EIS results indicate that surface modifying with an appropriate amount of magnesium can improve the electrochemical activity of LiFePO₄/C composite.

4. Conclusion

Mg-doped and MgO-coated LiFePO₄/C compounds were synthesized through a simple solid-state method, and the differences between super-valent cation doping and metal oxide coating on enhancing the electrochemical of LiFePO₄/C material were analyzed. MgO particles, distributing on the surface of LiFePO₄, improve the cyclic performance of electrode, and the discharge capacity of sample LFP/C-M_{2.5} does not fade after 100 cycles. The specific capacity and rate capability of LiFePO₄ are dramatically increased by Mg doping, and the discharge capacity of sample L_{0.985}M_{0.015}FP/C is 163 mAh g⁻¹, 145.2 mAh g⁻¹, 124.6 mAh g⁻¹, and 96.8 mAh g⁻¹ at 1 C, 2 C, 5 C, and 10 C, respectively. Thus, Mg doping is more prominent at prompting the capacity and rate capability of LiFePO₄, while MgO coating has an advantage at improving cyclic performance. CV and EIS results demonstrate that apparent diffusion coefficients, catalytic activity, and the reversibility of LiFePO₄ are improved by both Mg doping and MgO coating.

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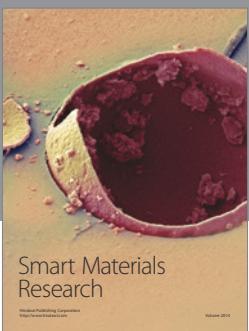
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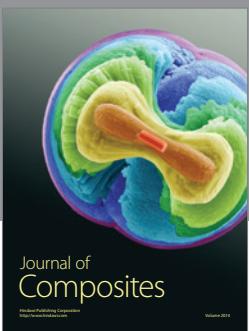
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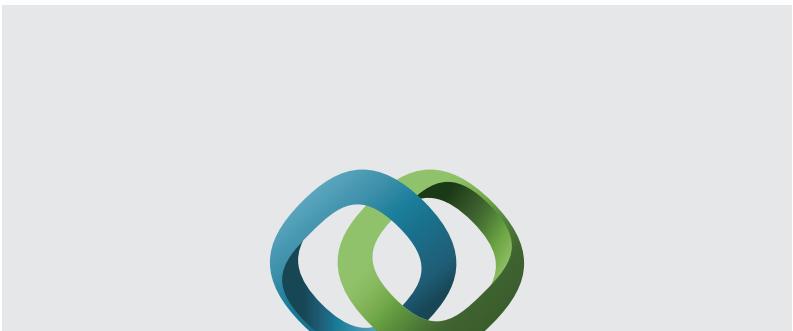
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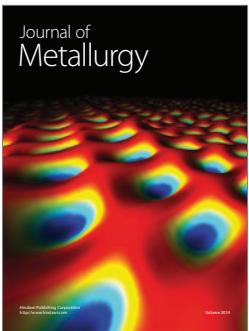


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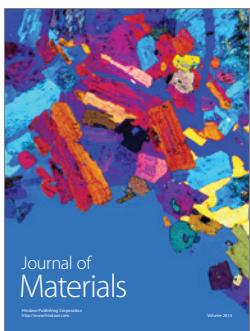
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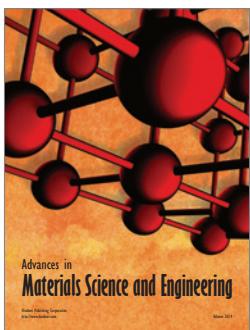
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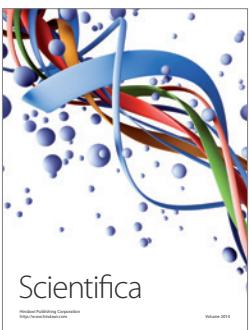
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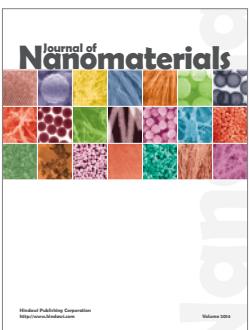
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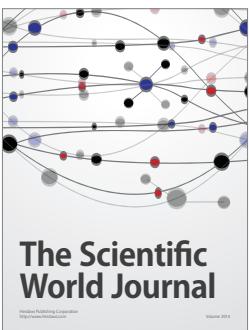
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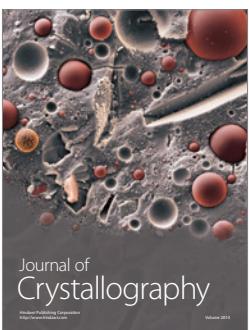
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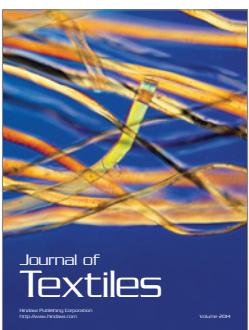
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