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ELECTROCHEMICAL SCIENCE AND TECHNOLOGY

Li Metal-Free Rechargeable LiMn_2O_4 /Carbon Cells: Their Understanding and Optimization

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ABSTRACT

So-called "rocking-chair" rechargeable batteries that use lithium intercalation compounds for the positive and negative electrodes should be safer than batteries that contain free-lithium metal. Such a cell, using the spinel LiMn_2O_4 as the positive electrode and carbon as the negative electrode, was optimized as a function of various operating parameters. These cells reversibly insert 0.32 Li atoms per Mn at an average output voltage of 3.7 V, yielding an effective specific energy of 250 mWh/g of electrode materials (3 times that of Ni-Cd). They can sustain high current rates similar to Ni-Cd batteries, and can be discharged to 0 V without any degradation of their operating conditions. By systematically studying the stability of several electrolyte systems, we were able to minimize electrolyte decomposition (by controlling drastically the charge cut-off voltage) so that these cells show a promising cycle life even at 55°C while maintaining 75% of their initial capacity.

It is now recognized that ambient temperature non-aqueous secondary lithium cells using lithium metal exhibit problems which make it difficult for their wide usage in the consumer market. These problems are: the short cycle life for deeply discharged cells, the high cost of the technology and, above all, the unsafe operating characteristics due to the high reactivity of lithium metal (1). To alleviate this problem, a "rocking-chair" battery that uses another intercalation compound as the anode has been proposed (2-8). Among the alternative materials that could replace lithium metal as the negative electrode, carbon provides the best compromise between large specific capacity and reversible cycling behavior (9-11). However, a price is paid in terms of average output voltage and energy density as compared to a lithium metal cell, and this is the reason why a strongly oxidizing intercalation compound must be used as the positive electrode. The layered compounds LiNiO_2 and LiCoO_2 and the three-dimensional compound LiMn_2O_4 with the spinel structure, are the only phases presently known to intercalate Li reversibly at voltage larger than 3.5 V vs. Li.

Recently, two battery companies, Moli Energy Ltd. and Sony Energytec Inc. have announced the future commercialization of Li^+ ion rechargeable batteries using a carbon intercalation compound as the negative electrode, and LiNiO_2 and LiCoO_2 or $\text{LiNi}_{0.2}\text{Co}_{0.8}\text{O}_2$ materials as the positive electrodes, respectively (12-14). We also have recently demonstrated the feasibility of such rocking-chair cells using the spinel LiMn_2O_4 and its lithiated parent compound $\text{Li}_2\text{Mn}_2\text{O}_4$ as the positive electrode at 25°C (15).

These rocking-chair batteries do not require a stringent manufacturing environment because the starting electrode materials (i.e. the lithiated manganese oxide and the carbon) are stable in ambient atmosphere. The cell is assembled in its discharged state, where the output voltage is close to 0 V, and, consequently, can be handled before use without any fear of irreversible degradation due to a short circuit. The battery is activated during the first charge. This is similar to the well-known and widely used Ni-Cd batteries that need to be charged prior to their first use.

The use of the spinel manganese oxide material offers the following advantages: LiMn_2O_4 is easy to prepare, compared to the several steps required for the synthesis of LiNiO_2 , and Mn is more abundant and cheaper than Co, so

that the overall electrode cost would be minimized if LiMn_2O_4 is used; The theoretical energy density of LiNiO_2 and LiCoO_2 is twice as high as that of LiMn_2O_4 , but in practice only half of the Li content can be removed, thus leading to the same order of usable energy density for the three materials. Moreover, the energy density of spinel-based manganese oxide cells can be increased if $\text{Li}_2\text{Mn}_2\text{O}_4$ is used (15). The ability to synthesize LiMn_2O_4 at temperatures as low as 400°C (lower than the temperature required to obtain LiNiO_2 and LiCoO_2) also makes this oxide an attractive material for microbatteries compatible with semiconductor technologies (e.g., self-powered electronics). Finally, most of the battery community is already familiar with manganese-based oxides since most of the commercial primary batteries (MnO_2/Li or MnO_2/Zn) use a manganese-based oxide as the positive electrode so that toxicity (if any) or recycling problems are already well known.

The use of a strongly oxidizing compound such as LiMn_2O_4 as the positive electrode is worrisome since the delithiation of this compound occurs at a very high voltage (about 4.1 V vs. Li/Li^+) where the electrolyte oxidation can occur (16). Moreover, such an electrochemical reaction would be accelerated upon increasing the temperature. We have systematically studied the electrolyte oxidation as a function of temperature and the liquid electrolyte used. We succeeded in minimizing the electrolyte decomposition and we show that the LiMn_2O_4 /carbon rechargeable cells can operate at 55°C with good reversibility and without measurable electrolyte oxidation.

This paper details the electrochemical and cycling behavior of the LiMn_2O_4 spinel manganese oxide/carbon rocking-chair cells as a function of various parameters: The ratio of positive and negative electrode masses; The electrode thicknesses; The charge and discharge cut-off voltages; The current and; The operating temperature. We also report the figure-of-merit of these cells as compared to the widely used Ni-Cd battery and to the recently announced Li^+ ion rechargeable battery (13). The electrochemical behavior of each individual LiMn_2O_4 and carbon electrode vs. Li in two different electrolytes will be presented first and separately, with emphasis on the influence of the operating temperature and the voltage limits for cycling. Along with this study, we also have determined the chemical diffusion of Li^+ in the spinel LiMn_2O_4 and in petroleum coke.

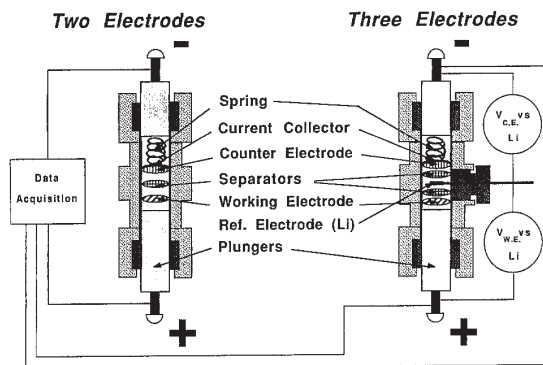


Fig. 1. The drawing of 2 and 3 electrodes Swagelock cells are shown in (a) and (b), respectively.

Experimental

The battery cycling studies presented herein have been made using standard and three electrode Swagelock cells shown in Fig. 1. The plungers were made out of a special stainless steel (Carpenter, Ref. 20Cb-3), that allows oxidative voltages as large as 4.7 V vs. Li without any leakage current due to the electrolyte oxidation. In such cells, the positive electrode (LiMn_2O_4) is in direct contact with the plunger, while the negative electrode (Li or carbon) is in contact with a half-inch diameter nickel disk. Both electrodes are separated by a 0.3 mm thick porous glass paper soaked in the electrolyte selected for the study. To ensure physical contacts between the cell components, a stainless steel spring that allows pressures of about 1 kg/cm² is placed between the nickel disk and the second plunger. In the three electrode design, the reference electrode, a Li foil plated onto a nickel wire sandwiched between two glass paper disks is placed between the positive and negative electrode pellets. These three electrode cells allow the simultaneous *in situ* determination of the three characteristics, $V_{\text{positive vs. Li}}$, $V_{\text{negative vs. Li}}$, and $V_{\text{positive vs. V}_{\text{negative vs. Li}}}$, providing us the information necessary to fully understand, and optimize, the behavior of these secondary cells. For the cyclovoltammetry measurements, a three electrode glass cell, consisting of a working electrode, a reference electrode (piece of lithium foil plated onto a nickel wire), and a counterelectrode (made the same way as the reference electrode) was used. The working electrode was either a glassy carbon electrode (to study the oxidation limit of some electrolytes) or a pellet of tested material compacted onto a platinum gauze (for the electrochemical characterization of this material). Both the Swagelock cells and the three electrode glass cell were always assembled in a helium atmosphere. Then, the air-tight Swagelock cells were removed from the dry box to be tested. No loss of electrolyte was detected for these cells even after a series of tests at 55°C during 1 month.

LiMn_2O_4 materials were prepared by reacting in air stoichiometric amounts of Li_2CO_3 and MnO_2 placed into an alumina crucible. In accordance with our previous work (17), because of their larger capacity, LiMn_2O_4 powders (1 to 2 μm particle size) prepared by three consecutive anneals at 800°C (24 h each) were used as the positive electrodes. Petroleum coke (Conoco, -200/400 mesh; i.e. particle size between 38 and 75 μm) was used instead of graphite as the negative electrode, because of a lower loss of capacity compared to graphite at the first cycle, although its capacity is twice as small (its maximum Li composition has been reported to be $\text{Li}_{0.5}\text{C}_6$ instead of LiC_6 (18, 19)). Composite electrode materials containing the active material, carbon black, and EPDM (ethylene propylene diene monomer) rubber were made by mixing the powders in a solution of EPDM in cyclohexane, evaporating the solvent, sieving the resulting powders to 100 μm and pressing them at a pressure of 10 tons/cm² into pellets. The resulting composite material will be denoted by three numbers [a, b, c] where a, b, and c will refer to the weight ratio of the active material, carbon black, and EPDM, re-

spectively, (for example a composite electrode containing 90% LiMn_2O_4 , 8% carbon black, and 2% EPDM will be denoted as [90, 8, 2]). A survey of several compositions was done for both composite electrodes in order to optimize the cyclability performance of the composite electrodes with respect to Li. The results of such a survey show that the compositions [89, 10, 1] and [94, 5, 1] give the best reversibility vs. Li for the positive and negative electrodes, respectively, as well as the lowest polarization (minimum difference in voltage between charge and discharge curves). These compositions were used in all the results reported here. Although thermogravimetric analysis measurements (TGA) of the composite electrodes did not reveal any trace of water, all the electrodes were heated at 150°C during 1 h, prior to being used as electrodes in rocking-chair batteries to eliminate any possibility of water contamination. For all the cells using LiMn_2O_4 as the positive electrode, we took the Li content $x = 1$ as the initial stoichiometry, and then the composition was automatically determined from the current, the mass of the active cathode material used, and the elapsed time. From the values of x , the equivalent capacity in mAh/g reported in several figures is simply obtained by multiplying the theoretical capacity 148.2 mAh/g by Δx .

Propylene carbonate (PC), with its low vapor pressure and high flash point, was used for the solvent. To increase the ionic conductivity of an electrolyte without changing the nature and content of the salt, mixtures of solvents are generally used. For instance, high dielectric constant solvents like ethylene carbonate (EC) or PC are mixed with low dielectric constant and low viscosity solvents (like 2-methyl-tetrahydrofuran (2Me-THF) or dimethoxyethane (DME)) (20). In this study, diethoxyethane (DEE) was selected instead of the commonly used 2Me-THF or DME, because its boiling point is higher (121°C compared to 80°C), thereby limiting safety hazards (e.g., explosions) and the solvent mixture consisting of EC and DEE, 50-50 by weight, was tried. The solvents (Aldrich) were dried using standard methods. For EC and PC, the solvents were degassed by 3 pump/refill cycles using Ar as the inert gas. EC was warmed to above the melting point. The solvents were dried over 4 Å molecular sieves for 48 h and then were distilled under reduced pressure (15 Torr). DEE was degassed by 3 freeze-pump-thaw cycles, and was distilled under Ar from K benzophenone ketyl.

Different electrolytes have been made by adding various widely used Li salts (heat-vacuum dried prior to their use), such as LiAsF_6 , LiCF_3SO_3 , LiBF_4 , LiPF_6 , $\text{LiN}(\text{CF}_3\text{SO}_2)_2$, and LiClO_4 to PC or EC + DEE (50:50). After the drying process, the x-ray powder diffractograms were controlled to exclude any possibility of degradation. The best Li-salt, defined in this study as the one allowing the largest electroactivity range at high voltage, was selected as follows. First, the oxidation limit of the electroactivity range of these electrolytes has been determined by cyclic voltammetry on a glassy carbon electrode. The oxidation current of the electrolytes is located in the range 4.6 to 5.0 V vs. Li, showing that all these salts could be used up to a voltage of 4.5 V vs. Li in any battery. This measurement, however, is not fully indicative since it does not reflect the true operating conditions because, the catalytic activity of any positive electrode material could be different from that of the glassy carbon, and the standard scan rate used for metal electrodes (0.1 V/s) is much higher than that used for cycling an intercalation electrode material (10^{-4} to 10^{-5} V/s). Thus, these different electrolytes have been tested under real operating conditions in LiMn_2O_4 /electrolyte/Li cells up to 4.5 V and the salt allowing for largest charge cut-off voltage on the LiMn_2O_4 material, over a large number of cycles without any oxidation of the electrolyte, is LiClO_4 . Other salts can also be used with LiMn_2O_4 , but they allow a smaller electroactivity range. Consequently, from this study, the two electrolytes PC + 1M LiClO_4 and EC + DEE (50:50) + 1M LiClO_4 were selected and Fig. 2 shows their oxidation limit measured by cyclic voltammetry at the two temperatures used in this work. The PC-based electrolyte allows higher voltage values, but for both electrolytes the oxidation limit is lowered by 0.2 V when the operating temperature is increased to 55°C.

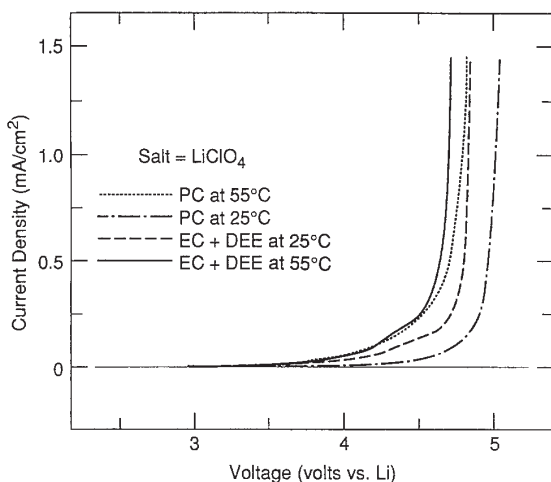


Fig. 2. Cyclic voltammograms at glassy carbon electrode of EC + DEE (50:50) + 1M LiClO₄ and PC + 1M LiClO₄ electrolytes at 25 and 55°C. Scan rate = 0.1 V/s.

The ionic conductivity and the water content for the selected electrolytes used for our experiments were determined using a high frequency impedance analyzer and a Karl-Fisher coulometer, respectively. Ionic conductivity values of 10.4 and 5.62 mS/cm at room temperature (25°C) and water contents of 45 and 40 ppm were obtained, for the two electrolytes EC + DEE (50:50) + 1M LiClO₄, and PC + 1M LiClO₄, respectively.

The chemical diffusion coefficients of Li⁺ ions in both LiMn₂O₄ and carbon electrodes were measured using the potentiostatic intermittent titration technique (PITT) (21, 22) by means of a special multichannel galvanostatic/potentiostatic system "Mac-Pile." In this system the potentiostatic mode acts as a coulometer so that the experimental parameter is the charge increment *vs.* time and not the current *vs.* time as usually measured. Thus, in our experiment, the potential of the material/Li cell is monitored by steps, and the decay of the charge increment (the integral of the current) during each step is measured as a function of time. The resolution of Fick's second law for times $t \ll l^2/D$ and $t \gg l^2/D$ leads to the Eq. [1] and [2], respectively, for the variation of the charge increment as a function of time

$$q(t) = \frac{2FSD^{1/2}\Delta C}{\pi^{1/2}} \sqrt{t} \quad [1]$$

$$q(t) = \frac{8FSl\Delta C}{\pi^2} \exp\left(-\frac{\pi^2 Dt}{4l^2}\right) \quad [2]$$

where *t* is the elapsed time from the beginning of the step, *F* is the Faraday constant, *S* is the total surface area of the grains of the active material, ΔC is the variation of Li concentration into the material during the voltage step, *D* is the chemical diffusion coefficient of Li⁺ into the material, and *l* is the size of the material grains. The chemical diffusion coefficient *D* of Li⁺ can then be determined by the slope of the linear plot of *q vs.* \sqrt{t} in the short time approximation, and by the slope of the linear plot of $\log q$ *vs.* *t* in the long time approximation. The accuracy of this technique directly depends on the evaluation of the total surface area. In this work, we have estimated this surface area by assuming that the grains are spherical with a diameter equal to the average particle size.

Results and Discussion

Behavior of LiMn₂O₄ *vs.* Li.—Figure 3a shows the room temperature cycling behavior of a LiMn₂O₄/Li cell under constant current in the two electrolytes, EC + DEE (50:50) + 1M LiClO₄ and PC + 1M LiClO₄. The cell is first charged from its rest voltage (close to 3.3 V *vs.* Li) to 4.5 V

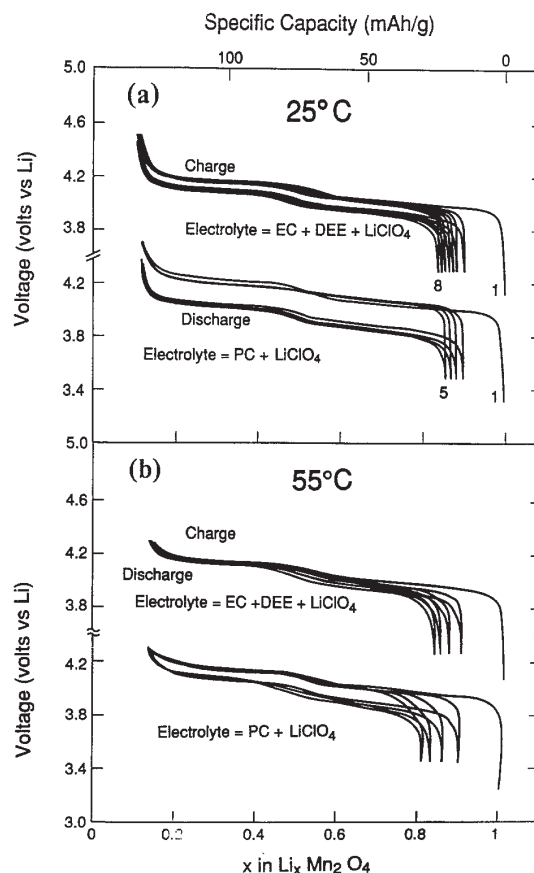


Fig. 3. Typical cycling behavior at 25 (a) and 55°C (b) of LiMn₂O₄/Li cells using EC + DEE (50:50) + 1M LiClO₄ and PC + 1M LiClO₄ electrolytes. In (a) the cells contain 30 mg (of active material) and were cycled between 3.5 and 4.5 V at a current of 0.4 mA/cm² ($\Delta x = 1$ in 22 h). In (b) the cells contain 10 mg (of active material) and were cycled between 3.5 and 4.3 V at a current of 0.2 mA/cm² ($\Delta x = 1$ in 15 h).

vs. Li. The removal of one Li⁺ from the structure (leading to λ -MnO₂) occurs in two steps close to 4.1 V *vs.* Li as shown in previous work (17, 23-26). On discharge, λ -MnO₂ converts back reversibly to LiMn₂O₄ in two steps. Independently of the electrolyte used, the material shows the same reversible capacity after the first charge-discharge cycle, $\Delta x \approx 0.8$, corresponding to 119 mAh/g of active material. The loss in capacity during the first cycle ($\Delta x \approx 0.2$) may arise from particles that become electrically disconnected and also results from the fact that the battery is cycled under constant current without any relaxation so as to allow the system to reach equilibrium upon Li⁺ intercalation. For composite LiMn₂O₄ electrodes of the same thickness, the polarization ($V_{\text{charge}} - V_{\text{discharge}}$) is about three times lower in the EC- than in the PC-based electrolyte (Fig. 3a), consistent with the higher ionic conductivity measured for the EC- than the PC-based electrolyte.

In order to determine the maximum voltage to which rechargeable Li cells based on LiMn₂O₄ as the positive electrode can operate safely, we have tried several cut-off voltages on charge. Figure 4 shows what happens if the charge cut-off voltage of a LiMn₂O₄/Li cell is increased after a few cycles. A plateau is obtained at 4.59 V *vs.* Li, corresponding to an irreversible electrochemical reaction. As a result, the next discharge curve is shifted to lower values of *x*, with a value corresponding to the capacity length of this plateau. This reaction could be the oxidative degradation of the λ -MnO₂ framework or the oxidation of the electrolyte at the surface of the material. The x-ray diffraction pattern of the sample after this treatment shows no difference

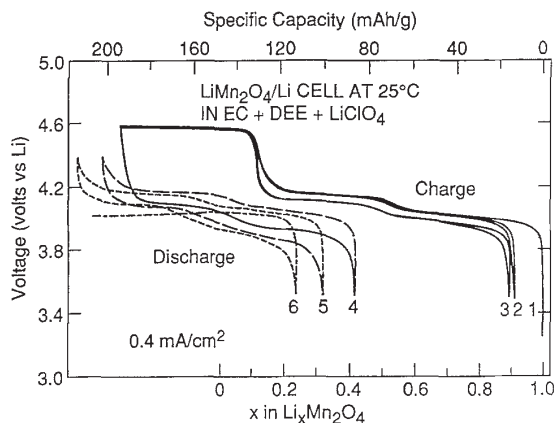


Fig. 4. Cycling behavior at 25°C of a $\text{LiMn}_2\text{O}_4/\text{Li}$ cell in EC + DEE (50:50) + 1M LiClO_4 when the charge cut-off voltage is increased above 4.5 V. The pellet was 30 mg (active material) and a current rate of 0.4 mA/cm^2 was used ($\Delta x = 1$ in 22 h). The charge cut-off voltage was 4.5 V for the 2 first charges, then 4.6 V for the third charge, and then 4.4 V for the following charges. The discharge cut-off voltage was always 3.5 V.

with the one for $\lambda\text{-MnO}_2$, excluding the first possibility. In an attempt to avoid further oxidation of the electrolyte, the following charges were limited to 4.4 V vs. Li. However, at each successive cycle, even if the voltage is kept lower than 4.4 V vs. Li, the voltage-composition curves show lower capacity and larger polarization than a cell that has not been charged above 4.5 V vs. Li. In addition, these curves are shifted to larger capacity values, due to some irreversible behavior coming from the oxidation of the electrolyte. After a few cycles, the high voltage cannot be reached and the delithiation of the spinel no longer occurs (e.g. the cell cannot be used any longer). These results indicate that once the oxidation of the electrolyte has been initiated by an increase in cut-off voltage, its decomposition voltage is lowered into the cycling window so that it cannot be stopped even by lowering the cut-off voltage on subsequent cycles. This emphasizes the importance of carefully controlling the overvoltage in charging these cells: 4.45 V vs. Li is a safe limit at room temperature that allows one to use the whole capacity without risk of electrolyte decomposition.

The behavior of the LiMn_2O_4 material at high voltage has been studied further by cyclic voltammetry at room temperature. The three electrode technique allows one to study only the contribution of the $\text{LiMn}_2\text{O}_4/\text{electrolyte}$ interface and consequently avoid any phenomenon occurring at the Li electrode or any secondary reaction susceptible to occur at the metal current collector in Swagelok test cells. The voltammograms of Fig. 5 show two peaks in oxidation and two peaks in reduction, when the electrode material is cycled between 3.3 and 4.45 V vs. Li, which are the signature of the two-step reversible transformation between LiMn_2O_4 and $\lambda\text{-MnO}_2$ (17, 21-23). If the higher voltage limit is increased above 4.5 V vs. Li, an oxidation current appears on the anodic scan direction. On the following cathodic scan, above 4.5 V vs. Li, this current is always oxidative, so it leads to irreversible capacity. This current is intrinsic to the material, due to oxidation of the electrolyte on the electrode material surface, because the oxidation of the electrolyte on the Pt holder remains slow in the same voltage range (dot-dashed line in Fig. 5). On the subsequent cycles, the current peaks become larger and badly defined, in contrast to that observed if the voltage is always kept below the limit 4.5 V vs. Li. This degradation of the behavior of the manganese oxide could be due to the deposition of an insulating layer, composed of the products of the electrolyte oxidation, at the surface of the grains. Comparing the curves obtained in both electrolytes, we see that the voltage separation between the peaks in the oxidation scan direction and in the reduction scan

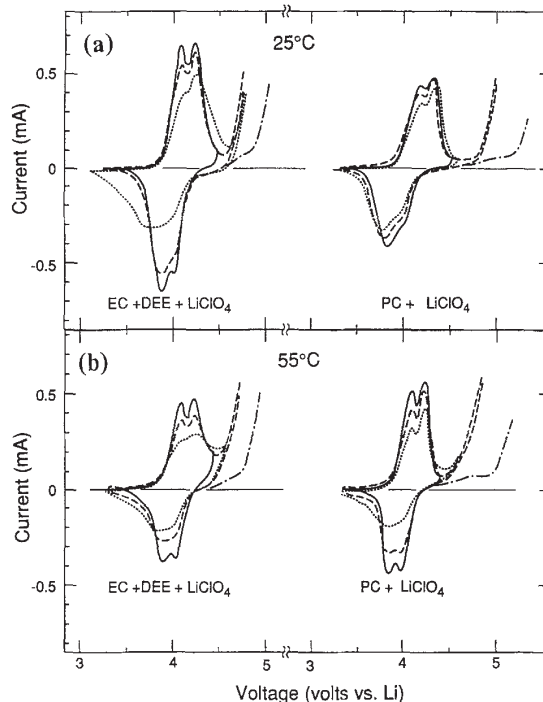


Fig. 5. Cyclic voltammograms at LiMn_2O_4 in EC + DEE (50:50) + 1M LiClO_4 and PC + 1M LiClO_4 electrolytes at 25°C (a) and at 55°C (b). For all the voltammograms the starting voltage is close to 3.2 V and the scan rate is of 0.1 mV/s. The solid line, dashed line and dot line refer to the 1st, 2nd, and 4th cycle, respectively, while the dot-dashed line is obtained as a blank on the Pt grid holder alone until very high voltage is used. For the voltammograms (a) the voltage is first scanned to 4.45 V and back to 3.3 V (solid line) and then to 4.75 (for EC-based electrolyte) or 4.95 V (for PC-based electrolyte) for following sweeps in the positive direction. For the voltammograms (b) the voltage is first scanned to 4.42 (for EC-based electrolyte) or 4.37 V (for PC-based electrolyte) and back to 3.2 V (solid line) and then to 4.7 (for EC-) or 4.85 V (for PC-) for following sweeps in the positive direction.

direction (e.g., the polarization) is lower in EC- than in PC-based electrolyte, because the ionic conductivities of the two media are different. Moreover, the oxidation of the electrolyte on the material surface occurs at a lower voltage and the degradation of the shape of the voltammograms occurs faster in the EC-based electrolyte. From this study, we conclude that the degradation of the reversible cycling behavior of the LiMn_2O_4 material after an overcharge exceeding 4.5 V vs. Li is intrinsic to the $\text{LiMn}_2\text{O}_4/\text{electrolyte}$ interface and arises from the electrolyte oxidative decomposition at the surface of the material grains.

Another important question to be addressed is what happens at higher operating temperature where a battery operates in practice. Figure 3b shows the cycling behavior of $\text{LiMn}_2\text{O}_4/\text{Li}$ cells at 55°C under constant current in the two electrolytes we selected. Note that a lower charge cut-off voltage than the one used at room temperature (4.3 V vs. Li instead of 4.5 V vs. Li) was sufficient to obtain the complete capacity. Again, the polarization is greater in PC- than in EC-based electrolytes but less than that observed at room temperature, as expected, because of an increase in the ionic diffusion rate with increasing temperature. The loss of capacity during the first few cycles is slightly larger than at room temperature, and is slightly larger in PC- than in EC-based electrolytes.

The same cyclic voltammetry study as was done at room temperature shows that at 55°C the electrolyte oxidation limit is effectively lowered by about 0.2 V in EC and 0.25 V in PC (Fig. 5b). As observed at room temperature the electrolyte oxidation still occurs at a higher voltage in PC than

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