Nanoconfinement of Low-Conductivity Products in Rechargeable Sodium–Air Batteries

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Abstract

To alleviate the high polarization and short cycle lifetime of rechargeable Na–air batteries, ordered mesoporous carbon (OMC) was synthesized and evaluated as a nanostructured conductive matrix to host low-conductivity products generated during the discharge of Na–air batteries. The OMC had high specific surface area ($1544 \text{ m}^2/\text{g}$) and a narrow pore size (2.7 nm), with the voltage polarization of 1.5 V, lower than that of the commonly used Super P carbon black ($\sim 1.8 \text{ V}$). Although the carbonate-based electrolyte was decomposed to produce Na_2CO_3 , the OMC cathode allowed reversible formation and decomposition of Na_2CO_3 and exhibited stable cycling behavior with low polarization for 20 cycles with a delivered capacity of 500 mAh/g at the current density of 100 mA/g.

Keywords

Na–air battery; Porous carbon material; Discharge product morphology; Size-confinement; Carbonate-based electrolyte

1. Introduction

Recent interest in developing eco-friendly renewable energy sources has been largely stemmed from the uncontrolled use of fossil fuels, which has sharply raised awareness of environmental problems around the world and has inevitably led to rapid exhaustion of natural reservoirs of fossil fuels. Development of renewable energy resources requires high-efficiency energy

conversion and large-scale, low-cost, high-density energy storage devices. The energy density of traditional Li ion batteries is inadequate to meet the increased requirements, especially for electric or hybrid electric vehicles. Li-,¹⁻⁴ Fe-,⁵ Si-,⁶ Zn-,⁷ Al-,⁸ and Na-air⁹⁻¹² batteries have attracted a great deal of attention mainly because of the extremely high energy density of the metal-air battery system, which is close to the energy density of fossil fuels. One advantage of Na-air batteries over Li-ion or Li-air batteries is its low material cost and its natural abundance, as sodium is 30 times cheaper than lithium. Moreover, the energy density of an Na-air battery is similar to the maximum practical energy density of a Li-air battery (1600 Wh/kg).⁹ As a common challenge to be addressed for all air-based battery technologies, excessive discharge products can accumulate on the cathode surface; most of these have very low electrical conductivity, and thus tend to inactivate surface reaction sites and block oxygen diffusion channels. Hence, the over-accumulation of discharge products can lead to the increase in polarization during charging of the Na-air battery and eventual termination of the oxygen evolution reaction.¹³ To reduce the accumulation of discharge products for decreased charge potential and improved cycling performance, morphological properties of the cathode (e.g. surface area, pore volume, and pore size) need to be tailored while employing different catalysts. Recently, Liu *et al.* demonstrated that the graphene nanosheets (GNSs) can be used as cathode materials in Na-air batteries, delivering a high discharge capacity and maintained stable cycling behavior up to 10 cycles.¹⁴ However, the batteries' charge potential increased gradually during cycling due to the accumulation of discharge products. Li *et al.* employed nitrogen-doped GNSs as a cathode material in Na-air batteries and confirmed the excellent electrocatalytic activity of this material for the oxygen reduction reaction, delivering a discharge capacity double that of undoped GNSs; however, the charge potential also increased during cycling.¹⁵ Jian *et al.*

demonstrated that the use of 3D interpenetrating carbon nanotube paper as a binder-free cathode material in Na–air batteries yielded high discharge capacity and low overpotential; however, these batteries' charge potential increased drastically during charging.¹⁶ Clearly, it is necessary to limit the accumulation of discharge products; however, to the best of our knowledge, there have been no reports of research efforts to confine the size or arrangement of discharge products in the vicinity of the cathode to decrease the charge potential and improve the cycling behavior of Na–air batteries. In the present work, OMC having 2.7 nm size pore¹⁷ (OMC-2.7) was employed as a cathode material to control the formation and subsequent decomposition of discharge products, and thus to reduce polarization and improve cycling behavior.

2. Experimental

2.1. Synthesis of Ordered Mesoporous Carbons (OMCs)

For synthesis of OMC-2.7, triblock copolymer Pluronic® F127, resols synthesized according to a previously reported method,¹⁸ and tetraethyl orthosilicate were prepared as a template, carbon source, and silica source, respectively. With slight modification and scale-up from a previously reported OMC preparation method,¹⁹ large amounts of OMC-2.7 powder could be synthesized. Typically, 24.0 g of block copolymer F127 was dissolved in a solution of 120 g of ethanol and 7.5 g of 0.2 M hydrochloric acid, and stirred for at least 1 h at 40 °C to make a clear solution. Next, 38.65 g of a 20 wt% ethanolic resol solution and 15.6 g of tetraethyl orthosilicate were added in sequence. The mixture was stirred for 5 h and then transferred into flat and wide dishes. Ethanol in solution was evaporated by heating the samples for 24 h at 40 °C, and then the samples were thermopolymerized for 24 h at 100 °C. The temperature transition from 40 to 100 °C was carried out slowly to avoid boiling of remaining ethanol, which could destroy the desired

mesoporous structure. The solidified transparent product was scraped from the dishes and ground into fine powders, which were then calcined to yield a carbon–silica nanocomposite. This calcination was carried out in a tubular furnace for 2 h at 900 °C under argon flow, the heating rates used to reach this temperature were 1 °C min⁻¹ below 600 °C and 5 °C min⁻¹ above 600 °C. To remove the silica from the carbon–silica nanocomposites, they were immersed in a 10 wt% solution of HF in deionized water; the samples were then washed with distilled water and ethanol before finally dried for 12 h at 100 °C under vacuum.

The OMC-17 was synthesized using poly(ethylene oxide)-b-polystyrene block copolymer (PEO-b-PS) as a structure-directing agent.²⁰ The molecular weight of PEO-b-PS was 30,200 g mol⁻¹ (16.5 wt % PEO) and its polydispersity index was 1.34. In a typical synthesis, 0.4 g of PEO-b-PS was dissolved in 10 mL of tetrahydrofuran (THF). Then, 0.19 g of 0.2 M HCl was added. After 10 min, 0.78 g of TEOS and 3.87 g of resols' ethanolic solution were added and the solution was stirred for 1 h. The solution was poured into a Petri dish for THF evaporation. After 8 h, the composite film was thermopolymerized at 100 °C in an oven for 1 day. The as-synthesized film was carbonized in N₂ atmosphere at 450 °C for 3 h and 900 °C for 2 h in a tube furnace (heating rate, 1 °C min⁻¹). Finally, the C-SiO₂ nanocomposite was treated with a 10 wt % HF solution to remove SiO₂.

2.2. Electrode Preparation and Measurement

The carbon material (OMC-2.7, OMC-17, and Super P) and polyvinylidene fluoride binder were mixed in a mass ratio of 9:1 in N-methyl-2-pyrrolidone, and the resulting slurry was coated on the gas diffusion layer (GDL; SGL-35BC carbon paper, SGL Carbon) in a dry room. The coated electrode was dried for 12 h at 100 °C under vacuum to remove residual solvent. Circular

electrodes 1.8 cm in diameter were cut from the coated GDL material; the loading of carbon materials on the GDL was about 0.5 mg. R2032 coin-type cells were assembled in an argon-filled glove box with water and oxygen content both less than 0.1 ppm. The cell consisted of a metallic Na foil anode (thickness 400 μm) and the prepared carbon electrodes, separated by glass fiber paper (GF/A microfiber filter paper, Whatman; thickness 0.26 mm, pore size 1.6 μm), and filled with a solution of 0.5 M sodium triflate (NaCF_3SO_3) in propylene carbonate (PC) as the electrolyte. For comparison, we also used 0.5 M solutions of NaCF_3SO_3 in DEGDME, TEGDME, and DME as ether-based electrolytes. Cells were placed in an oxygen-filled chamber with pressure slightly higher than 1 atm at room temperature and electrochemically tested under galvanostatic cycling applied using a potentiostat (VMP3, Biologic Instruments). Full discharge and charge testing was performed over the voltage range of 1.8–4.3 V with a current density of 100 mA/g. To test the cells' cycling performance, charge–discharge cycling tests were carried out in the time-controlled mode at a current density of 100 mA/g for 5 h, or 200 mA/g for 10 h. Specific capacities were calculated based on the carbon weight (lower X-axis of graph) and we also expressed the absolute capacity on the upper X-axis. Cyclic voltammetry (CV) measurements were carried out at room temperature using the VMP3 potentiostat, scanning at the rate of 0.1 mV/s in the voltage range of 1.8–4.3 V.

2.3. Characterization

The morphology of the carbon electrode materials and discharge products were observed by means of field emission scanning electron microscopy (SEM, S-4800, Hitachi) and high resolution transmission electron microscopy (HR-TEM, JEM-2010, JEOL). The discharged electrodes were protected from exposure to moisture during the transfer to the SEM and TEM

chamber by transporting them in a portable vacuum oven applied in the glove box. The surface area and porosity of mesoporous carbon were measured by using an automated gas sorption analyzer (Autosorb iQ, Quantachrome), using nitrogen gas as the adsorbate at 77 K. The specific surface area was calculated using the Brunauer–Emmett–Teller method, and the pore diameters were obtained from the desorption of the isotherm calculated using Density Functional Theory method. The calculated model is a N₂ at 77 K on carbon (slit/cylindrical pore, Non Local Density Functional Theory equilibrium model) and fitting error is 0.508% (lower than 1% is considered reliable). To examine the discharge products on the electrode after discharging or charging, after the electrochemical testing using the PC-based electrolyte the electrodes were collected, washed with PC, and carefully wiped for further characterization. These electrodes were vacuum-dried for XRD analysis (D/MAX 2500, Rigaku; Cu-K α radiation source); this analysis was conducted over the experimental 2 θ range between 29.5° and 42.5° at a scan rate of 0.3°/min. XPS analysis (Perkin-Elmer, Model PHI 5600) was conducted to investigate the electronic state of Na in the electrodes after discharging and charging. Transmission-mode FTIR analysis (FTIR, Nicolet iS50, Thermo) was conducted in an N₂-filled glove box, although it was difficult to distinguish the discharge products from the infrared-absorbing OMC-2.7 and GDL substrate materials. Moreover, making pellets for this analysis can damage the discharge products in the OMC-2.7 electrodes. Accordingly, we equipped the FTIR instrument with an attenuated total reflectance (ATR) apparatus and used this ATR-FTIR to analyze the peaks of the discharge product. HEXRD analysis was carried out at sector 11-ID-C of Advanced Photon Source (APS) of Argonne National Laboratory. The wavelength of the X-ray beam was fixed at 0.11165 Å.

3. Results and discussion

3.1. Structure of OMC and Super P electrodes

To benchmark the electrochemical performance of OMC as a cathode material in the Na–air battery system, commercially purchased Super P carbon black was also evaluated as a cathode material. Cathodes fabricated from these two materials were imaged using scanning electron microscopy (SEM) and high-resolution transmission electron microscopy (HR-TEM). The OMC consisted of large carbon particles including many regularly spaced nanoscale pores each approximately 5 nm wide (Figure 1A and inset), whereas the commercial Super P consisted of carbon nanoparticles of average diameter around 50 nm (Figure 1B); HR-TEM analysis of the Super P particles revealed densely packed irregular 002 lattice fringes within each particle (Figure 1B inset). Porosity of the two materials was investigated by nitrogen adsorption–desorption isotherm analysis (Figure 1C); the Brunauer–Emmett–Teller surface areas of the OMC and Super P were thus calculated to be 1544 and 66 m²/g, respectively. The pore diameter of the OMC-2.7 was calculated to be 2.7 nm, consistent with the TEM results, and the pore volume of the OMC-2.7 was 1.56 cc/g, much higher than the 0.267 cc/g pore volume of the Super P. Meanwhile, we also collected the SEM, TEM and BET data of another kind of OMC (OMC-17) having a bigger pore size (17 nm) (see Figure S1A and B) to check the impact of the pore size on Na-air batteries.

3.2. Electrochemical properties

For electrochemical testing of Na–air batteries, we tried using various ether-based electrolytes — sodium triflate salt (NaCF₃SO₃) in diethylene glycol dimethyl ether (DEGDME),

tetraethylene glycol dimethyl ether (TEGDME), and dimethoxyethane (DME)— but their use always led to erratic potential profiles during charging (see Figure S2). These potential behaviors make operation of the battery impossible.²¹ For this reason, we used a carbonate-based electrolyte rather than an ether-based electrolyte; namely, the electrolyte used was a solution of NaCF_3SO_3 in propylene carbonate (PC). Linear sweep voltammograms of the 0.5 M NaCF_3SO_3 in PC showed that this PC electrolyte decomposed when the potential was raised to 4.5 V (vs. Na^+/Na), meaning that the voltage of the battery using this electrolyte cannot be higher than 4.5 V (Figure S3). On the other hand, the lower charge potential can increase stability during charging and discharging.

Figure 2A shows the voltage profiles of Na–air batteries using OMC-2.7 and Super P cathodes during the first cycle conducted at a constant current density of 100 mA/g. During their first discharges, the OMC-2.7 cell and Super P cell showed specific capacities of 7987 and 4864 mAh/g, respectively. Corresponding cyclic performance data (Figure 2A inset) reveals that both carbon materials suffered from capacity fading because excessive discharge product was not able to be fully decomposed during the subsequent charge. Despite the capacity fading, the OMC-2.7 cathode maintained greater discharge capacity than the Super P cathode throughout their first ten cycles. In addition, it is obvious from the voltage profiles that the difference in the discharge and charge potentials of the OMC-2.7 cathode is smaller than that of the Super P cathode. This is confirmed by cyclic voltammograms collected for each cathode versus Na metal reference electrodes in oxygen atmosphere (Figure 2B). The cathodic peak potential of the oxygen reduction reaction of OMC-2.7 (2.01 V) was higher than that of Super P (1.86 V). The onset potential of the oxygen evolution reaction of OMC-2.7 was 3.26 V whereas that of Super P was about 3.71 V, showing that the OMC-2.7 facilitated the oxidation of the discharge product during

the charging process. Moreover, both the anodic current and the cathodic current of the OMC-2.7 cathode were much higher than those of the Super P cathode, suggesting that the OMC-2.7 cathode could sustain higher operation rates. Meanwhile, there were no pronounced cathodic and anodic peaks in the cyclic voltammograms of either sample when tested in argon (the inset of Figure 2B), indicating that oxygen is the reaction source for the Na-air battery.

The cycling performances of the two cathodes were also tested by controlling the discharge time to 5 h to limit the discharge capacity to 500 mAh/g. The charge potential of the OMC-2.7 cathode was mostly unchanged throughout the first 20 cycles, but that of Super P increased progressively with cycling (from 3.968 to 4.399 V). The OMC-17 with a larger pore size was also tested with the same condition (see Figure S1C), and its charge potential increased progressively with cycling like Super P. It seems that large pores of OMC-17 don't have an effect on stabilization of charge potential. Furthermore, when the time limit was increased to 10 h and the corresponding capacity was increased to 1000 mAh/g (Figure S4A, B), the charge potential of both carbon materials increased with the cycle number after the 5th cycle, but the OMC-2.7 cathode showed relatively lower charge potential increase up to the 10th cycle (from 3.743 to 4.038 V) than Super P (from 3.955 to 4.435 V). This observation further confirmed that deep discharging could produce excessive discharge products that become increasingly harder to be decomposed during subsequent charging, reducing the reversibility of the battery. The cycling performance results at a higher capacity (2000 mAh/g) with the higher current density (200 mA/g) are plotted in Figure S4C and D; in these tests, the OMC-2.7 cathode also showed lower charge potential than the Super P cathode. In short, OMC-2.7 cathodes showed lower charge potential and polarization than Super P cathodes in all tested conditions because the OMC-2.7 cathode can significantly relieve the problem of pore clogging and cathode encrustation by

discharge products, similar to the effect of using high-surface-area GNSs.¹⁴ For the rate test at the same capacity condition (500 mAh/g), OMC-2.7 showed a good rate capability with little increase in charge potential at a higher rate (see Figure S7b).

3.3. Characterization of the discharge product

Although it was clear that the highly ordered pore structure and large surface area of the OMC-2.7 cathodes improved their electrochemical performance, the relevant mechanism and the discharge products needed to be investigated for better understanding of the Na–air battery system. Based on previous work on the Li–air battery system,^{22–24} we speculated that both the structure of the cathode and the morphology of the discharge product could affect the electrochemical performance. To identify the discharge products in the Na–air battery, the OMC-2.7 and Super P cathodes were analyzed after discharging and charging by means of X-ray diffraction (XRD) using a Cu K α source (Figure 4A). No peaks corresponding to Na₂O₂ were found, which is believed to be the main discharge product of Na–air batteries using ether-based electrolytes. Instead, several peaks assignable to Na₂CO₃ were detected in X-ray diffraction patterns of the Super P cathode; these were likely formed by the decomposition of the carbonate electrolyte during discharging. However, there was no corresponding XRD evidence of Na₂CO₃ on the OMC-2.7 cathode after discharging, even though the Na–air batteries using OMC-2.7 and Super P both used the same PC electrolyte. To verify the presence of Na₂CO₃, the OMC-2.7 and Super P cathodes were analyzed by means of X-ray photoelectron spectroscopy (XPS) after discharging and charging were carried out (Figure 4B). The Na 1s edge was shifted to 1071.7 eV, which matched the binding energy of Na 1s in Na₂CO₃.²⁵ The binding energy for Na 1s of Na₂O₂ is 1072.5 eV;²¹ thus, the observed product was most likely Na₂CO₃, not the Na₂O₂ that was

found in cells using ether-based electrolytes. The Na 1s peak decreased in intensity after charging. Moreover, attenuated total reflection Fourier transform infrared (ATR-FTIR) analysis of the OMC-2.7 cathode after the first discharge showed characteristic peaks of Na_2CO_3 (Figure S5).²¹ It is worth mentioning that the characterization techniques of XRD using a Cu $K\alpha$ source, Raman spectroscopy, and FTIR are surface-sensitive techniques with limited penetration depth. The surface material detected by these techniques could have been exposed to the carbonated-based electrolyte, and potentially also to CO_2 generated by the decomposition of solvents, for an extended period of time. Hence, it is not surprising to observe Na_2CO_3 on the surface. It is of more interest to characterize the material under the surface, and especially material that might be buried deep inside channels of the OMC-2.7 cathode. Hence, high-energy X-ray diffraction (HEXRD) using 115 keV X-ray source ($\lambda = 0.11165 \text{ \AA}$) was utilized to directly investigate the discharge products inside the Na–air cells without the need to open the cells after charging or discharging. When the cells were discharged, a doublet was observed in the HEXRD spectra at about 2.1° ($\lambda = 0.11165 \text{ \AA}$), consisting of the (110) peak of Na_2O_2 and the (202) peak of Na_2CO_3 . In addition, the width of these spectral peaks was greater for the OMC-2.7 cathode than for the Super P cathode, indicating that the crystallite size of the discharge products in the OMC-2.7 was smaller. HEXRD data clearly showed that the discharge product, $\text{Na}_2\text{O}_2/\text{Na}_2\text{CO}_3$, appeared after discharging and disappeared after charging (Figure 4C).

The morphology and structure of the discharge product on both carbon cathodes were further investigated after discharging by means of TEM, energy-filtered TEM (EF-TEM) and selected area electron diffraction (SAED). TEM analysis showed that the structures of OMC-2.7 and Super P did not change after discharging; especially, the pore structure of OMC-2.7 was well maintained throughout the discharge/charge processes (Figure 5A, D). Interestingly, the Na_2CO_3

particles produced on the Super P cathode were approximately 1 μm in size, and thus large enough to be easily detected (Figure 5D), whereas the Na_2CO_3 on the OMC-2.7 cathode was not visually detected (Figure 5A). Accordingly, EF-TEM analysis was conducted to visualize the distribution of Na, showing that Na penetrated inside pores of the OMC-2.7 rather than depositing on its surface (Figure 5B). This result is mainly attributed to capillary action, which forces the electrolyte to infiltrate the OMC-2.7 pores and causes Na compounds to form within the pores. Contrastingly, Na_2CO_3 discharge products formed primarily on the surface of the Super P cathode, and formed much larger aggregated particles (Figure 5E). Crystalline Na_2CO_3 (JCPDS No. 77-2082) could barely be detected in selected area diffraction patterns of the OMC-2.7 cathode (see Figure 5C), but was clearly detected in the Super P cathode.

A common problem for batteries based on metal–oxygen reactions has been the formation of compounds with poor electronic conductivity during discharging, such as oxides,²⁶ peroxides,^{27,28} and carbonates;^{29,30} the portion of these products that cannot be decomposed during charging accumulate and eventually degrade electrochemical performance. Therefore, to improve cycling performance, systems that generate only peroxides are being widely pursued.^{31,32} Contrastingly, the present work shows that Na_2CO_3 can also be reversibly decomposed when this discharge product is confined in nanochannels of the carbon matrix. Na_2CO_3 and Na_2O_2 have very low electronic conductivity, and thus it is obvious that only thin layers of $\text{Na}_2\text{CO}_3/\text{Na}_2\text{O}_2$ on the carbon surface can find good electron conducting pathways that will allow them to be electrochemically decomposed during the successive charge. By contrast, excessive deposition of $\text{Na}_2\text{CO}_3/\text{Na}_2\text{O}_2$ on the Super P carbon will lead to the formation of a thick layer of $\text{Na}_2\text{CO}_3/\text{Na}_2\text{O}_2$ (see Figure 5D); subsequent decomposition will occur primarily at the inner surface close to the carbon, cutting away the outer shell of material and removing its

electron-conduction pathway. The remaining $\text{Na}_2\text{CO}_3/\text{Na}_2\text{O}_2$ shell will in turn act as a physical barrier to the transportation of O_2 and Na^+ , leading to an incremental decrease of active electrochemical surface area (see Figure S6) and a continuous increase of the charge potential for Na–air cells using Super P cathode (see Figure 3B). In the case of Na–air cells using OMC-2.7 cathodes, the discharge products ($\text{Na}_2\text{CO}_3/\text{Na}_2\text{O}_2$) were primarily confined in nanochannels (2.7 nm in diameter) of the OMC, thereby limiting the maximum distance between the discharge products and the carbon surface to about 2.7 nm, and ensuring the effective decomposition of $\text{Na}_2\text{CO}_3/\text{Na}_2\text{O}_2$ during the successive charging process without causing the loss of the active electrochemical surface area; this allowed a stable charge potential to be maintained (see Figure 3A). Although charge potential was also a little increased during further cycling (see Figure S7), the effect of OMC-2.7 nanochannels can be verified by comparison with Super P. Meanwhile, when the cells were deeply discharged to 1000 mAh/g, the amount of $\text{Na}_2\text{CO}_3/\text{Na}_2\text{O}_2$ generated could exceed the hosting capacity of the nanochannels, causing the deposition of a thick layer of $\text{Na}_2\text{CO}_3/\text{Na}_2\text{O}_2$ on the surface of OMC-2.7 and leading to the same degradation mechanism as for the Super P cathode. This can also explain why good cycling performance of air-based batteries was achieved in other studies exclusively by actively controlling the batteries' discharge capacity, rather than their discharge potential.^{27,28,31-33}

4. Conclusion

When used as a cathode material, OMC-2.7, which has high surface area, large pore volume, and a highly arranged porous structure, delivered greater discharge capacity, lower polarization, and better cycle life performance than Super P. Moreover, we found that the highly ordered pores of OMC-2.7 dictated the morphology and crystallinity of the discharge product, leading to

improved electrochemical performance of the Na–air battery. Although the PC electrolyte that was decomposed during discharging to produce Na_2CO_3 , the OMC-2.7 cathode allowed reversible, controlled formation and subsequent decomposition of this Na_2CO_3 , and thus performed better than the Super P cathode. Our finding implies that the morphology of the carbon matrix plays a critical role in determining the electrochemical performance of air-based battery technologies; pore volume dictates the achievable reversible capacity and pore size influences the reversibility, and thus cycle lifetime, of the battery.

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Vitae

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Figures

Figure 1. Characterizations of OMC-2.7 and Super P materials: (A, B) SEM and (inset) TEM micrographs of (A) OMC-2.7 and (B) Super P; (C) Nitrogen adsorption–desorption isotherm and (inset) pore size distribution plots for OMC-2.7 and Super P.

Figure 2. (A) Voltage profiles during the first cycle conducted at the current density of 100 mA/g; (inset) corresponding discharge capacities versus cycle number. (B) CV curves of Na–air batteries with OMC-2.7 and Super P cathodes; scan rate: 0.1 mV/s; (inset) CV curves taken in oxygen-free conditions (under argon gas).

Figure 3. Discharge-charge performance during the first twenty cycles of Na-air batteries using cathodes of (A) OMC-2.7 and (B) Super P. Galvanostatic, time-controlled mode; current density: 100 mA/g; capacity: 500 mAh/g; controlled time: 5 h.

Figure 4. (A) XRD, (B) XPS and (C) HEXRD spectra of OMC-2.7 and Super P cathodes after discharging and charging.

Figure 5. (A, D) TEM micrographs, (B, E) EF-TEM micrographs (the sodium element is denoted by green dots), and (C, F) selected area diffraction patterns of two carbon cathodes fully discharged to 1.8 V: (A, B, C) OMC-2.7 cathode and (D, E, F) Super P cathode.

Graphical abstract