

Article

Lattice Spacing, Morphology, Properties, and Quasi—In Situ Impedance of Ternary Lithium-Ion Batteries at a Low Temperature

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Abstract: The study about the low-temperature performance of lithium-ion batteries (LIB) is of great significance at extreme temperatures, such as polar scientific research, space exploration, deep-sea exploration, military fields, and so on. In this study, normal devices and symmetrical devices were fabricated by ternary $\text{Li}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ as cathode and graphite as anode at 25 and -20°C . The results show that the specific discharge capacity of normal device is up to 120 mAh g^{-1} at 1 C and 25°C . The specific capacity and energy density at 0.2 C and -20°C are 106.05 mAh g^{-1} and 376.53 mWh g^{-1} , respectively, which can reach 92.82% of that at 1 C and 25°C . The value of activation energy E_a of the interface reaction of the LIB is calculated to be 63.72 kJ/mol by the Arrhenius equation. When the temperature dropped from 25 to -20°C , the lattice spacing of $\text{Li}_{1-x}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ hardly changed, while the lattice spacing (002) of graphite reduces $0.00248\text{ \AA}$. In addition, some cracks were observed on the charged cathode at -20°C . We carried out quasi-in situ electrochemical impedance spectroscopy (EIS) when the voltages of normal device discharged to 3.8, 3.6, 3.4, 3.2, and 3.0 V. Unlike the relationship of voltage–resistance at 25°C , the values of the series resistance (R_s), charge transfer resistance (R_{ct}), and ion transfer resistance (R_{it}) gradually decrease as the voltage decreases at -20°C . Compared with the resistance of the symmetrical device based on the anode at 25°C , the values of R_s and R_{it} at -20°C both obviously increase. The main reason of performance degradation for normal device at -20°C is large ion transfer resistance and the decrease of lattice spacing of the graphite (002).

Keywords: lithium-ion batteries; lattice spacing; morphology; impedance



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1. Introduction

The invention of LIB has provided great convenience and benefits to us and the world, so John. B. Goodenough [1], Whittingham M. Stanley [2], and Akira Yoshino [3] had won the 2019 Nobel Prize. Although LIB have been commercialized in some fields, LIB have many disadvantages in the following aspects, such as low-temperature performance [4], fast charge–discharge technology [5], solid-state battery [6], endurance mileage [7], safety performance [8], and so on [9,10]. Among them, the study of low-temperature performance is of great significance in extreme temperatures, such as polar scientific research, space exploration, deep-sea exploration, military fields, and so on. At present, the research on low temperature performance of LIB mainly has the following directions. The first is to modify the material of LIB. For example, the modification or substitution of anode materials (C, Ti, Li alloying, Si, and Ge-based anodes and composite anodes) [11], the modification or substitution of cathode [$\text{Li}_3\text{V}_2(\text{PO}_4)_3$ phosphates and $\text{LiNi}_{1-x-y}\text{Co}_x\text{Al}_y\text{O}_2$ oxides] [12], and the optimization of the composition of electrolytes [13]. Another solution is to add a heating device and control system, such as adding a heating device outside the battery or battery pack, or adding a heating electrode inside the battery [14]. Although adding a heating device inside or outside of LIB can effectively increase the performance of LIB at low

temperature, it will increase the cost and danger of the whole battery. Therefore, a complete understanding of the reasons for the performance degradation of LIB at low temperature is of great significance for the next step to improve the performance of LIB at low temperature [15]. AC impedance technology is an advanced method of measuring the resistance in a perturbation way. There are many studies on measuring the internal resistance of LIB at room temperature by AC impedance [16], but there are few studies on measuring the internal resistance of lithium-ion battery at low temperature by AC impedance [17–20]. Most of the studies used a coin battery as the research object. The performance of coin battery at the room temperature can reflect the performance of a commercial high-capacity cylindrical battery. However, compared with room temperature conditions, small coin batteries under low temperature conditions often can not fully and truly reflect various internal resistance, mass transfer, heat transfer, and performance of commercial high-capacity batteries. In this study, we use the commercial cylindrical battery as the research object to study the battery performance at low temperature. In addition, the morphology of the electrode at low temperature is rarely studied in the previous research. Here, we studied the morphology change of the electrode at room temperature and low temperature. In addition, we found that, unlike the relationship of voltage–resistance at 25 °C, the value of the series resistance (R_s), charge transfer resistance (R_{ct}), and ion transfer resistance (R_{it}) gradually decreases as the voltage decreases at −20 °C. In this study, we fabricated normal devices and symmetrical devices by ternary $\text{Li}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ as cathode and graphite as anode at 25 and −20 °C. The normal device is defined as cathode/separate film-electrolyte/anode. The symmetrical device is defined as cathode/separate film-electrolyte/cathode or anode/separate film-electrolyte/anode. The change of lattice spacing of $\text{Li}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ and graphite was assessed. The specific capacity and energy density of normal devices were measured at 25 and −20 °C. The value of E_a on the interface reaction of the LIB is calculated by the Arrhenius equation at different temperatures (25, 0, −20, and −30 °C). The quasi-in situ EIS was carried out when a normal device discharged to different voltages (3.8, 3.6, 3.4, 3.2, and 3.0 V). The EIS of symmetrical cells based on cathode or anode were also studied at 25 and −20 °C.

2. Materials and Methods

Cathode preparation: $\text{Li}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$, acetylene black, and poly(vinylidene fluoride) were mixed in a mass ratio of 8:1:1. The solvent is *N*-methylpyrrolidone. The above mixture was ball milled for 3 h. The ball milling speed is 300 r.p.m. Then, the cathode paste was prepared on the aluminium foil by slit coating. The thickness of the aluminium foil is 20 microns. Then, the cathode film was dried at 120 °C. The cathode film was then pressed on a rod rolling machine.

Anode preparation: Graphite and acetylene black with poly(vinylidene fluoride) was mixed in a mass ratio of 8:1:1. The solvent is *N*-methylpyrrolidone. The above mixture was ball milled for 3 h. The ball milling speed is 300 r.p.m. Then, the anode was prepared on the copper foil by slit coating. The thickness of the copper foil is 11 microns. Then, the anode was dried at 120 °C. The anode was then pressed on a rod rolling machine.

The composition of the electrolyte: The solvent is a mixture of ethylene carbonate (EC), ethyl methyl carbonate (EMC) and dimethyl carbonate (DMC) with the 1:1:2 volume ratio of EC:EMC:DMC. The mass percentage of LiPF_6 and vinylene carbonate is 10% and 2%, respectively.

The sizes of normal device (cylindrical battery) are 14 mm diameter and the 50 mm height. The mass loadings on cathode are 6.25 g. The sizes of symmetrical device (coin cell) is 15 mm in diameter.

Measurement and Characterization

The conditions of the X-ray diffraction (XRD) measurement (SmartLab XRD, Rigaku Corporation, Tokyo, Japan) are 5 degrees per minute and a range of 10–80 degrees. The accessory device for low temperature is Anton Paar TTK450. The X-ray wavelength is

1.54056 Å [$\lambda(\text{Cu K}\alpha)$]. SEM was carried out by scanning electron microscopy (Thermo Fisher Scientific SEM GX4, Waltham, MA, USA). The solid electrolyte interphase (SEI) film was formed by a 0.05 C constant current charging for three hours, with 3.5 V cut-off voltage, and a 0.1 C constant current charging for 4 h with 4.0 V cut-off voltage. The battery performance is characterized by the Xinwei battery test cabinet. Charge–discharge curves were obtained at 1 C (100 mA g^{−1}) and 0.2 C (20 mA g^{−1}). After reaching cut-off voltage of the charge and discharge, the static time is 5 and 10 min, respectively. The cut-off voltage of the charge and discharge is 4.2 V and 2.7 V, respectively. The temperature of measurement is 25, 0, −20, and −30 °C, using a high-low temperature chamber (Ksun Test Equipment Co., Ltd., Guangzhou, China).

Electrochemical impedance test: The electrochemical impedance spectroscopy (EIS) was carried out on the photoelectrochemical workstation (CIMPS-2 pro, Zahner, Kronach, Germany). The frequency range is from 7 MHz to 0.05 Hz. The amplitude is 10 mA. During the discharge process (at 1 C and 25 °C and at 0.2 C and −20 °C), the quasi-in situ EIS test was carried out when the voltage drops by 0.2 V. The bias voltage for the EIS test of a symmetrical device is 0 V.

3. Results and Discussion

The morphology of $\text{Li}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ in the cathode was characterized by scanning electron microscopy (SEM), as shown in Supplementary Materials of Figure S1 in ESI. The size of $\text{Li}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ particles is 5–10 microns. $\text{Li}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ particles are composed of smaller particles. The size of smaller particles is between 500 nm and 1 micron as shown in Figure S2. The morphology of the charged cathode at 25 and −20 °C are shown in Figure 1a,b. After charging, some particles were formed on the surface of the cathode. Compared with that of the charged cathode at 25 °C, the number of particles on the charged cathode at −20 °C is less. The morphology of the anode is shown in Figure S2. The morphology of the anode after forming the SEI film is shown in Figure 1c at 25 °C and Figure 1d at −20 °C. It can be seen from Figure 1c that graphite cannot be observed after the SEI film is formed. The elements of the SEI film are shown in Figure S3. The results show that the SEI film contains C, O, F, and P elements, and their contents are 67.45, 24.85, 6.66, and 1.04%, respectively. Compared with that of the charged anode at 25 °C, some cracks were observed on the charged cathode at −20 °C.

The X-ray diffraction (XRD) of ternary $\text{Li}_{1-x}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ in cathode and SEI-graphite in anode were carried out at 25 and −20 °C. The diffraction pattern of ternary $\text{Li}_{1-x}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ is shown in Figure 2a,b. The enlarged image shows that the diffraction peak at 18.56 degree of the cathode has almost no shift, which means that the lattice spacing of $\text{Li}_{1-x}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ hardly changed when the temperature dropped from 25 to −20 °C. The diffraction pattern of SEI-graphite in anode is shown in Figure 2c,d. The enlarged graph shows that the diffraction peak of the anode shifts to a higher angle as the temperature drops from 25 to −20 °C. The peak (002) at 26.54 degree is moved to 26.56 degrees. According to the Bragg Equation (1),

$$2d \sin 2\theta = n\lambda \quad (1)$$

the (002) interplanar spacing of graphite is reduced from 3.35575 Å to 3.35327 Å, a decrease of 0.00248 Å. The small lattice spacing will increase resistance when the lithium migrates out from the graphite layers, which will affect the discharge capacity. In addition, in Figure 2a, the line gradually increases from 10 to 40 (2theta/degree). The reason for this phenomenon may be that $\text{Li}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ produces a certain degree of amorphous in the process of delithiation process at the cathode.

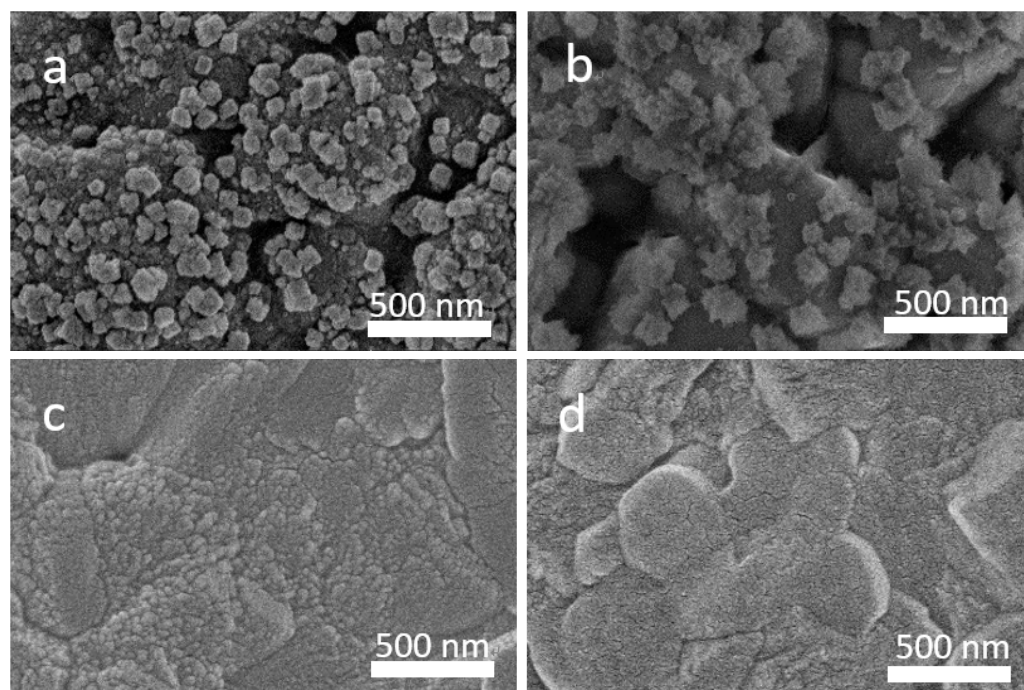


Figure 1. The images of $\text{Li}_{1-x}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ in cathode film (a) 25 °C, (b) −20 °C; the images of graphite in anode film (c) 25 °C, (d) −20 °C.

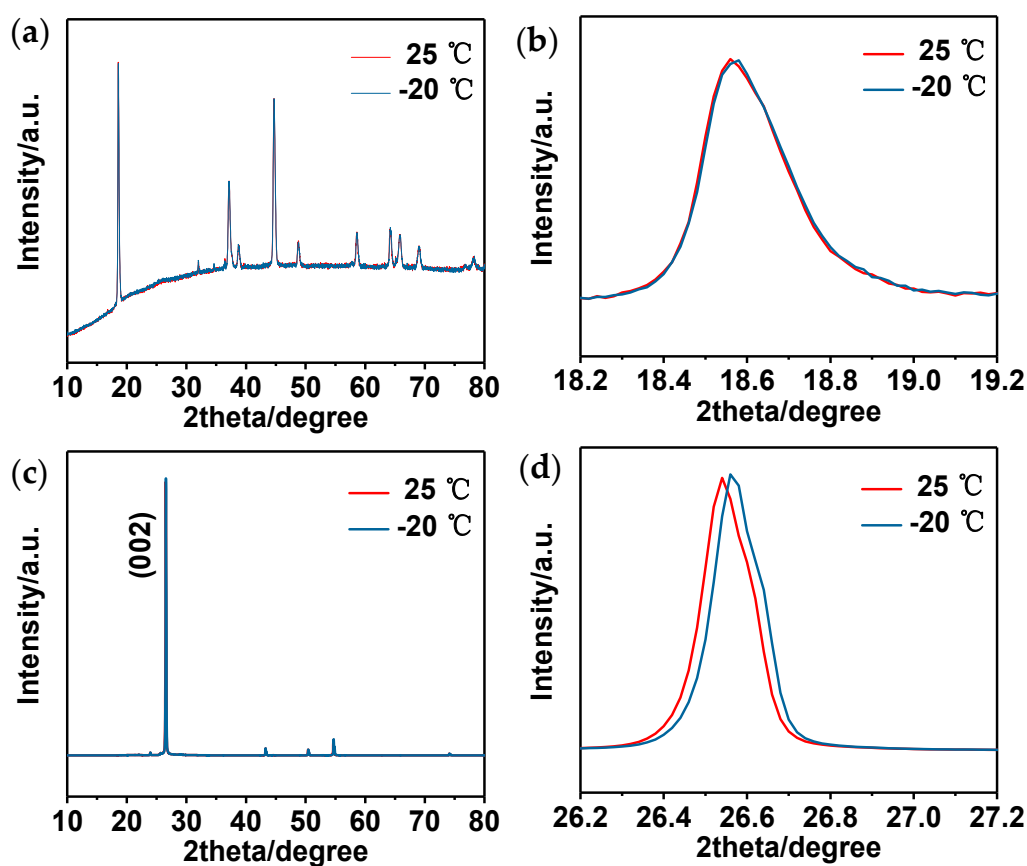


Figure 2. Powder X-ray diffraction (XRD) patterns of ternary $\text{Li}_{1-x}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ in cathode (a,b) and SEI-graphite in anode (c,d) at 25 and −20 °C.

The charge and discharge curves of normal device at 1 C (100 mA g^{-1}) and 25°C are shown in Figure 3a. The results show that the specific discharge capacity of the normal device is 118.40, 114.20, 108.23, 104.26, and 98.30 mAh g^{-1} at the 1st, 100th, 200th, 300th, and 400th cycle. Figure 3b shows the relationship between the cycle index, specific discharge capacity, and capacity retention. After 400 cycles of charge and discharge, the specific discharge capacity and capacity retention of normal device remain at 98.30 mAh g^{-1} and 82.40%, respectively. This result shows that the stability of the battery is good. The specific discharge and energy density curves of normal device at 25°C and -20°C are shown in Figure 3c,d, respectively. Compared with the performance at 25°C , the specific capacitance and energy density are reduced. The specific capacity and energy density at -20°C are $106.05 \text{ mAh g}^{-1}$ and $376.53 \text{ mWh g}^{-1}$, respectively. The results indicate that the performance at -20°C can reach 92.82% of that at 25°C .

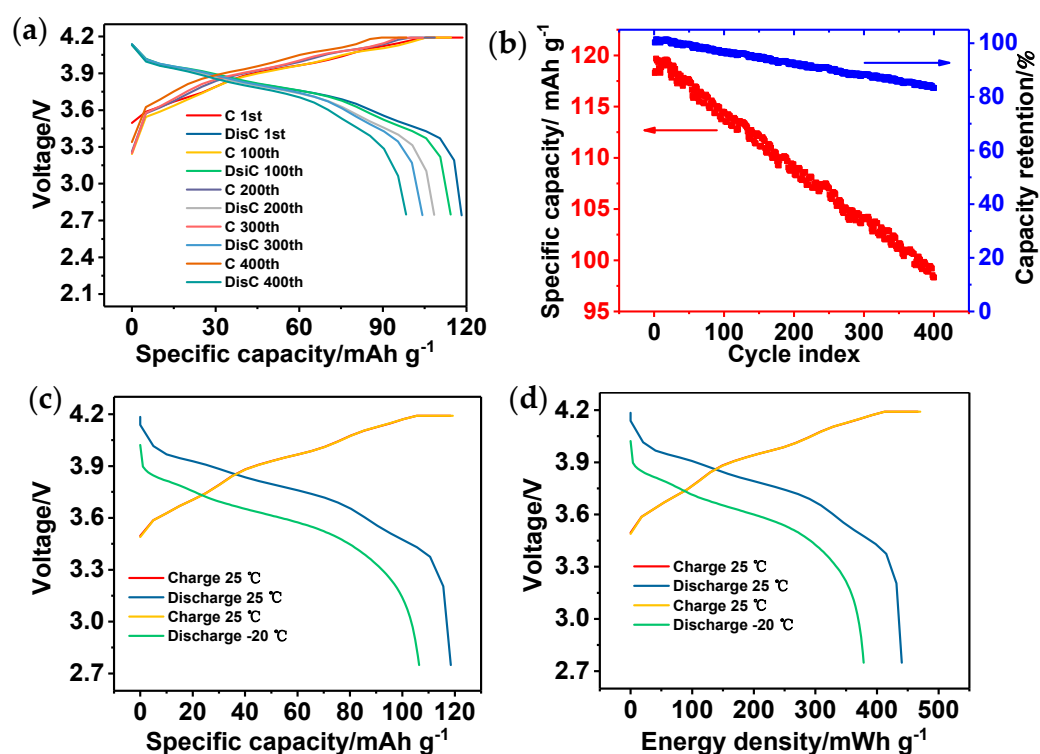


Figure 3. The charge–discharge performance of a normal device: (a) charge–discharge curves at 1 C (100 mA g^{-1}) and 25°C for 1st, 100th, 200th, 300th, and 400th cycle; (b) specific capacity and its retention over 400 cycles at 1 C (100 mA g^{-1}) and 25°C ; (c) the first charge–discharge cycle curves at 1 C (100 mA g^{-1}) and 25°C and at 0.2 C (20 mA g^{-1}) and -20°C ; (d) the first energy density cycle curves at 1 C (100 mA g^{-1}) and 25°C and at 0.2 C (20 mA g^{-1}) and -20°C .

To study the reason of performance degradation for a normal device at -20°C , we carried out EIS of normal devices at 25, 0, -20 , and -30°C . Schematic diagram and equivalent circuit for normal device are shown in Figure 4a. The curves were fitted with Z-view software and the impedance data were obtained. The impedance data are listed in Table 1. The series resistance (R_s) is the pure resistance for the device. In the discharge process, the charge transfer resistance (R_{ct}) is resistance of the delithiation process at the cathode, and ion transfer resistance (R_{it}) is resistance of the lithiation process at anode and SEI film. The value of R_{ct} and R_{it} is very important for evaluating the dynamic process of the lithiation process and the delithiation process. As the temperature decreases from 25 to -30°C , the value of R_s gradually increases from 0.086 to 0.128Ω , as shown in Figure 4b,c. The total resistance of R_{ct} at cathode and R_{it} at anode increased from 0.064 to 19.848Ω . The above results indicate that temperature has a greater influence on R_{ct} and R_{it} than the R_s . In other words, the reason of performance degradation for normal device at -20°C is the

increasing effect of R_{ct} and R_{it} . The relationship curve between the total resistance of R_{ct} and R_{it} and temperature shows an exponential function, as shown in Figure 4d,e:

$$k = Ae^{\frac{-E_a}{RT}} \quad (2)$$

$$\ln k = \frac{-E_a}{RT} + \ln A \quad (3)$$

$$\ln \frac{1}{R_{ct}} = \frac{-E_a}{RT} + \ln A \quad (4)$$

According to Arrhenius Equations (2)–(4), the value of E_a on the interface of the lithium ion battery electrode is 63.72 kJ/mol. The impedance of normal device is not clear when the voltage decreases in the discharge process. We carried out quasi-in situ EIS when the normal device discharged to different voltages (3.8, 3.6, 3.4, 3.2, and 3.0 V). The impedance curves of normal device for different voltages at 25 °C and −20 °C are shown in Figure 4f–h. As the voltage decreases from 3.8 to 3.0 V, the R_s of the battery gradually increases from 0.141 to 0.156 Ω at 25 °C. The value of total resistance of R_{ct} and R_{it} increases from 0.097 to 0.457 Ω . The above results show that, as the voltage reduces from 3.8 to 3.0 V at 25 °C, the increases of total resistance ($R_s + R_{ct} + R_{it}$) of the battery are small. In addition, the impedance curve is not composed of one semicircle but two semicircles when the battery voltage is 3.4, 3.2, and 3.0 V, as shown in Figure 4g. This result indicates that the two semicircles should be derived from the R_{ct} of the cathode and the R_{it} of the anode. Unlike the relationship of voltage–resistance at 25 °C, the R_s , R_{ct} , and R_{it} tend to gradually decrease as the voltage decreases at −20 °C, as shown in Figure 4i. The value of R_s is reduced from 0.094 to 0.091 Ω , and the value of total resistance ($R_{ct} + R_{it}$) is reduced from 7.139 to 4.445 Ω .

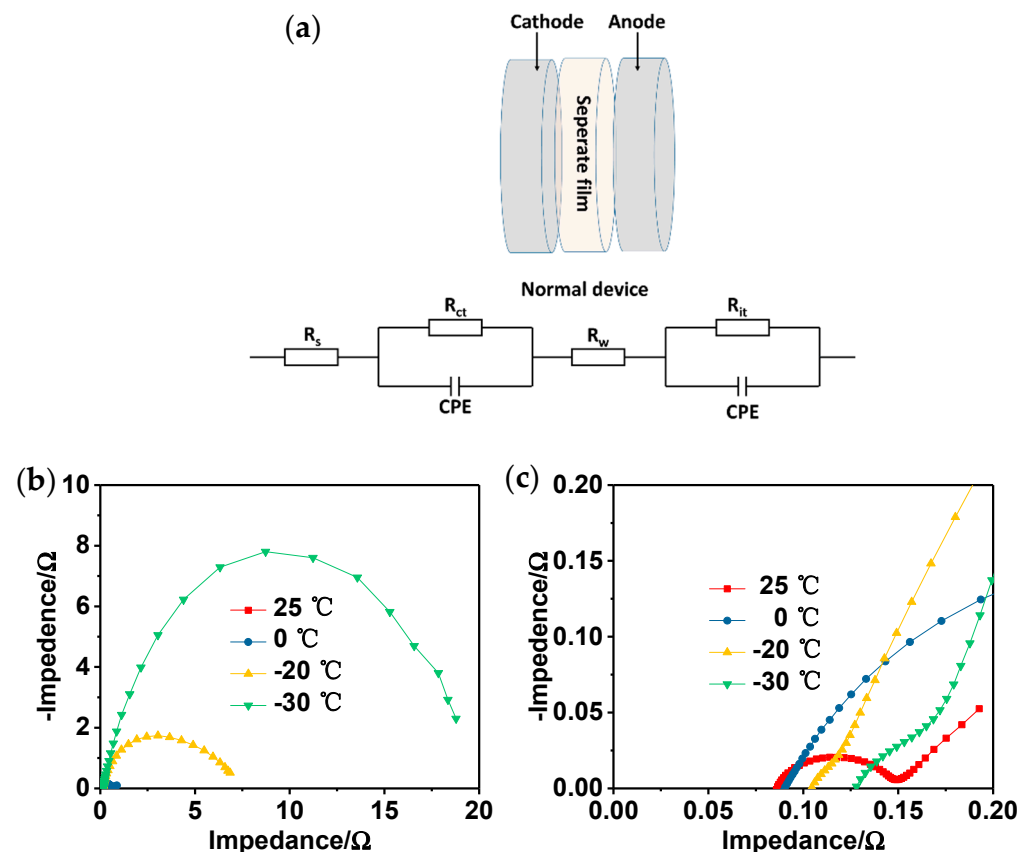


Figure 4. Cont.

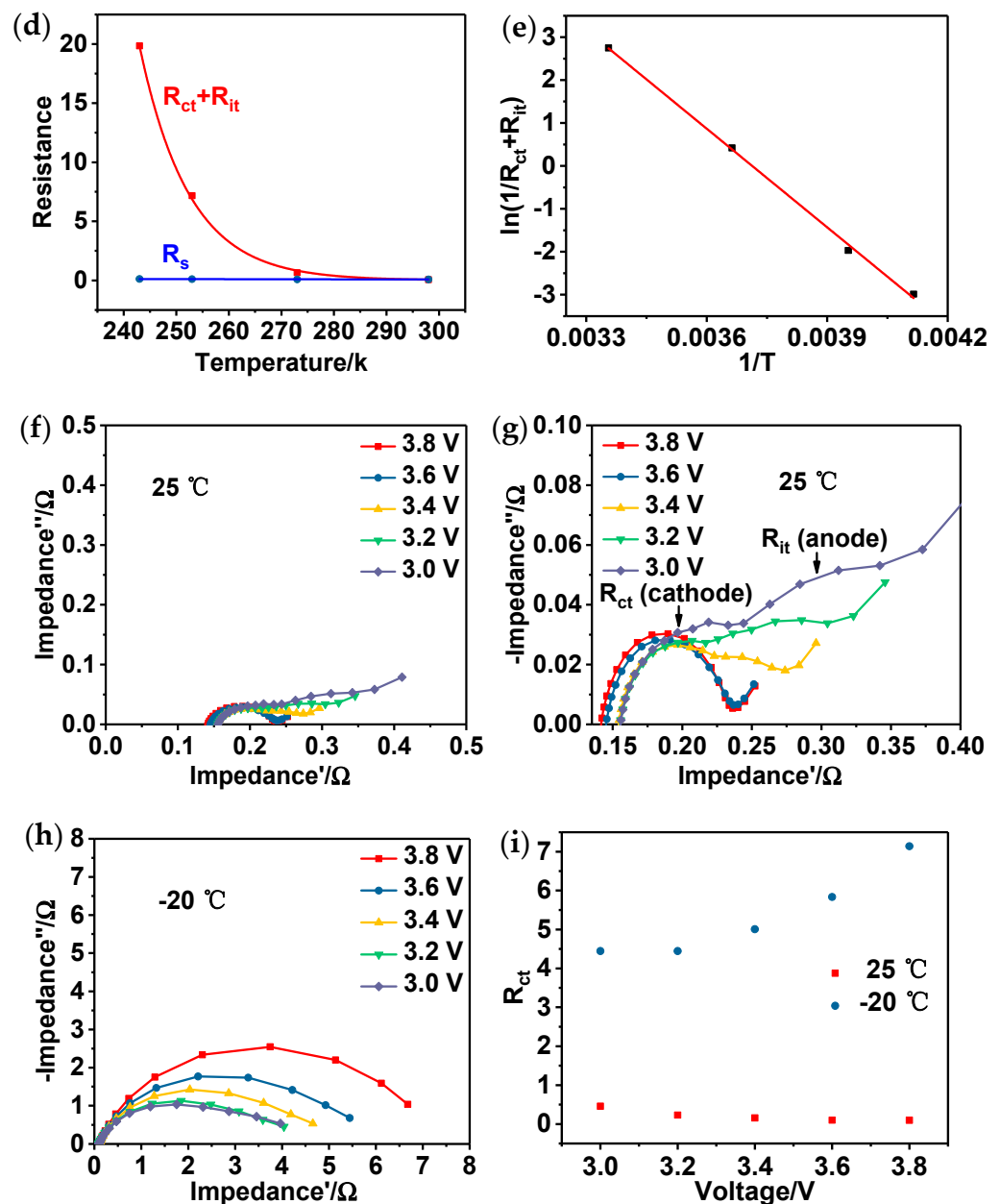


Figure 4. Electrochemical impedance spectroscopy (EIS) and equivalent circuit for normal device: (a) schematic diagram and equivalent circuit for normal device; (b,c) complex-plane plot of impedance at 25, 0, −20, and −30 °C; (d) the resistance-temperature plot; (e) the $\ln(1/R_{ct}) - 1/T$ plot; (f,g) complex-plane plot of impedance at 25 °C; (h) complex-plane plot of impedance at −20 °C; (i) the tendency of R_{ct} at different voltages.

To study the interface reaction between the cathode and the anode, we assembled a symmetrical device. The schematic diagram and equivalent circuit for symmetrical devices are shown in Figure 5a. The symmetrical device of the cathode is composed of the cathode/seperator-electrolyte/cathode. The symmetrical device of the anode is composed of the anode/seperator-electrolyte/anode. The impedance of the symmetrical device based on the cathode is shown in Figure 5b. The R_s and R_{ct} of the symmetrical device based on the cathode at 25 °C are 1.688 and 13.855 Ω , respectively. At −20 °C, the R_s and R_{ct} of the symmetrical device based on the cathode increased to 7.246 and 28.92 Ω , respectively. The impedance of the symmetrical device of the anode is shown in Figure 5c. The R_s and R_{it} of the symmetrical device based on the anode at 25 °C are 2.010 and 46.156 Ω , respectively. At

$-20\text{ }^{\circ}\text{C}$, the R_s and R_{ct} of the symmetrical device based on the anode increase to 16.327 and $105.67\text{ }\Omega$, respectively. The above results show that, compared with the resistance of the symmetrical device based on the anode at $25\text{ }^{\circ}\text{C}$, the values of R_s and R_{it} at $-20\text{ }^{\circ}\text{C}$ both obviously increase, which is related to the shrinkage of lattice spacing of the graphite (002) at $-20\text{ }^{\circ}\text{C}$. In addition, the values of R_{it} in the anode are larger than the values of R_{ct} in the cathode. This result shows that, when voltage of the battery reduces to 3.4, 3.2 and 3.0 V at $25\text{ }^{\circ}\text{C}$, the first semicircle in high frequency of the impedance curve is derived from R_{ct} in the cathode, and the latter semicircle at low frequency is derived from R_{it} in the anode.

Table 1. The impedance data of normal devices and symmetrical devices at different temperature and voltage.

Device Type	Temperature/ $^{\circ}\text{C}$	Voltage/V	R_s/Ω	$R_{ct} + R_{it}/\Omega$
Normal device	25	3.8	0.086	0.064
	0	3.8	0.090	0.658
	-20	3.8	0.105	7.174
	-30	3.8	0.128	19.848
	25	3.8	0.141	0.097
		3.6	0.145	0.100
		3.4	0.154	0.160
		3.2	0.156	0.231
		3.0	0.156	0.457
		-20	3.8	0.094
			3.6	0.093
			3.4	0.092
			3.2	0.092
			3.0	0.091
	Symmetrical cathode	25	0	1.688
		-20	0	7.246
Symmetrical anode	25	0	2.010	46.156 (R_{it})
	-20	0	16.327	105.67 (R_{it})

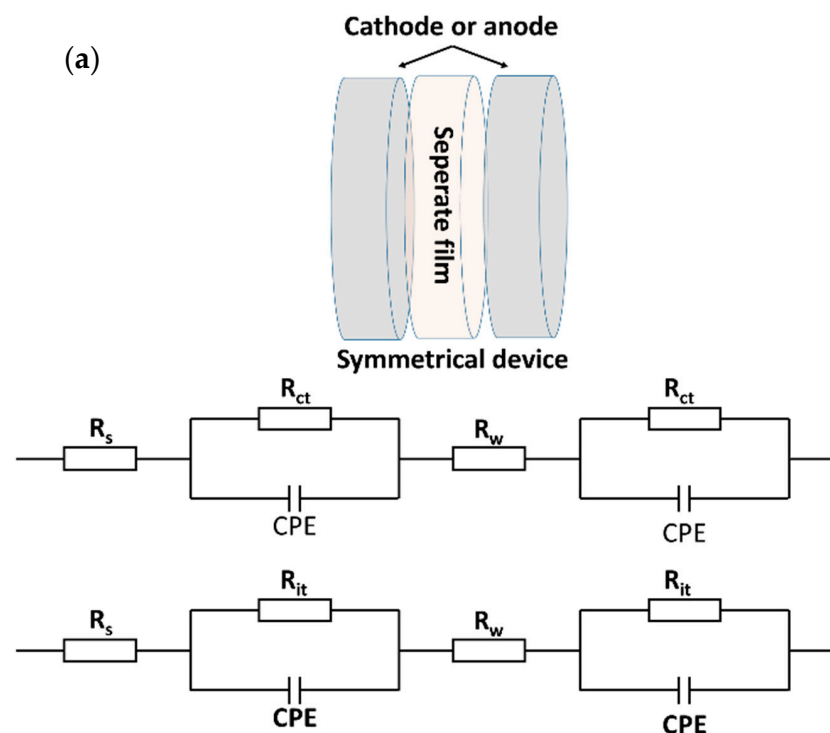


Figure 5. Cont.

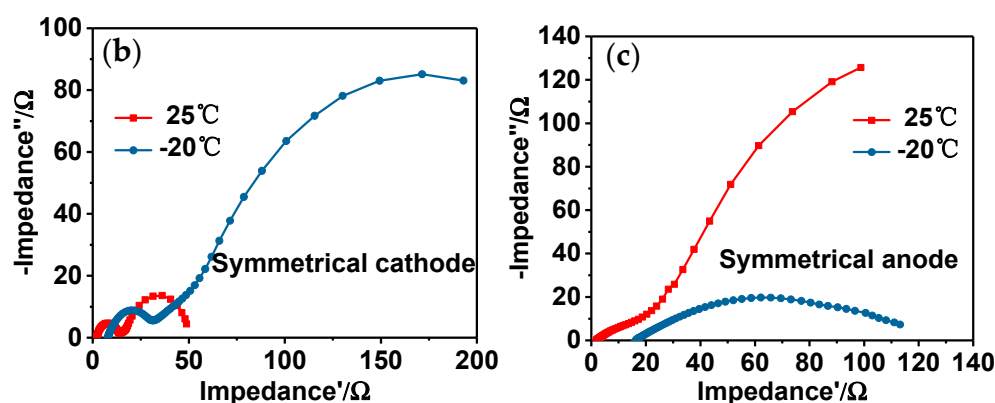


Figure 5. Electrochemical impedance spectroscopy (EIS) and equivalent circuit for symmetrical device: (a) schematic diagram and equivalent circuit for the symmetrical device; (b) complex-plane plot of impedance at 25 °C and −20 °C for the symmetrical device based on cathode; (c) complex-plane plot of impedance at 25 °C and −20 °C for the symmetrical device based on anode.

4. Conclusions

The performance of the normal device based on ternary $\text{Li}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ as cathode and graphite as anode at 0.2 C and −20 °C can reach 92.82% of that at 1 C and 25 °C. The value of E_a on the interface of the lithium ion battery electrode is 63.72 kJ/mol. When the temperature dropped from 25 to −20 °C, the lattice spacing of $\text{Li}_{1-x}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ hardly changed, while the lattice spacing (002) of graphite reduces. The results of quasi-in situ EIS indicate that, unlike the relationship of voltage–resistance at 25 °C, R_s , R_{ct} , and R_{it} tend to gradually decrease as the voltage decreases at −20 °C. Compared with the resistance of the symmetrical device based on the anode at 25 °C, the values of R_s and R_{it} at −20 °C both obviously increase. The main reason for performance degradation for a normal device at −20 °C is large ion transfer resistance and the decrease of lattice spacing of the graphite (002).

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/en15041410/s1>, Figure S1: The image of $\text{Li}(\text{Ni}_{0.5}\text{Mn}_{0.3}\text{Co}_{0.2})\text{O}_2$ in cathode film at 25 °C; Figure S2: The image of graphite in cathode film at 25 °C; Figure S3: The elements of the SEI film.

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