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Thin Film Rechargeable Room Temperature Batteries Using Solid Redox Polymerization Electrodes

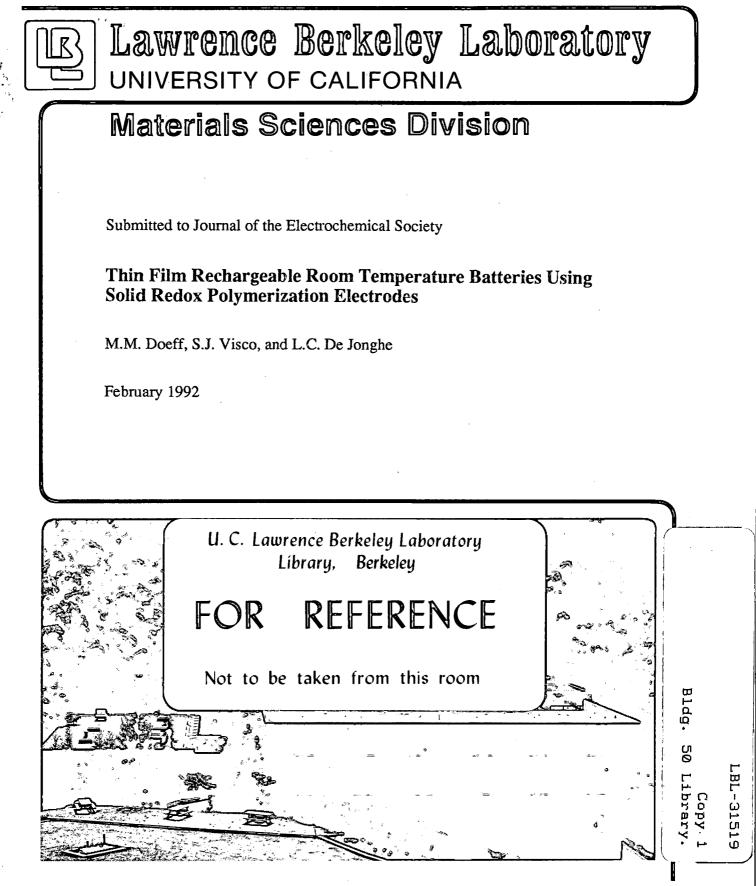
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THIN FILM RECHARGEABLE ROOM TEMPERATURE BATTERIES USING SOLID REDOX POLYMERIZATION ELECTRODES

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Abstract

Thin-film solid-state batteries consisting of lithium foils, amorphous PEO separators and solid redox polymerization electrodes (SRPEs) were assembled, discharged and cycled at room temperature. No solvents were added to any of the components, nor were structural additives used. Performances were studied as a function of cathode thickness and composition of separator and SRPE. At 50 μ A/cm², cells could be discharged to a depth of 0.6 to 1.3 C/cm², at 100 μ A/cm² to a depth of 0.5 C/cm², and at 200 μ A/cm² to a depth of 0.25 C/cm². It was also possible to pulse batteries at higher current densities for short periods of time (ranging from 0.1 to 3 seconds) with instantaneous recovery of open circuit potential after the pulses. One cell was cycled 100 times, with inadvertent overdischarge and overcharge, before significant deterioration of performance occurred. Batteries may be designed to be paper thin, or may consist of several cells stacked together to give a somewhat thicker device. Practical energy and power densities were calculated as a function of component dimensions (cathode and current collector thicknesses) for paper thin batteries consisting of lithium anodes, amorphous PEO separators, SRPEs and metallized plastic current collectors. Power densities of 30 W/I (continuous discharge) and pulse (0.1 seconds) power densities over 1000 W/I may be achieved for these ultra thin devices.

Introduction

In the past decade, there has been much work directed towards the development of thin-film solid-state lithium polymer batteries, in which the conventional liquid electrolyte has been replaced by an ion-conducting polymer, most commonly, polyethylene oxide.¹ This feature eliminates the

This research was supported by the Assistant Secretary for Conservation and Renewable Energy, Deputy Assistant Secretary for Utility Technologies, Office of Energy Management, Advanced Utility Concepts Division of the U.S. Department of Energy under Contract No. DE-AC03-76F00098.

¹ M. A. Ratner and D. F. Shriver, Mat. Res. Soc. Bull., 9, 39, (1989).

hazards associated with the combination of lithium and an organic liquid. Another recent development has been the use of solid redox polymerization electrodes (SRPEs) as positives in thin film batteries. The active materials in SRPEs are polymeric disulfides that undergo reductive depolymerization upon discharge, and oxidative polymerization upon charge (equation 1).²

$$(SRS)_{n} \xrightarrow{2n e^{-}} n \ SRS^{-} \qquad (1)$$

SRPEs display a number of highly desirable characteristics, such as low cost and toxicity, good cyclability and high power and energy densities. In general, it is necessary to operate cells with polymeric electrolytes above 60° C to maintain the polyethylene oxide complexes in an amorphous, and thus, conducting state. The addition of small amounts of nonvolatile organic liquids such as propylene carbonate or short chain polyethylene glycols allows operation at room temperature (e.g., solvent-swollen films³) but the disadvantages associated with use of liquid electrolytes remain in such systems. Another approach has been to synthesize new or modified polymers with high room temperature ionic conductivities⁴, which would allow construction of truly solid state room temperature batteries. Most of these polymers contain ethylene oxide groups, either as side chains (for example in MEEP, methoxyethoxy polyphosphazene⁵, or as units in block co-polymers or linked polymers. An example of the last is amorphous PEO⁶. Conductivities of lithium salt complexes of oxymethylene linked polyethylene glycol 400 (PEG 400) are as high as 10⁻⁴ ohm-cm⁻¹ at room temperature⁷. Surprisingly, few reports of all solid-state amorphous PEO (or other solid polymer electrolyte) room temperature batteries appear in the literature⁸. For this

² S. J. Visco, M. Liu, M. B. Armand and L. C. De Jonghe, *Mol. Cryst. Liq. Cryst.*, 190, 185, (1990).

³ K. M. Abraham and M. Alamgir, *J. Electrochem. Soc.*, **137**, 1657, (1990).

b) I. E. Kelly, J. R. Owen and B. C. H. Steele, J. Power Sources, 142, 13 (1985).

⁴ a) F. Croce, S. Passerini, A. Selvaggi and B. Scrosati, Solid State Ionics, 40/41, 375 (1990).

b) M. C. Wintersgill, J. J. Fontanella and M. K. Smith, Polymer, 28, 633 (1987).

c) J. M. G. Cowie in *Polymer Electrolyte Review I*, C. A. Vincent and J. MacCallum eds. Elsevier, London (1987) pp. 69-102.

⁵ M. Blonsky, D. F. Shriver, P. Austin and H. R. Allcock, J. Am. Chem. Soc., **106**, 6854 (1984).

⁶ C. V. Nicholas, D. J. Wilson and C. Booth, Brit. Polymer Journal, 20, 289 (1988).

⁷ C. Booth, C. V. Nicholas and D. J. Wilson in *Polymer Electrolyte Reviews II*, J. R. MacCallum and C. A. Vincent eds. Elsevier, London, 1989 pp. 229-254.

⁸ a) M. Gauthier, D. Fauteux, G. Vassort, A. Belanger, M. Duval, P. Ricoux, J.-M. Chabagno, D. Muller, P. Rigaud, M. B. Armand and D. Deroo, *J. Electrochem. Soc.*, **132**, 1333 (1985).

study, batteries consisting of lithium anodes, amorphous PEO separators and SRPEs were assembled, discharged, pulsed, and cycled successfully at room temperature, with no addition of solvents or plasticizers.

Experimental

2,5-dimercapto-1,3,4-thiadiazole (Aceto Corp.), LiOH (Aldrich, 99.9+%), iodine (Baker, sublimed) and acetonitrile (Aldrich, HPLC grade) were used as received. Samples of amorphous PEO were received as gifts from Duward Shriver of Northwestern University and Christian Nicholas of Manchester University, and were stored under an inert atmosphere. Carbon black (Shawinigan acetylene black, Chevron), and various lithium salts were dried under vacuum at 50°C and stored under an inert atmosphere. Lithium foil of approximately 5 mil thickness (Lithco Co., battery grade) or 1 mil thickness (Cyprus Foote Mineral Company, experimental product) was stored under helium and was cut into disks (18 mm diameter) directly prior to use.

2,5-dimercapto-1,3,4-thiadiazole was converted to the lithium thiolate salt by reaction with a stoichiometric amount of LiOH. The salt was then oxidized with iodine in acetone/water to the polymer, X1. The light yellow polymer was washed well with hexane, acetone and water and dried for at least 48 hours under vacuum at 50° C, then stored under an inert atmosphere. Positive electrodes were prepared from a mixture of X1, carbon black, carbon dispersant and amorphous PEO/lithium salt (LiX) in a ratio of 20 ethylene oxide units per Li in acetonitrile. Some cathode mixtures were formulated without the lithium salts. Films of about 10-100 μ m thickness were cast onto Teflon or directly onto stainless steel current collectors and air-dried. Films cast on Teflon were cut to 2 cm², and then dried *in vacuo* for at least 48 hours before use. Freestanding amorphous PEO₂₀LiX separator films of comparable thicknesses were prepared similarly.

Test cells were assembled under an inert atmosphere and discharged at 25° C. In most cases, a larger SRPE was shielded down to an area of 1 or 2 cm² with a 19 μ m thick polypropylene spacer. The Li electrode capacity exceeded that of the positive electrode by more than 400%, and thus did not limit performance. Discharge and cycling characteristics were examined with a PAR 173 or 371 potentiostat/galvanostat using software developed in this laboratory. Cell internal

b) G. Vassort, M. Gauthier, P. E. Harvey, F. Brochu and M. B. Armand in *Proceedings of the Symposium on Lithium Batteries*, Honolulu, 1987, Vol. 88-1, ed. A. N Dey, the Electrochemical Society, Pennington, NJ, 1988.

c) S. Pantaloni, S. Passerini, F. Croce, B. Scrosati, A. Roggero and M. Andrei, *Electrochim. Acta*, **34**, 635 (1990).

d) C. Arbizzani, M. Mastrogostini, T. Hamaide and A. Guyot., Electrochim. Acta, 35, 1782 (1990).

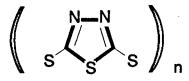
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resistances were determined *in situ* by a pulse technique described elsewhere⁹. Galvanostatic discharge or charge was performed at various current densities and cell OCVs were monitored periodically throughout the experiments.

Results and Discussion

The goal of this study was to evaluate the performance of solid state room temperature cells with lithium anodes, polymeric electrolytes and SRPEs, and to ascertain whether they might have practical uses, for example, in consumer applications. Experiments were designed to explore their characteristics and limitations, especially in terms of discharge, cycling and pulse behavior. One of the most important factors in designing such cells is an appropriate choice of positive electrode material. Although a number of organopolydisulfides have been discharged successfully in 90° C lithium polymer electrolyte cells¹⁰, electrochemical characterization of these materials have shown that simple aliphatic disulfides exhibit slow electron transfer at room temperature and large activation energy for charge transfer¹¹. These materials, although appropriate for higher temperature uses, most likely would not perform satisfactorily at room temperature because of their limited rate capability. It has been further shown that organodisulfides containing N-C-S bonds exhibit the fastest electrode kinetics and lowest activation energies for charge transfer. In addition, many of these compounds lack the characteristic stench associated with mercaptans. For these reasons, the polydisulfide of dimercaptothiadiazole, termed X1, was chosen for the room temperature studies.

The structure of X1, the active material in the SRPEs used for this study is shown below.



The open circuit potential for X1/Li room temperature cells ranges from 3.0 to 3.2 V. The currentoverpotential response during the pulse tests was linear over a range of about 200 mV, showing that kinetics do not limit performance at room temperature. Discharge profiles were flat and cells

⁹ S. J. Visco and M. Liu, submitted to J. Appl. Electrochem.

¹⁰ M.M. Doeff, S.J. Visco and L.C. De Jonghe, submitted to the *J. Electrochem. Soc.*

¹¹ a) M. Liu, S.J. Visco and L.C. De Jonghe, J. Electrochem. Soc., **137**, 750 (1990).

b) M. Liu, S.J. Visco and L.C. De Jonghe, *Proceedings of the Symposium on Primary and Secondary Lithium Batteries*, **91-3**, 62 (1991).

could be discharged to a depth of 0.6 to 1.3 C/cm² at a current density of 50 μ A/cm², depending upon the composition of the components (type of amorphous PEO lithium salt complex used in the cathode and separators). Figure 1 shows typical first discharge profiles for several room temperature devices at this rate. Open circuit potentials were monitored by turning the current off periodically during the discharges for brief periods (the spikes in the profiles).

Two different types of amorphous PEO were used in this study, as well as several different lithium salts. The type of amorphous PEO (hereafter abbreviated as aPEO) used in the SRPEs and separators had little effect on the discharge characteristics. However, separators made from lower molecular weight aPEO tended to creep more than those made from higher molecular weight materials, and consequently cells containing the former were somewhat more prone to shorting.

SRPEs that did not contain added lithium salts could be discharged to a greater depth (1.1 to 1.3 C/cm^2) than those containing lithium salts (compare curves a and b to c, d and e in Figure 1), but performance upon recharge was less satisfactory. Indeed, the best cycling results to date at room temperature have been in cases where the positive electrode contained added lithium salt. Cells containing LiClO₄ discharged poorly (results not shown), but those containing lithium triflate in both cathode and separator (curves d and e) could be discharged to over 0.8 C/cm².

In SRPEs that contain added lithium salts, the lithium dithiolate produced during discharge may cause a "salting out" effect, resulting in premature cell polarization. In contrast, SRPEs with no added lithium salts may be discharged further (the discharge products providing ionic conductivity in the cathode), but upon charge, the lithium dithiolate in the positive electrode is depleted, causing a decrease in conductivity and poor rechargeability.

Figure 2 shows 50 μ A/cm² first discharges for cells with cathodes of various thicknesses, but that had identical compositions. Evidently, these batteries are always discharged to the same depth regardless of cathode thickness; thus utilizations are correspondingly lower as capacity is increased. In contrast, utilization in cases where the capacity is less than 1 C/cm² is typically 80% or greater at 50 μ A/cm². Fortunately, calculations (*vide infra*) show that maximum practical power and energy densities are also obtained when cathode thickness is limited to below 1 C/cm².

The tendency to discharge to a constant depth upon the first cycle regardless of the cathode capacity holds true at higher current densities as well. Thus, these examples may be discharged to about 0.8 C/cm² at 50 μ A/cm² (or over four and a half hours operating time), about 0.45 C/cm² at 100 μ A/cm² (over one hour) and 0.27 C/cm² at 200 μ A/cm² (twenty-four minutes) (Figure 3).

The OCV measurements taken throughout these experiments show that overpotential increases during discharge and that the rate of increase grows faster for higher current densities. The voltage drops precipitously at the end of discharge (although battery voltages were prevented from

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dropping below 1.8 to prevent deterioration.) This polarization is probably due to diffusional processes, and the performance therefore is limited primarily by the relatively low ionic conductivity of the polymer electrolyte. The "salting out" effect noted earlier may also play a role in this. It has already been recognized that very thin components are required in batteries with SPEs to offset the low ionic conductivities; this is even more important for optimum performance of the room temperature aPEO cells.

The results presented in Figures 1-3 were generally very reproducible, but very occasionally cells performed poorly on first discharge. They often improved markedly on subsequent cycles, however, discharging to the depths shown in the figures. It is not entirely clear what causes this conditioning, but it may be due to the presence of a poorly conducting contaminant on the lithium surface or in one of the other components that is removed upon initial discharge or charge. After recovery to the usual depths of discharge, the anomalous cells performed normally; i.e. depth of discharge either remained the same or decreased somewhat upon cycling. Unless otherwise noted, the results presented in this paper show first discharges for normal examples.

Figure 4 shows the first twenty-one cycles of a cell that was discharged at 50 or 100 μ A/cm² and recharged at 25 μ A/cm² 100 times. (After this point, discharges were always 50 μ A/cm².) This is an example of conditioning upon cycling; the first cycle is markedly worse than subsequent ones. For the first through the twentieth cycle, depth of discharge was 0.8 C/cm² at 50 μ A/cm². After this, an inadvertent overdischarge occurred (reversal) due to a malfunction in the cycling equipment. This had a deleterious effect at first (depth of discharge decreased to 25% of cycle 20). By cycle 37, however, the cell had recovered to 70% of cycle 20. A similar pattern was seen after an accidental overcharge of 100%. By cycle 93, depth of discharge was 50% of cycle 20. There was evidence of formation of micro dendrites during several cycles manifested as voltage instability during charge, but these "cycled out." Performance deteriorated irreversibly after 100 cycles, perhaps due to cumulative effects of micro dendrites. It is apparent, however, that the X1 room temperature cells are remarkably robust as shown by their ability to withstand abuses such as overcharge and high rate discharge, with minimal loss of function.

Because lithium is known to react with many organic materials, it is critical to assess the stability of any new polymer electrolyte towards this metal in a battery, whether at rest or when undergoing cycling. The relatively long term cycling results presented above (almost two months) suggest that aPEO is indeed suitable for use in lithium batteries.

The ability of the Li/aPEO/X1 cells to recover from severe polarization suggests that this system might be good for high rate pulse applications. In order to test this possibility, newly assembled, or partially discharged cells were subjected to pulses of varying lengths at different current densities. Some were pulsed for 3-5 seconds at 1.5 mA/cm². After three seconds the voltage dropped to 1 V and after four seconds, to 0 V, but recovery to the original OCV occurred

-6-

essentially instantaneously. Cells pulsed for one second at 0.5 mA/cm² did not polarize; the voltage only dropped to 2.9 V and recovered instantaneously. The behavior did not seem to depend upon the previous history of the batteries (whether newly assembled or partially discharged), although OCVs were somewhat lower for the latter. Figure 5 shows a cell (15% discharged) pulsed at various current densities up to 3 mA/cm² for 0.1 seconds with a 0.15 second rest period between pulses. Recovery to the original open circuit potential occurred in less than 0.15 seconds even when the voltage dropped to nearly 0 upon pulsing.

Table 1 lists dimensions of components and practical energy densities for a paper thin Li/aPEO/X1 room temperature device. This battery would have a three second pulse power density of 500 W/l, based upon the discharge behavior seen for 1.5 mA/cm² pulses. Figure 6 shows the variation of power density with current density for the 0.1 second pulses presented in Figure 5, for several cathode and current collector thicknesses. The maximum power density of over 1000 W/l is obtained when 1 μ m current collectors, 0.5 C/cm² SRPEs and a current density of 1.7 mA/cm² is used.

The calculations for the paper thin example presented in Table 1 and Figure 6 assume that lithium is used in fourfold excess over X1 in a cathode 0.5 to 1.2 C/cm² thick and that either 1 or 5 μ m metallized plastic is used as current collectors (Such materials are already widely used in the capacitor industry and have been proposed for use in high temperature solid polymer electrolyte batteries¹².) One advantage to these solid state devices is that the thin film components may easily be stacked in series to form a larger battery pack. Durable devices may be fabricated from cells containing the same size components as above, but with thicker outer current collectors. This also results in slightly better practical energy and power densities. For example, the battery shown schematically in Figure 7 consists of four cells with 5 μ m thick internal current collectors and 20 μ m thick external current collectors. It would be only 225 μ m thick (similar to heavy stock paper) and would have an energy density of 140 Wh/kg (150 Wh/l), and continuous power density of 30 W/l. It might be possible to reduce the thickness (and increase energy density) further by using 1 µm metallized plastic internal current collectors (also commercially available), for a total thickness of 193 µm and volumetric energy density of 163 Wh/I. These values, although somewhat lower than those for Li/PEO/SRPE cells operating at 90° C, are compatible with the requirements for powering small consumer devices.

Figure 8 shows the variation of practical power and energy density of single cells as a function of cathode capacity (lithium always in fourfold excess over X1 and 10 μ m separators), for two different thicknesses of current collectors. The energy densities are calculated using the average

¹² a) M. Z. A. Munshi and B. B. Owens, Solid State Ionics, 38, 87 (1990).

b) M. Z. A. Munshi and B. B. Owens, Solid State Ionics, 38, 95 (1990).

-7-

operating voltage during 50 μ A/cm² discharge, and the power densities are calculated assuming either 50 or 100 μ A/cm² discharges. The results clearly show that there is little to be gained in terms of energy density for SRPE thicknesses above 1 C/cm², even if it were possible to discharge to this extent. Likewise, the best compromise between energy and power densities is obtained when the cathode capacity is below 1 C/cm².

The calculations presented in Figure 8 show that selection of appropriate component dimension is critical for good battery design as well. Devices intended for pulsed applications where power densities above 100 W/I may be required should have very thin cathodes (0.5 C/cm^2 or about 9 μ m). If energy density and continuous application are more important than power density, thicker SRPEs should be used. Another consideration for cell design, of course, is economic. The raw material costs for these batteries are estimated to be quite low¹³, but as cathode thickness is decreased below about 0.5 C/cm², relative expense increases due to the dominance of the current collector cost and that of producing the ultrathin lithium.

The results presented herein demonstrate that all solid state batteries using aPEO as the ion conducting medium can be operated at room temperature successfully without additives or special methods of construction. Indeed, the cycling behavior, pulse behavior, energy and power densities and cost analysis for room temperature aPEO cells with SRPEs are among the best results to date for room temperature solid state lithium batteries. SRPEs have already attracted much attention in high temperature thin film cells because of their low cost, low toxicity and impressive discharge and cycling characteristics. Development of a room temperature SRPE battery for commercial use would represent an exciting breakthrough in solid state battery technology. Future research in this laboratory will be directed towards the understanding of fundamental processes in this system and optimization of performance, particularly with regard to cycle life.

Acknowledgment

The authors would like to thank Professor Duward Shriver of Northwestern University, Professor Colin Booth and Dr. Christian Nicholas of Manchester University for samples of amorphous PEO and for helpful discussion.

¹³ S. J. Visco, M. M. Doeff, and L. C. De Jonghe, *Fifth International Seminar on Lithium Battery Technology and Applications*, Deerfield Beach, FI 1991.

 Table 1. Component Dimensions and Energy Densities for a Li/aPEO/SRPE Cell.

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Component	Thickness (μm)	Weight/cm ² , grams
Lithium (4 C/cm ²)	6	0.0003
aPEO separator	10	0.0015
SRPE (1 C/cm ²)	18	0.0026
Metallized current collectors	5 (x2)	0.00092
Total	44	0.00532
Energy Density	190 Wh/I	160 Wh/kg

Figure Captions

Figure 1. First discharges of Li/aPEO/SRPE cells at 25° C, 50 μ A/cm²; components contain various kinds of aPEO and lithium salts. a) and b) Cathode contains X1 and high molecular weight aPEO but no lithium salt. b) Separator contains lithium triflate. c) Separator and cathode contain lithium salt, and lower molecular weight aPEO. d) Separator and cathode contain high molecular weight aPEO and lithium triflate. e) Separator contains high molecular weight aPEO and lithium triflate. e) Separator contains high molecular weight aPEO and lithium triflate. e) Separator contains high molecular weight aPEO and lithium triflate.

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Figure 2. Utilization as a function of cathode thickness or capacity for cells consisting of a lithium anode, $aPEO_{20}LiSO_3CF_3$ separator and SRPE containing X1 and $aPEO_{20}LiSO_3CF_3$, undergoing first discharges at 50 μ A/cm².

Figure 3. Discharges of cells consisting of lithium anodes, $aPEO_{20}LiSO_3CF_3$ separators and X1 cathodes with $aPEO_{20}LiSO_3CF_3$ at various rates (first cycle).

Figure 4. First twenty-one cycles of a Li/aPEO/X1 cell. Discharges were either 50 or 100 μ A/cm², and charges were 25 μ A/cm². Rates for cycles 22-100 (not shown) were 50 μ A/cm² discharge and 25 μ A/cm² charge.

Figure 5. Pulse behavior of a cell (15% discharged) consisting of a lithium anode, $aPEO_{20}LiSO_3CF_3$ separator and $aPEO_{20}LiSO_3CF_3/X1$ cathode. The top curve (dotted line) shows pulses ranging from 40 to 300 μ A/cm², and the bottom curve (solid line) shows pulses from 0.4 to 3 mA/cm². Pulses lasted 0.1 seconds with 0.15 second rests in between. Note that recovery to the open circuit voltage was very rapid.

Figure 6. Pulse power densities calculated from data shown in Figure 5, for devices with either 0.5 or 1.2 C/cm^2 thick cathodes, 10 μ m separators, lithium in fourfold excess and either 1 or 5 μ m metallized polyethylene terephthalate (PET) current collectors. Pulses are 0.1 seconds long with 0.15 seconds rest in between pulses.

Figure 7. Schematic of a device containing four internally stacked cells. Cells consist of lithium foil, an amorphous PEO separator, and an SRPE made with amorphous PEO. Current collectors are metallized plastic. Dimensions of the components are from Table 1. The entire cell is 225 μ m thick.

Figure 8. Dependence of energy density (upper two lines) and power density (lower four lines) on capacity, for 1 cm² cells with 1 μ m or 5 μ m thick metallized plastic current collectors, 10 μ m thick aPEO separators and lithium in fourfold excess. Power densities were calculated for two different current densities, either 50 μ A/cm² or 100 μ A/cm² as noted. Energy densities are calculated using voltages for a cell undergoing discharge at 50 μ A/cm².

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Cell Voltage (V)

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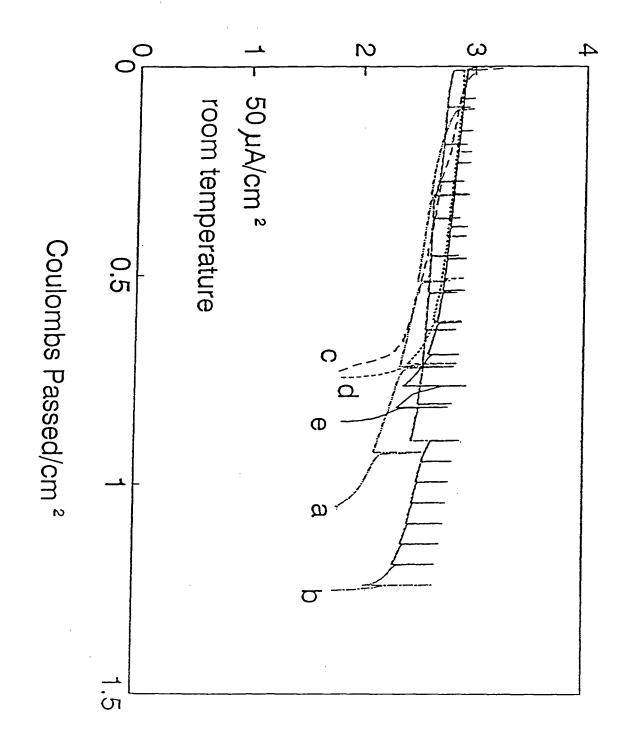


Figure 1.

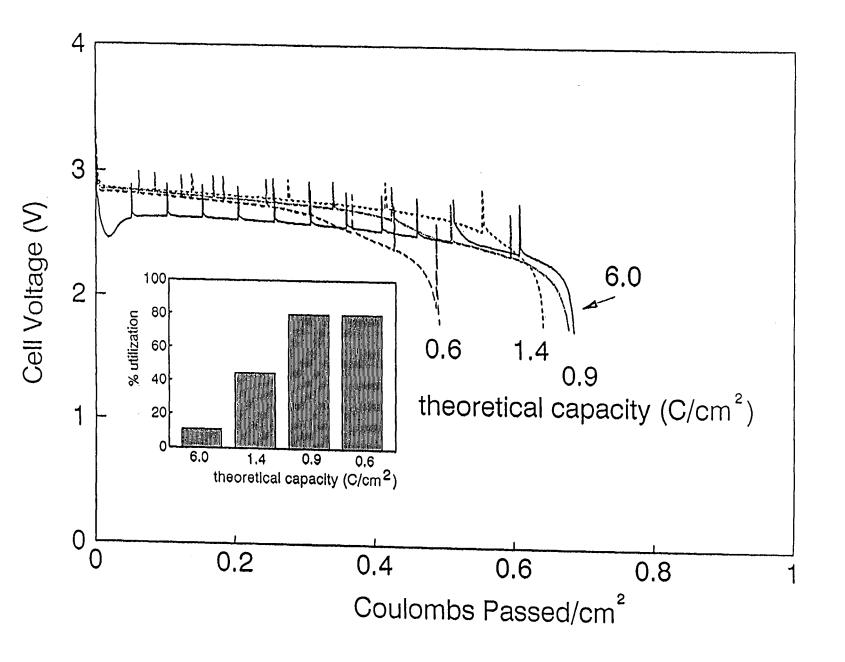
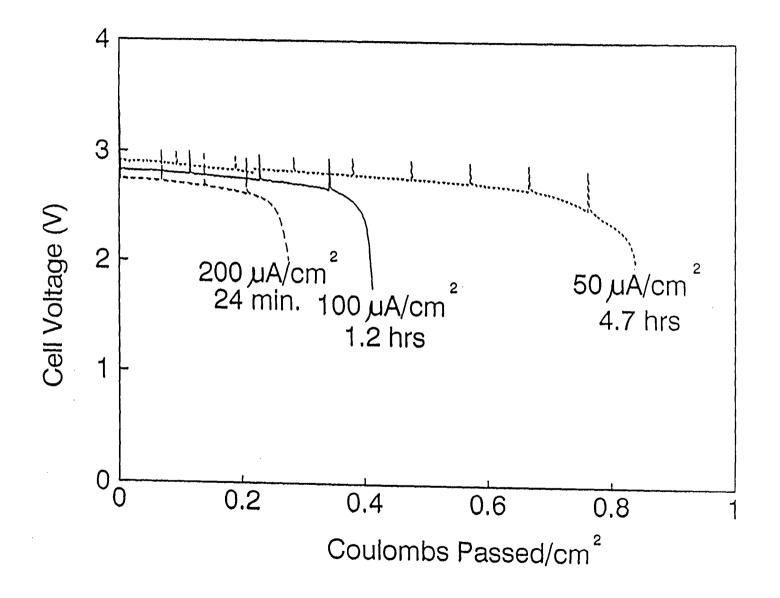


Figure 2.

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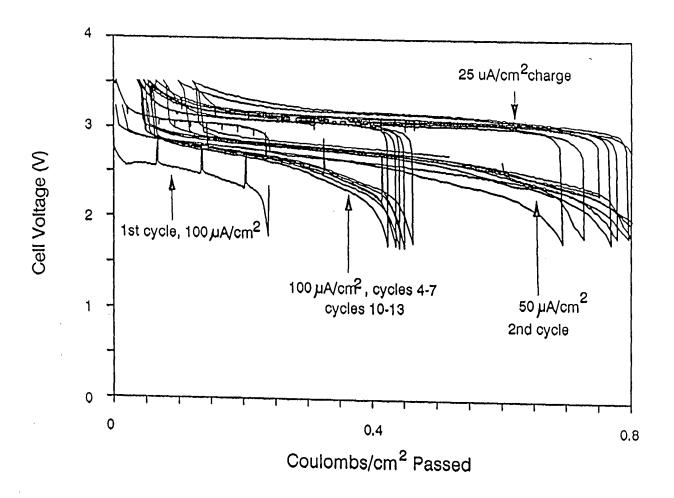
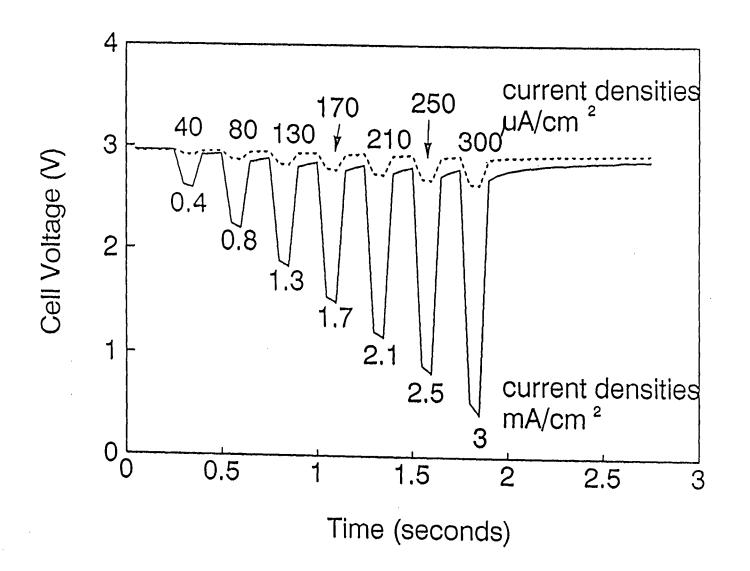
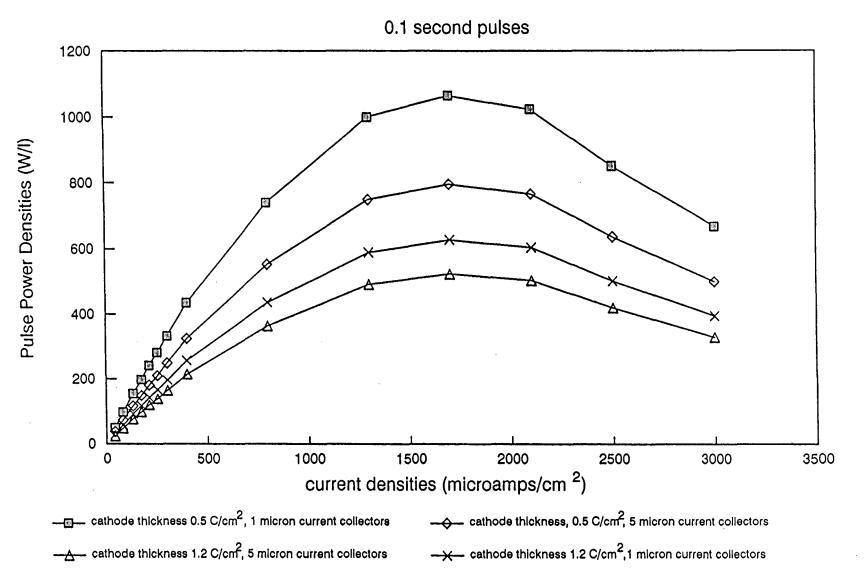


Figure 4.



Pulse Power Densities



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Figure 6.

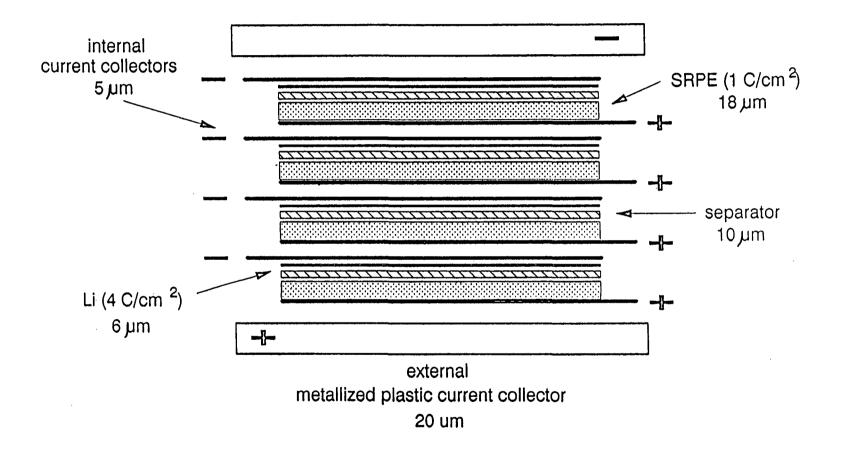
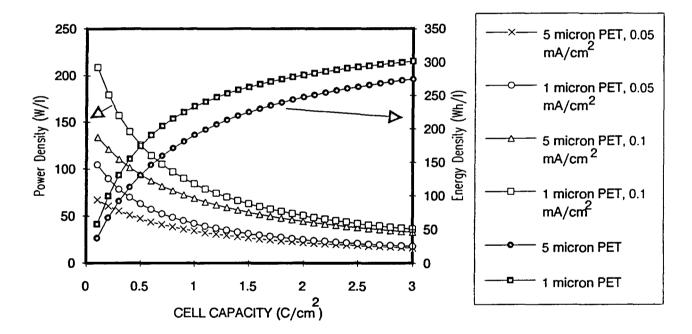


Figure 7. -18-



Variation of Energy and Power Density with Cell Capacity

Figure 8.

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