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Uniform NiO nanoparticles used as anodes in Li-ion batteries

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Abstract. The uniformly nanosized NiO were prepared via chemical co-precipitation method. Electrochemical characterization of the NiO nanoparticles showed the initial specific discharge capacity reaches 900 mAh/g at 0.1 A/g and 500 mAh/g at 2 A/g, which reveals that the as-synthesized material could be potential candidate of anode material for LIBS.

1. Introduction

Nowadays, to meet expanded appetite for energy, LIBs have been highlighted in various transportable electronics because of its high energy density and environmentally friendly. Graphite, a class of conventional anode materials, cannot satisfy the increasing capacity requirement of lithium ion battery owing to its low academic specific capacity (372 mAh/g).^[1-3] Thus, considerable efforts should be made to explore promising alternative anode materials.

Transition metal oxides (TMOs) afford at least twice the capacity of graphite and could meet the energy demand.^[4-7] However, TMOs suffer rigorous bulk change during reaction, which may arouse the collapse of material structures, suffering the poor cycling stability. And damaged material structures would lead to the reformation of SEI film. Moreover, to solve these problems, utilizing nanosized TMOs as active materials were demonstrated as the efficient strategy, which could remit volume change during charge or discharge process, shorten lithium ions transport pathways, thus improving the electrochemical properties.^[4, 8-10] Thus far, the synthesis of NiO as anodes in LIBs have been mostly realized by hydrothermal or solvothermal methods.^[8, 9] Comparing with these methods, the co-precipitation is a facile, energy-saving and cost-effective strategy to synthesis NiO with shorter reaction periods. however, there are infrequent reports about preparing NiO nanoparticles by chemical co-precipitation method.

In our work, a facile co-precipitation method was developed to prepare uniform NiO nanoparticles as anode materials. And this product exhibits outstanding electrochemical property as anode materials for LIBs.



2. Experimental Section

2.1 Materials

The NiO nano-powder was synthesized by co-precipitation method. 4 g of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ were dissolve in 50 ml DIW and form lucid solution. Additional liquor was formed by dissolving 2.13 g of sodium hydroxide and 3.22 g of sodium carbonate in deionized water. Then the liquor was added dropwise to aforesaid solution with constant stirring after aforesaid solution was heated to 85 °C. Temperature of the entire process was kept at 85 °C for 1 h. When the final solution was naturally waited to be cooling, the simple was cleansed with DIW for 3 times, and dried for 12 h at 65 °C under vacuum. Finally, NiO simples were annealed at 450 °C for 6 h.

2.2 Material characterizations

The as-synthesized NiO power was characterized by a PANalytical X' Pert X-ray diffractometer (Holland), a field emission scanning electron microscope (SEM, Hitachi8010), and transmission electron microscope (TEM, JEM-2100F).

2.3. Electrochemical characterizations

The performances of NiO were investigated by button battery (CR2032). The separator was a thin round sheet of microporous polythene (Celgard 2400). The electrolyte was solution of 1 M LiPF_6 in EC, DMC and EMC (1:1:1). The glovebox (Super 1200/750/900) was the assembled environment of the battery. The charge/discharge test was proceed by the NEWARE-BTS at 0.005-3.0 V. The cyclic voltammetry (CV) test was carried out by an Autolab (PGSTAT302N) potentiostat at 0.001-3.0 V (vs. Li^+/Li) at 0.1 mV/s. Electrochemical impedance spectroscopy (EIS) was explored, which the frequency window was 0.1 MHz to 0.01 Hz.

3. Results and discussion

The NiO nanoparticles were synthesized by a co-precipitation way. The crystalline phases of simples were explored by XRD in Fig.1a. A series of distinct peaks were observed

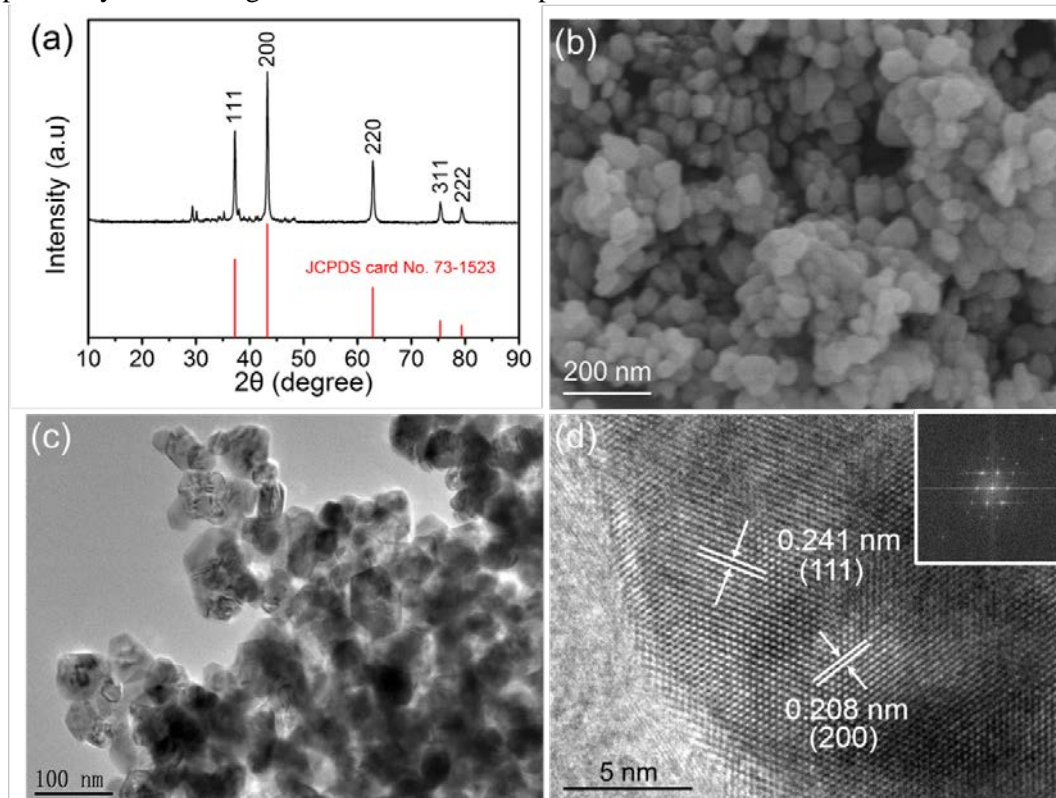


Figure 1. (a) XRD of nano NiO. (b) SEM of nano NiO. (c) TEM of nano NiO and (d) TEM of NiO, the inset showing its corresponding FFT pattern.

at 2θ degree values of 37.2, 43.3, 62.8, 75.4 and 79.3, which correlated with the (111), (200), (220), (311) and (222) lattice plane of NiO with a cubic structure (JCPDS No.73-1523), respectively. Some tiny peaks at 2θ degree values of ~ 30 could be noted from the XRD pattern, which may result from minor impurities. Fig.1b, c show the microscopic structure of NiO nanoparticles, which are displayed by SEM and TEM. The high magnification SEM image presents uniform NiO nanoparticles. The diameter of ~ 30 nm for the nanoparticles are observed by TEM image. The HR-TEM image (Fig.1d) displays the polycrystalline nature of NiO. Interplanar spacing of 0.241 and 0.208 nm could be correlated with the (111) and (200) planes of NiO, respectively. The homologous FFT (the inset) was taken from the HRTEM image of the part of Fig. 1d.

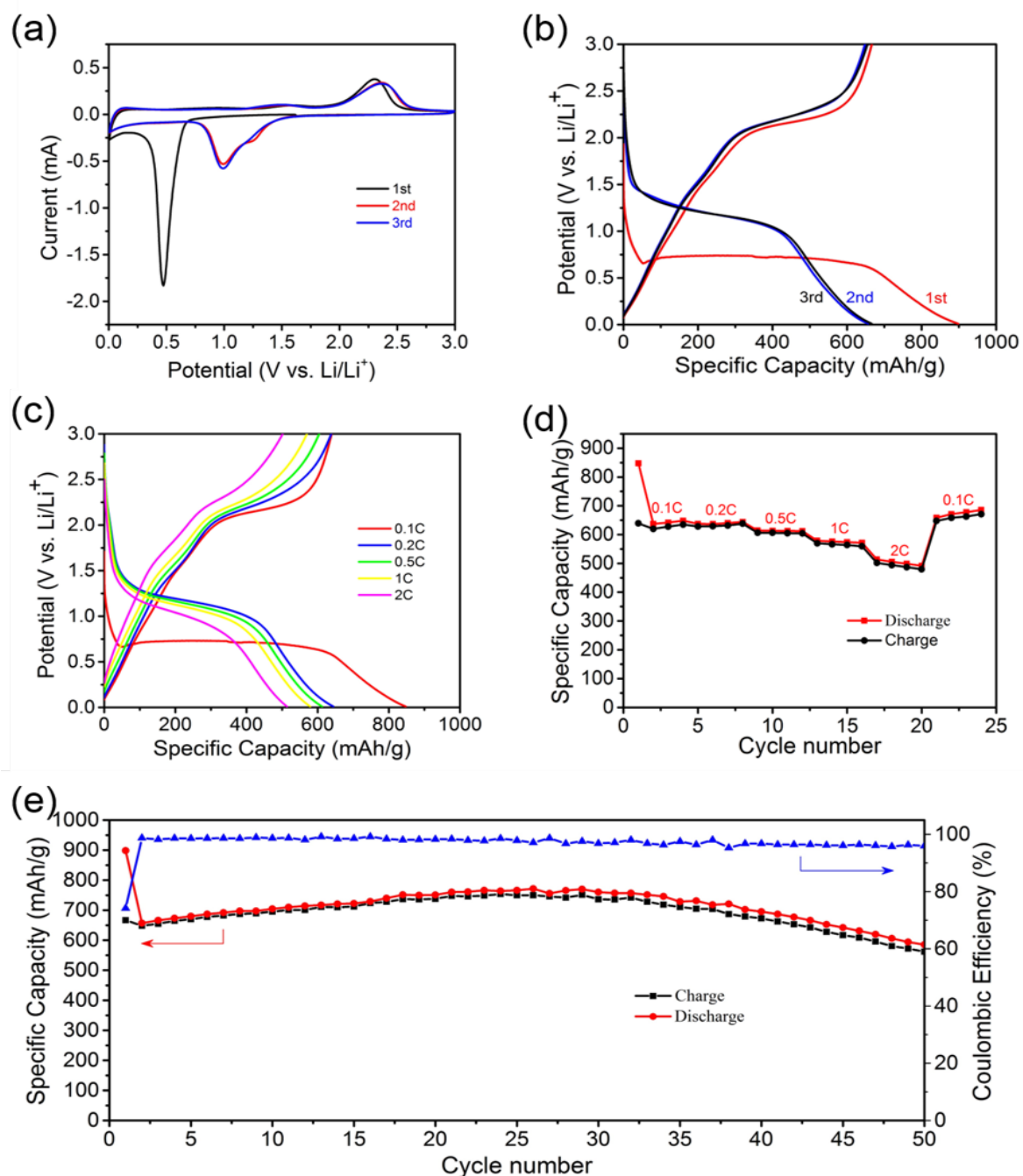


Figure 2. (a) First three consecutive CV of nano NiO. (b) Galvanostatic discharge/charge curves and (c) Galvanostatic discharge/charge curves of NiO at varying discharge rate. (d) Rate performances of NiO. (e) Cycling performance of NiO at 100mA/g.

Fig.2a display cyclic voltammograms (CV) of the NiO nanoparticle. In first loop, One dominant peak at 0.5V can be viewed as to the initial transformation from NiO into Ni, which Followed the equation: $(\text{NiO}+2\text{Li}^++2\text{e} \rightarrow \text{Ni}+\text{Li}_2\text{O})$. In the following loops, the cathodic peak became broad and shifted to 1.1 V due to the structural or structure qualification during the initial lithium process. Meanwhile, the anodic peaks at ~ 2.3 V could be regarded as the transformation from metallic Ni into NiO, which Followed the equation: $(\text{Ni}+\text{Li}_2\text{O} \rightarrow \text{NiO}+2\text{Li}^++2\text{e})$, and have not transformed significantly in the following loops. The galvanostatic charge and discharge curves for chief 3 cycles of NiO samples at the current density of 0.1 C (100 mA/g) are revealed in Fig.2b. Between 0.5 and 0.7 V, a long plateau of voltage was discerned in the initial discharge curve of NiO samples, corresponding to the redox peak in CV (Fig.2a). The potential range in the subsequent loops came to unanimous which the peak located in CV curve.^[9] Rate capabilities of NiO anodes were explored at different current density of 0.1 - 2 A/g (Fig.2d). At 0.1, 0.2, 0.5, 1, and 2 A/g, the NiO products exhibit medial discharge capacity of 648, 644, 613 and 575, 500, 677 mAh/g, respectively, which are much better than other previous reported NiO anodes (Fig.2c). It is noted that the NiO electrode exhibits even larger capacity (677 mAh/g) at 0.1 A/g after rate test. The cycling performance of nano NiO are shown in Fig. 3e at 100 mA/g. The samples display a preferable first discharge capacity of ~ 900 mAh/g. Then it fades to 656 mAh/g after the 2nd discharge, and finally retain ~ 590 mAh/g after 50 loops. And the marvelous capacity may be accredit to the generation of SEI layer during the first loop. Moreover, NiO anodes reveal a high Coulombic efficiency of $\sim 99\%$ after first loop.

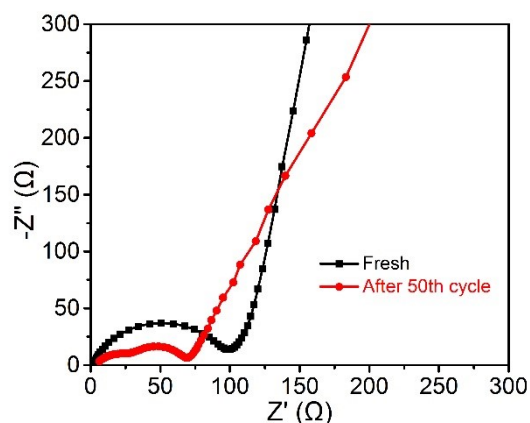


Figure 3. Nyquist plots for the first and 50 cycles.

The Nyquist plot is displayed in Fig.3. The charge-transfer resistance (R_{ct}) of the NiO anode was calculated as 97Ω for the first cycle. After 50 cycles, it determined a smaller R_{ct} value of 65.5Ω . This transformation is mainly be accredit to the impedance of a surface chemical layer and a charge-transfer resistance dominating reaction process of the electrode.

4. Conclusion

In short, NiO nanoparticles were synthesized using a simple co-precipitation method. The NiO electrode exhibit a preferable first capacity of ~ 900 mAh/g and kept a high capacity of ~ 590 mAh/g at 0.1 A/g, and exhibited superb rate capability. Therefore, the properties of NiO materials can be effectively improved by nanocrystallization.

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