Short communication

A universal matching approach for high power-density and high cycling-stability lithium ion capacitor

Liming Jin [a,b,c], Xin Guo [a,b], Chao Shen [c], Nan Qin [a,b], Junsheng Zheng [a,b,*], Qiang Wu [c], Cunman Zhang [a,b], Jim P. Zheng [b,c,**]

[a] Clean Energy Automotive Engineering Center, Tongji University (Jiading Campus), 4800 Caoan Road, Shanghai, 201804, China
[b] School of Automotive Studies, Tongji University (Jiading Campus), 4800 Caoan Road, Shanghai, 201804, China
[c] Department of Electrical and Computer Engineering, Florida A&M University-Florida State University College of Engineering, FL, 32304, USA

HIGHLIGHTS

- A practical and universal matching approach is proposed.
- The approach for common lithium ion capacitor system is systemically demonstrated.
- The approach brings new insight for anode candidates of lithium ion capacitors.

ARTICLE INFO

ABSTRACT

Lithium ion capacitor is one of the most promising energy storage technologies with a higher power density and a longer cycling lifespan than Li-ion battery, but also a higher energy density than supercapacitor. However, the unbalanced electrochemical performance between the cathode and anode will inevitably result in unsatisfied power density and reduced cycling lifespan, which largely hinders its commercial applications. In order to eliminate the unbalanced kinetic behavior and cycling stability between the cathode and anode, in this study, we propose a practical and universal electrode matching approach for constructing high performance LIC systems. The effect of effective anode potential range on the performance of a typical LIC system was investigated. An optimal anode potential range is determined and then realized through pre-lithiation to pair with cathode, by which a much improved LIC with high energy density, large power density as well as long cycling lifespan is achieved. Based on the above findings, this approach brings some new insights on ideal anode material candidates for future LIC systems.

1. Introduction

Hybrid lithium ion supercapacitors, also called lithium ion capacitors (LICs), are considered as next generation capacitive energy storage devices [1,2]. Generally, LICs are composed of a capacitive cathode (e.g. activated carbon, AC) and a battery-type anode (e.g. graphite, hard carbon, HC), operating in a Li-containing organic electrolyte [3]. In comparison with traditional electrical double-layer capacitors (EDLCs), LICs have the potential to deliver higher energy density without sacrificing their power density and cycling lifespan, which results from the lower reaction potential and larger specific capacity of battery-type anode materials than those of capacitive anode materials of EDLCs [4,5].

However, the key limitations in achieving high-performance LICs are the imbalances in the kinetic behavior and cycling capability between the two electrodes, which stems from their distinct energy storage mechanisms (i.e. physical adsorption-desorption mechanism for capacitive cathode and chemical intercalation-deintercalation mechanism for battery-type anode) [6,7]. The unbalanced electrochemical properties would directly result in unsatisfied power density and reduced cycling lifespan. To solve this problem, a great deal of novel materials with desirable high-rate performance and/or cycling stability has been explored as anode for LICs, such as insertion materials (e.g. $Li_4Ti_5O_{12}$, TiO_2, Nb_2O_5) [8–13], conversion materials (e.g. Fe_3O_4, SnO_2) [14,15], and alloy materials (e.g. Si, Sn) [16–18]. Still, these efforts only mitigate

but do not eliminate the inherent differences between the two electrodes [10,19,20]. Therefore, it is still vital to develop effective approach to elimite the imbalanced electrochemical performance between the two electrodes.

Recently, pre-lithiation technologies are proposed to be widely applied into LICs by our group, including stabilized lithium metal power (SLMP), lithium stripe, and ultrathin lithium foil as lithium sources, which are capable to enhance the energy density of LICs substantially [21–25]. Meanwhile, the usage levels of lithium are flexible and tunable and the varying anodes with different potentials can be obtained by controlling the pre-lithiation degrees [21]. On the other hand, for LIC systems, the anode is usually constructed with much higher capacity than cathode for better power density and cycling lifespan. Moreover, the anode materials generally display various electrochemical properties at different potentials so that selecting optimal potential range to pair with cathode should be effective to eliminate the unbalanced electrochemical performance between the cathode and anode. Based on all above analysis, pre-lithiation technologies are capable to reasonably select a segment of effective operating potential ranges of anode to pair with cathode, which exhibits great potential to successfully improve the displayed electrochemical performance of anode materials and then power density and cycling lifespan of LICs.

Herein, we choose a most common LIC system (AC//HC) as case study to illustrate a universal matching approach for improved LICs with desirable power density and cycling lifespan. We firstly carefully evaluate the optimal working potential range of anode (HC) and certify the electrochemical performance differences of anode when operating in varied potential ranges. An optimal anode potential range with superior reaction kinetic behavior is identified, and then realized by pre-lithiation technologies, which can be used to well match with cathode for LICs with desirable power density and cycling lifespan. Next, we summarize the above finding and propose a matching approach to minimize the unbalanced kinetic behavior between cathode and anode. One highlight of this practical approach is that it can be universally applied into any other LIC systems. In addition, this approach also brings some new insights on anode materials selection for future LIC systems.

2. Result and discussion

2.1. Electrochemical evaluations of cathode and anode of LIC

Generally, the cathode and anode of LICs deliver distinct electrochemical performances due to their different energy storage mechanisms as mentioned above. In this context, AC and HC are most common cathode and anode materials of LICs, respectively, which are thus selected as research objects to evaluate the electrochemical performance differences [7,26]. The AC (YP–50F, Kurruray, Japan) with a high specific surface area of $1709 \, m^2 \, g^{-1}$ and abundant micropores structure (see Fig. S1) is thus capable to store the energy by ion adsorption-desorption mechanism at the potential of 2.0–4.0 V vs. Li/Li$^+$, displaying linear charge and discharge profiles in Fig. S2a. In contrast, the HC (carbotronP (J), Kureha, Japan) achieves energy storage by two mechanisms, i.e. ion insertion-desertion into the layers of carbon, and ion adsorption-desorption on edges of defects at the potential of 0–3.0 V vs. Li/Li$^+$ without any discharge plateau (see Fig. S2b). The detailed electrochemical performances including multi-rate performance and cycling stability are shown in Fig. S3. It needs to be noted that AC and HC in this study deliver inferior electrochemical performance in comparison with the previous literatures due to their extremely high loading and high tap density, e.g. $8.0 \, mg \, cm^{-2}$ for AC and $9.0 \, mg \, cm^{-2}$ for HC, which are practical in applications [27–30]. In brief, the AC electrode displays a low specific capacity of $53.8 \, mAh \, g^{-1}$ at the low current density but excellent high rate (>75% capacity retention at $2.0 \, A \, g^{-1}$) and excellent cycling stability (>70% capacity retention after 3000 cycles at $0.5 \, A \, g^{-1}$), while the HC delivers a high specific capacity of $220 \, mAh \, g^{-1}$ at $0.1 \, A \, g^{-1}$ but poor high rate and cycling stability as shown in

Figs. S3c–3d. These results strongly demonstrate two electrodes of LIC deliver unbalanced electrochemical performance (Fig. 1a), which are mainly attributed to their inherent energy storage mechanisms. The inferior high rate and cycling stability of anode will inevitably result in poor power density and reduced cycling lifespan when fabricating full cells.

2.2. The optimal anode working potential range evaluations

Previous reports proposed to establish a capacity-unbalanced structure to charge the anode at a shallow potential range, which is capable to largely minimize the properties difference of cathode and anode (see Fig. 1b), i.e. the capacity of anode is several times higher than that of cathode in LIC systems [31]. Under this circumstance, there are various choices to match the cathode and anode following their potential profiles (from a-a' to b-b') due to the excess capacity of anode. Meanwhile, the anode at different potential ranges usually delivers distinct electrochemical performance due to the specific electrochemical process. Therefore, selecting the optimal fragment or range of anode potential profiles to match with AC potential profiles should be beneficial to fabricate the LIC systems with high power density and long cycling lifespan. In theory, the optimal working potential range, which can be systemically evaluated and determined by electrochemical impedance spectroscroscopy (EIS) [6,7,26], galvanostatic intermittent titration technique (GITT) [32], potentistatic intermittent titration technique (PITT) [32], and galvanostatic charge and discharge (GCD) tests [6,7,26, 33,34], etc. are capable to deliver superior reaction kinetic behavior and cycling stability.

In this context, to determine the optimal fragment or range of HC potential profile, firstly, the lithium ion storage mechanism for HC is discussed. During the discharge process from 3.0 V to 0.01 V, HC undergoes two main energy storage mechanisms: (*I*) lithium ion adsorption-desorption processes on the defect site at the edge of graphite layer; (*ii*) lithium ion intercalation-deintercalation processes in the carbon layers. The adsorption-desorption processes with relatively low capacity occurring at the high potential over 1.0 V are not suited to be utilized. In comparison, intercalation-deintercalation processes with over 90% capacity at low potential displays great potential for pairing with AC [35,36]. Combining the electrochemical processes and potentials, the discharge profiles can be divided roughly into four regions as shown in Fig. 1c. In Region I, the HC stays an original state, which charges with an adsorption-desorption mechanism at the defects of HC. This process with relatively high potential range is not suitable to pair with AC cathode. Ragone II exhibits an ion intercalation-deintercalation mechanism where the average carbon layer spacing is relatively small, so that the HC exhibits poor reaction kinetic. In Region IV, the HC also displays slow kinetic behavior and even poor stability, which are due to the facts that (*I*) the lithium ions occupy most of the carbon layer with limited space to bear more; (*II*) the fully intercalated carbon layer with large materials strain leads to poor cycling stability. Moreover, the over-low reaction potential will possibly lead to lithium dendrite growing and thermal runaway in LIC devices (see Fig. 1b). Alternatively, the HC in Region III with a relatively large carbon layer spacing, abundant free lithium ion sites, and low material strain delivers both desirable high rate performance and cycling stability, exhibiting as the optimal potential range to pair with AC.

As mentioned above, the potential range above 1.0 V is too high and of low capacity to be utilized to pair with AC, so the reaction kinetic behavior at the potential of 0.01 V–1.0 V was selected to be illustrated carefully. we carried out a quasi-in-operando EIS technologies in a single test cell to survey the impedance of HC, which is capable to not only evaluate the reaction kinetic behavior changes by the total resistances but also quantify resistance changes by equivalent circuit fitting to correspond with the electrochemical process as mentioned above. As shown in Fig. 2a–c, the EIS is consisted of two semicircles and a inclined straight line, which correspond to solid electrolyte interface (SEI)
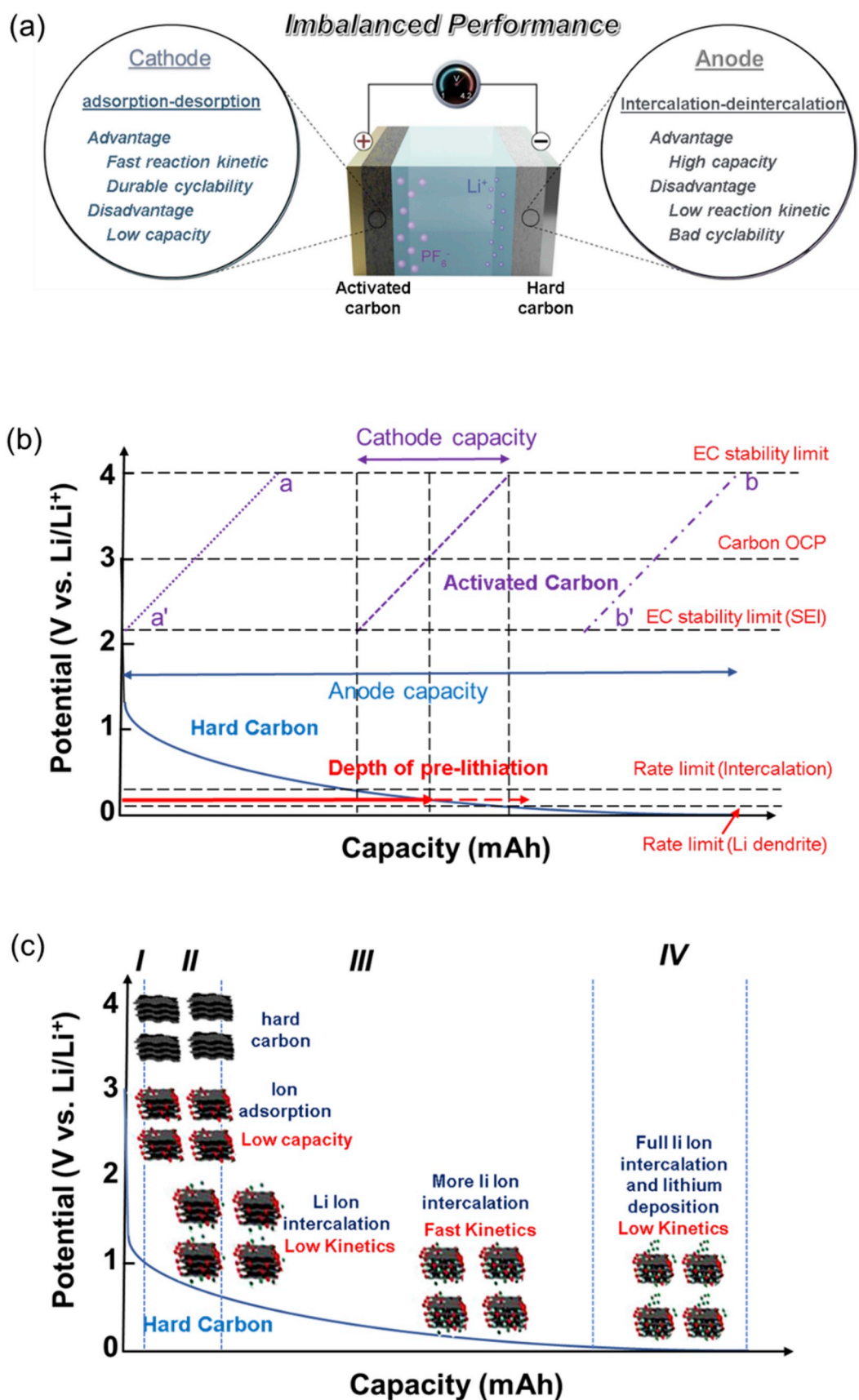
Fig. 1. (a) the scheme image of LIC and unbalanced performance; (b) the potential profiles matching of electrodes for LIC; (c) the reaction mechanism scheme image of HC anode.

resistance (R_{SEI}), charge transfer resistance (R_{ct}), and ion diffusion resistance (R_i), respectively [6,7,26]. The corresponding quantitative resistances are obtained from the equivalent circuit fitting as shown in the insertion of Fig. 2d. In comparison with other types of resistances, the R_o is extremely small so that it can be ignored. When the potential reduces from 1.0 V to 0.1 V, the R_{SEI} essentially keeps constant and the R_{ct} decreases gradually, respectively. When the potential further reduced to ultralow 0.01 V, the R_{SEI} and R_{ct} increase suddenly and simultaneously. These results are fully consistent with the reaction principles as shown in Fig. 1c. Obviously, the total resistance displays a similar variation tendency with R_{ct} as shown in Fig. 2d. The total resistance decreases gradually with the potential reducing from 1.0 V to 0.1 V indicating the enhanced reaction kinetic behavior. However, the impedance increases when the potential lower to 0.01V due to the increased R_{SEI} and R_{ct}.Therefore, by this comprehensive EIS analysis combining discharge mechanism of electrode materials, an optimal charge-discharge potential ranges from 0.1 V to 0.7 V can be determined.

To further confirm this optimal charge-discharge potential range, a series of electrochemical evaluations are carried out, including multi-rate performance and long cycling performance. Fig. 3a displays the multi-rate performance of HC within various potential windows from 0.01–1.0 V, 0.01–0.7 V, 0.1–0.7 V, and 0.1–1.0 V, respectively. When the HC is charged within 0.1–0.7 V, the test cell delivers the best high-rate performance with a capacity retention of >25% at 1.0 A g^{-1}, much better than the cells which are tested within other potential ranges. Meanwhile, the test cells within the potential range of 0.1–0.7 V exhibit much better cycling stability with the highest capacity retention after 1000 cycles at 0.5A g^{-1}. The low potential (<0.1V) exhibits significant negative effect on both high-rate performance and cycling stability. Due to the excellent cycling stability of AC in AC//HC full cell, the HC in the full cell can be considered to deliver a constant capacity in each cycle. Therefore, a constant capacity test in half cell configuration is also utilized to evaluate the cycling stability of HC within different cut-off potentials at the current density of 0.5 A g^{-1}. Fig. 2c and Fig. S4 display the potential profiles of HC with the constant capacity of 30 mAh

g^{-1} when the low cut-off potential of 0.01 V, 0.1 V and high cut-off potential of 1.0 V was applied, respectively. It can be observed in the inserts of Fig. 2c and Fig. S4 that voltage ranges extend obviously with the increasing cycles, indicating the performance degradations of HC. On the contrary, the cell with a low cut-off potential of 0.1 V only shows a slight potential extension from 0.310–0.672 V to 0.340–0.690 V after 1000 cycles, revealing excellent cycling stability. These results all demonstrate 0.1–0.7 V as a preferable potential range for HC with desirable high-rate performance and cycling stability to match with AC.

2.3. The electrodes matching achieved by pre-lithiation technologies

After evaluating the optimal working potential range of anode, the next step is to precisely match the electrodes. The anode with desirable pre-lithiation degree is capable to operate within the optimal working potential range when pairing with cathode as shown in Fig. 1b. The effect of the pre-lithiation level can be demonstrated by comparing various full cells at different electrodes matching states, in which the anode operates at different operating potential ranges.

In this context, firstly, we doped various amount of SLMP within 0 wt %~20 wt% of HC onto the HC anode and the pre-doped HC electrodes were assembled in a half cell and rested for 96 h. The relationship between the potentials of HC after pre-lithiation and the SLMP contents have been studied as shown in Fig. 4a. Then, we selectively pre-treated the HC under the condition of m$_{SLMP}$: m$_{HC}$ at 5%, 10%, 15% and 20%, in which the HC exhibits different electrochemical processes as illustrated in Fig. 1c. To clearly demonstrate the effect of selected potential ranges on the performance of AC//HC full cell, the tested full cells are assembled using AC and HC with same settled empirical weight ratio of 1:1 and tested with same empirical narrow voltage window of 2.2–3.8 V, in which the cathode and anode both operate in safe potential range and the anode property is the key factor to the performance of fabricated AC//HC full cells.

Fig. 4b–d displays the multi-rate performance with the current density from 0.2–1.0 A g^{-1} based on the weight of HC and the cycling performance of 5000 cycles of AC//HC systems, respectively. As
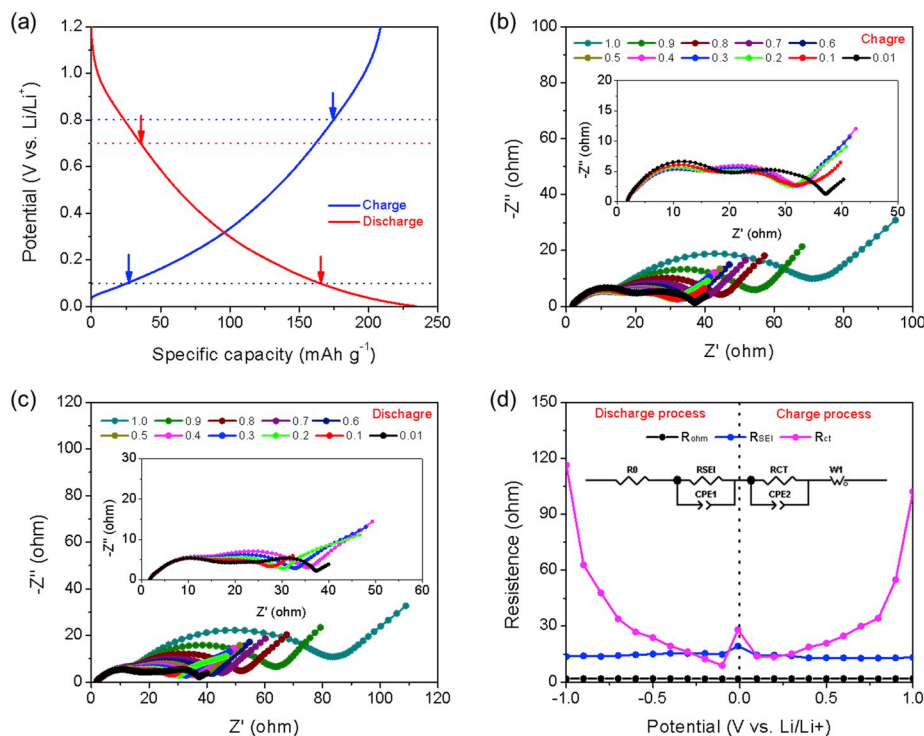

Fig. 2. (a) The charge and discharge profiles of HC and optimal working potential range; (b–c) The EIS results of HC at various potentials during charge and discharge processes (inserts are the enlarge curves at 0.01–0.4V); (d) The corresponding resistance for R$_{ohm}$, R$_{SEI}$ and R$_{ct}$ at various potentials.
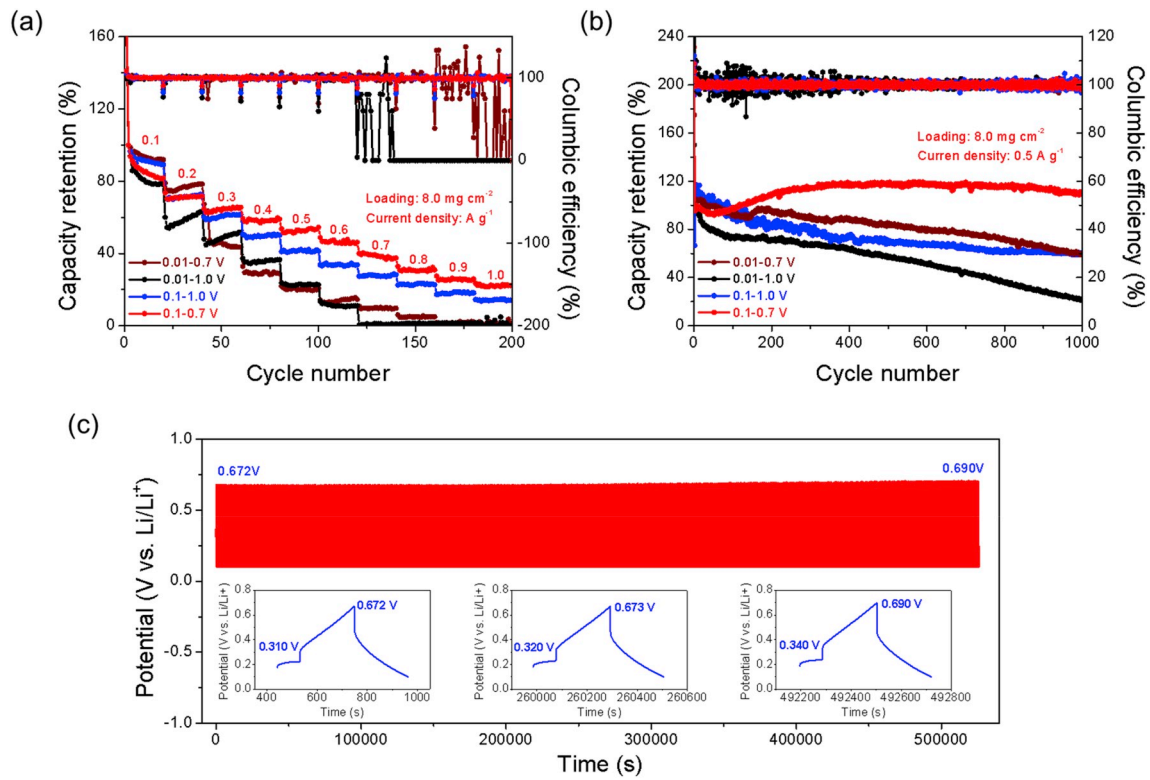
Fig. 3. (a) The multi-rate performance comparison and (b) cycling stability comparison of HC at various potential ranges; (c) The charge and discharge profiles of constant capacity cycling with a cut-off potential at 0.1V.
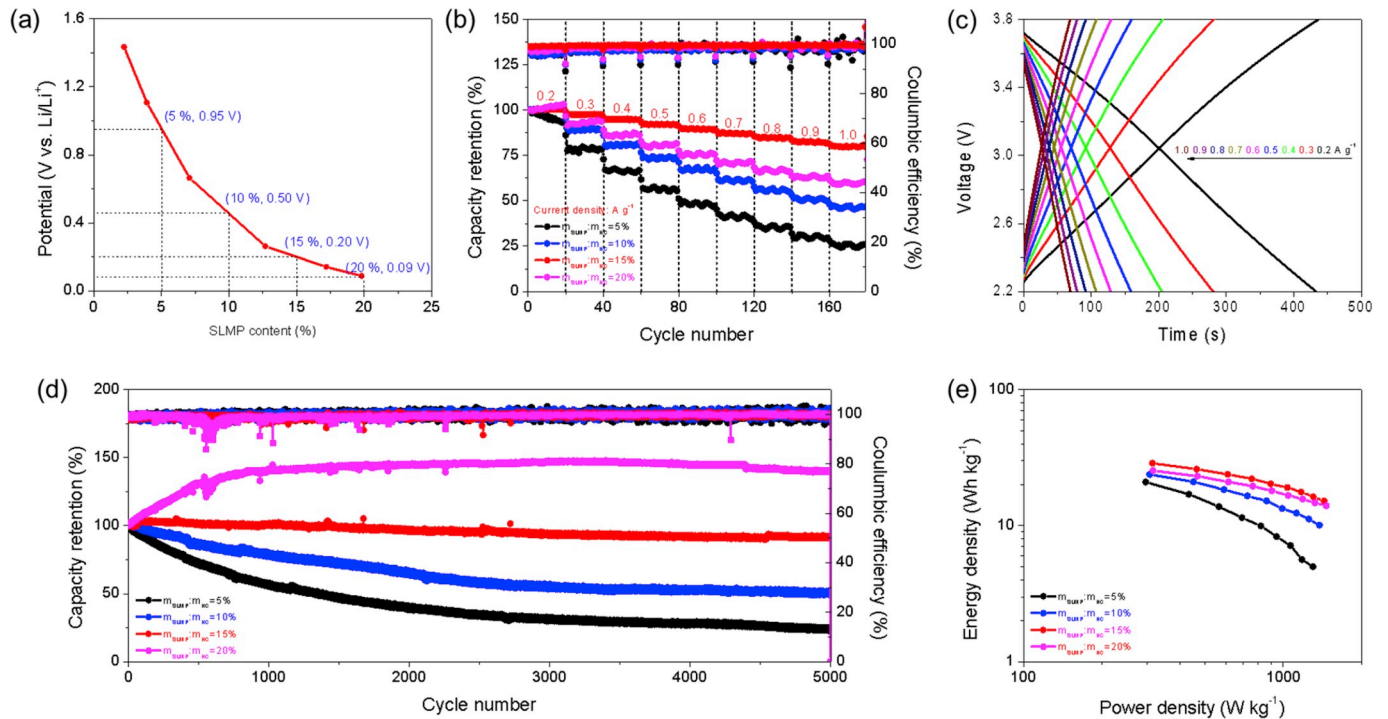

Fig. 4. (a) The relationship between potential and pre-lithiation degree; (b) multi-rate performance comparisons; (c) Charge and discharge profiles of cells with 15% pre-lithiation degree; (d) Cycling stability comparisons; (d) Ragone-plot comparisons.

expectedly, the cell with pre-lithiation degree of 15% displays the best multi-rate performance with a capacity retention of over 80% at 1.0 A g^{-1}. The voltage profiles (Fig. 4c and Fig. S5) suggest the cell with 15% pre-lithiation degree delivers the lowest IR-drop indicating best

reaction kinetic behaviors, which is consistent with the result in half cell studies. Moreover, the cell with 15% pre-lithiation degree exhibits much better cycling stability of over 95% capacity retention after 5000 cycles. In comparison, the cells only obtained 55% capacity retention for 10%

pre-lithiation degree and 25% capacity retention for 5% pre-lithiation degree, respectively. It is somewhat surprising that the cells with 20% pre-lithiation degree deliver a continuously increased capacity, which is probably due to the degradation of HC at range IV at the first 1000 cycles and extension of operating potential of HC from range IV to range III in the following cycles (see Fig. 1c). And the superior behavior in range III of HC enables the tested cells to display excellent cyclability after 1000 cycles. The Ragone-plot graph has been obtained as shown in Fig. 4e, which suggests the AC//HC with 15% pre-lithiation degree shows a much improved electrochemical performance in comparison with other cells. These results above all demonstrate selecting optimal operating potential range achieved by pre-lithiation technologies to pair with cathode is effective to improve the electrochemical performance of LIC devices.

2.4. A universal matching approach by pre-lithiation technologies

Overviewing the entire processes as demonstrated above, this approach by selecting optimal operating potential range of anode to match with cathode is effective, which should be also suitable to be applied into any other LIC systems. In general, the universal matching approach for all LIC systems can be mainly divided into three steps as shown in Scheme 1.

(I) Electrochemical performance evaluations of anode materials to detriment the optimal anode potential range, i.e. P_L (low potential limit) to P_H (high potential limit) and corresponding effective anode capacity, which could be carried out by EIS, GITT, PITT, GCD, etc.;

(II) Realization of designed anode potential by pre-lithiation technologies to the desired depth of pre-lithiation. The charge and discharge profiles of cathode can be divided into parts theoretically, including the PF_6^- and Li^+ adsorption-desorption processes. The latter one is achieved by pre-lithiation of anode and the potential profile should be lower than open potential. Therefore, the desired depth of pre-lithiation (point P_{pre-Li} in Scheme 1) corresponds to the position where the cathode operates at the open circuit potential (e.g. 3.0 V for AC);

(III) Electrode matching with desirable electrodes weight ratio, and working voltage, where the electrode weight ratio can be determined bas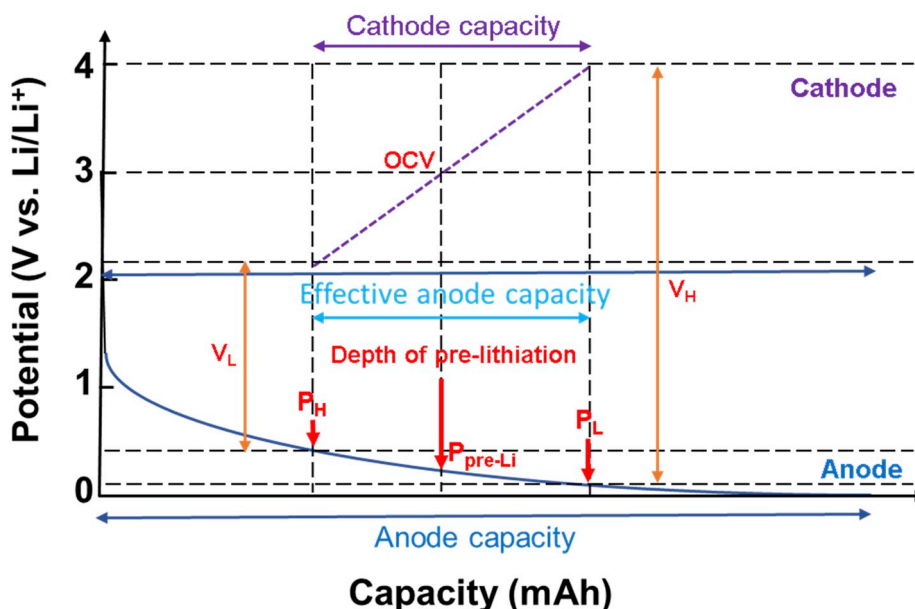ed on effective specific capacities of cathode and anode, and the working voltage window (V_L to V_H) is determined by potential profiles of both cathode and anode during cell operation.

Therefore, all the parameters by this matching approach could be collected directly based on the optimal potential profiles of cathode and anode in different LIC systems. In comparison with previous matching approaches to determine the pre-lithiation degrees, electrodes weight ratios, and working potentials by complex parallel experiments, respectively [37–41], this approach enables a convenient and practical "one-step" tuning of multiple matching parameters.

On the other hand, during the past years, the researchers also struggled to search for ideal anode candidate for anode with desirable potential, reaction kinetic behavior, as well as cycling stability. However, there is no anode candidate with above properties simultaneously. From this approach, we get an insight that in practice indeed only a fragment of potential profile of anode is utilized into full cell fabrications. Therefore, different from the case for ideal anode candidates for rechargeable Li-ion batteries, some anode materials with excellent reaction kinetic behavior and cycling stability at only a certain potential range but not the full potential range are also desirable anode candidates for LICs.

3. Conclusion

In conclusion, based on our systematical investigation on a typical AC//HC LIC system, we have proposed and systemically illustrated an effective and practical electrodes matching approach for LICs with improved energy density, power density as well as cycling lifespan, which is achieved based on the rational selection of anode operating potential range and pre-lithiation technologies. One highlight of this practical approach is that it can be universally applied into any LIC systems. The universal matching approach for all LIC systems can be mainly divided three steps. (*I*) electrochemical performance evaluations of anode materials to detriment the optimal anode potential range; (*ii*) realization of designed anode potential by pre-lithiation technologies; (*iii*) electrode matching with desirable pre-lithiation degree, electrodes weight ratio, and working voltage. In addition, this approach brings some new insights on anode materials selection for future LIC systems.

Scheme 1. The scheme graph of a practical and universal approach for electrode matching for LICs.

Acknowledgements

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jpowsour.2019.227211.

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