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Intercalation of Lithium in Pitch-Based Graphitized Carbon Fibers Chemically Modified by Fluorine: Soft Carbon With or Without an Oxide Surface

Ching-cheh Hung
Glenn Research Center, Cleveland, Ohio

Aniko Prisko
Cleveland State University, Cleveland, Ohio

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This report contains preliminary findings, subject to revision as analysis proceeds.

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INTERCALATION OF LITHIUM IN PITCH BASED GRAPHITIZED CARBON FIBERS CHEMICALLY MODIFIED BY FLUORINE: SOFT CARBON WITH OR WITHOUT AN OXIDE SURFACE

Ching-cheh Hung
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Aniko Prisko
Cleveland State University
Cleveland, Ohio 44115

SUMMARY

The effects of carbon structure and surface oxygen on the carbon's performance as the anode in lithium-ion battery were studied. Two carbon materials were used for the electrochemical tests: soft carbon made from defluorination of graphite fluoride, and the carbon precursor from which the graphite fluoride was made. In this research the precursor was graphitized carbon fiber P-100. It was first fluorinated to form $CF_{0.68}$, then defluorinated slowly at 350 to 450 °C in bromoform, and finally heated in 1000 °C nitrogen before exposed to room temperature air, producing disordered soft carbon having basic surface oxides. This process caused very little carbon loss. The electrochemical test involved cycles of lithium intercalation and deintercalation using C/saturated LiI-50/50 (vol %) EC and DMC/Li half cell. The cycling test had four major results: (1) The presence of a basic oxide surface may prevent solvent from entering the carbon structure and therefore prolong the carbon's cycle life for lithium intercalation-deintercalation, (2) The disordered soft carbon can store lithium through two different mechanisms. One of them is lithium intercalation, which gives the disordered carbon an electrochemical behavior similar to its more ordered graphitic precursor. The other is unknown in its chemistry, but is responsible for the high-voltage portion (>0.3V) of the charge-discharge curve, (3) Under certain conditions, the disordered carbon can store more lithium than its precursor, (4) These sample and its precursor can intercalate at 200 mA/g, and deintercalate at a rate of 2000 mA/g without significant capacity loss.

INTRODUCTION

Carbon material as the anode of lithium-ion batteries has been the subject of recent research. It has been reported that both the structure and the surface of the carbon materials are important parameters affecting their performance as the anode in lithium-ion batteries. The surface is important in forming a surface-electrolyte interface (SEI) layer during the first cycle of lithium intercalation. The SEI layer, if properly formed, can effectively allow lithium ions to enter the carbon layers and at the same time protect the intercalated lithium by keeping the solvent out of the carbon structure. The carbon structure, on the other hand, is responsible for the capacity of lithium intercalation. Recently, it has been reported that, for certain types of carbon, the capacity of lithium storage in the lithium-ion battery can exceed the value predicted by the generally accepted theory (Li/C = 1/6) (refs. 1 to 3).

The objective of this research is to further investigate the effects of carbon structure and carbon surface on the performance of the carbon as the anode in the lithium-ion battery. Some of the carbon materials to be tested were modified by fluorine in order to observe the change of the electrochemical behavior due to the modification in structure and surface. The performance of the carbon materials are characterized from the point of view of capacity, cycle life, and rate of charge and discharge.

EXPERIMENTAL

To conduct the study, the electrochemical behaviors of two different kinds of carbon materials were compared whose differences and similarities in surface and structure either were well-documented or characterized. Two such carbon materials were the precursor (Amoco P100 graphitized carbon fiber) and the product (soft carbon) of a process that consists of fluorination of P-100 and defluorination of the fluorinated P-100 thus obtained.

The precursor and the products were then tested by the standard half-cell where constant current was used as the power source. The data thus obtained were then used to characterize and compare their electrochemical properties. From such study, the affects of bulk carbon structure and surface oxygen on the performance of this material as anode of lithium-ion batteries can be inferred.

Carbon Material

Amoco P-100 graphitized carbon fibers were used as the precursor of the fluorination-defluorination reaction described above. It was chosen because its synthesis and properties have been studied in great detail. Surfaces of pitch-based carbon fibers have also been studied extensively. Its reaction products after the fluorination-defluorination process have also been examined at length in the past several years (refs. 4 to 7).

The commercially purchased P-100 was first fluorinated to form $CF_{0.68}$ (refs. 7 to 9). It was then defluorinated, resulting in a new carbon material.

Depending on the environment and the temperature of this fluorination-defluorination process, a large number of different disordered carbon products can be obtained from the P-100 graphite precursor (ref. 7). In this research, the fluorination-defluorination process was designed such that the products were soft carbon, which was found to be a good material for anode in lithium-ion batteries as previously reported (ref. 3). This was accomplished by slowly heating of the $CF_{0.68}$ fibers in bromoform to 350 to 450 °C in 1 hr and kept at that temperature for 1.5 hr. The product was then stored in ambient air for some time before further treated in 1000 °C nitrogen to remove the residual fluorine and oxygen in the carbon fibers. The product was then stored in ambient air again before the electrochemical experiment.

This process removed fluorine in the graphite fluoride without causing much carbon loss. From the x-ray diffraction, the diameter and the resistivity data, it was concluded that the product was soft carbon containing disordering areas and possibly carbon branches connecting adjacent graphene layers or adjacent crystallites (refs. 6 and 7). Its physical and structural properties were compared to is precursor and are listed in table I.

This carbon material may not contain oxygen right after the 1000 °C nitrogen heating. However, after some time of ambient air storage, its energy dispersive spectra (EDS) indicated the presence of a small amount of oxygen. It is believed that some oxygen atoms from the ambient air reacted to the active sites on the surface of the carbon material and, as a result, produced an oxide surface layer.

Bismarck et al. observed that, under a similar treatment of 905 °C nitrogen heating followed by 25 °C air exposure, a basic, hydrophobic surface oxide was formed on the high tensile and high modulus carbon fibers. They also reported that thermally oxidized fibers of the same types were acidic and hydrophilic (ref. 10). It is believed that the fibers in this study had the same basic, hydrophobic surface as that described by Bismarck et al. (ref. 10).

Each sample in the form of 1 to 1.5 cm fiber strand was tied by a 0.13 mm nickel wire to a 0.5 mm nickel wire, which in turn was connected to a 3 mm nickel rod from which the constant current source was connected. Two methods were used to remove surface oxide described above: overnight (about 15 hr) vacuum heating at 180 °C immediately before assembling the half cell, and overnight nitrogen heating at 540 °C immediately before assembling the half cell. The former method was believed to degas, remove moisture and some oxygen in carbon. The latter method, on the other hand, was believed to cause carbon-oxygen reaction because of the high temperature heating at 540 °C. This removed not only the majority of the oxygen, but also some carbon.

The preparation of the carbon samples electrochemically tested in this research is summarized in figure 1.

Half Cell Test

The electrochemical properties of the carbon materials were measured in a three-electrode cell containing a counter electrode and a reference electrode of lithium metal, and a working electrode made from a carbon material. Both lithium and carbon fibers were connected to nickel rods, which served as an electrode connecting to the electrical instruments. The electrolyte used was saturated LiI in a 50/50 (vol %) mixture of ethylene carbonate and dimethyl carbonate. The half-cell was prepared in a glove box purged with argon.

During tests, the current was controlled, and the voltage measured. For the purpose of visually observing the possible interference of lithium salt, a pyrex glass weighing bottle was used as the cell, and LiI was used as the lithium salt. In this arrangement, whenever and wherever I_2 is formed in the cell during reaction, it can be identified by its color. For example, the color of the electrolyte did not change if deintercalation was stopped at 0.7 V, but gradually turned from light yellow to orange after several cycles of cell test if the "cut-off voltage" was 3.0 V.

Trial runs were made using commercially purchased crystalline graphite powder as working electrodes. The capacity for the first intercalation to 0 V was measured to be 380 mAhr/g, within experimental error of the literature value (390-420 mAhr/g) (ref. 11).

RESULTS AND DISCUSSION

Immediately after heating the soft carbon described in table I in 540 °C nitrogen to remove the surface oxides, the carbon sample was tested electrochemically in the half cell against lithium metal. Figure 2(a) is comparison of reversible (i.e., deintercalation) capacity between this sample and its precursor (P100). Their reversible capacities were about the same if the cut-off voltage of the working electrode (V_{wr}) was 0.3 V. The higher total reversible capacity for this carbon material over its precursor is mostly due to its ability to release more lithium after V_{wr} was above 0.3 V.

Figure 2(b) is the enlargement of the lower voltage portion of figure 2(a). It can be seen that the curves for both samples show plateaus at 80, 120, and 190 mV, representing different stages of lithium deintercalation.

The above data indicate that the disordering of carbon atoms due to the fluorination-defluorination treatment of a carbon precursor may not affect its ability to electrochemically intercalate and deintercalate with lithium when $0 < V_{wr} < 0.3$ V, but does create new lithium intercalation/absorption sites which are active when $V_{wr} > 0.3$ V. Such creation of lithium sites results in an increase of reversible, or useable capacity for a lithium-ion battery.

Whether these newly created lithium sites are absorption sites or intercalation sites is not known. However, they are more likely to be absorption sites since it has been reported that most lithium deintercalation takes place when $0 < V_{wr} < 0.3$ V (ref. 12).

Table II describes the intercalation and deintercalation capacities of these two samples (P100 as the precursor and soft carbon without surface oxides as the product). It is noted that for both samples, the deintercalation capacity was always smaller than the intercalation capacity. This indicates that in every charge-discharge cycle a small but significant amount of lithium was inserted into the carbon structure and lost. If these carbon materials are to be used as the anode in a lithium-ion battery, the cycle life as well as the irreversible capacity would need to be further tested. A possible reason for such lithium loss is that it reacted to some solvent that was also inserted into the carbon structure during the lithium intercalation process. To prevent such possibility, a better carbon surface may be needed as the solvent barrier.

In order to test the effects of carbon surface on its electrochemical behavior, the above experiment was slightly modified. Instead of overnight high temperature (540 °C) preheating which caused carbon-oxygen reactions and removed surface oxygen, the carbon sample was preheated to 180 °C in vacuum immediately before the half-cell preparation. The resulted intercalation and deintercalated capacities are described in table III and figure 3. It can be seen that, with oxides on their surfaces, both the precursor and the soft carbon product had very small noticeable lithium loss after several cycles of the intercalation-deintercalation process. These data suggest that carbon containing such surface oxides can have a long cycle life as the anode in lithium-ion battery.

The effect of surface oxide on the carbon's capacity to store lithium is complicated. Comparing table II and table III, it can be seen that for the carbon containing surface oxides, the precursor (P100) stored more lithium than the P-100 without carbon did, but the soft carbon product stored less.

The deintercalation curve for the carbon containing surface oxides (fig. 3) indicated that the reversible capacity of the disordered carbon was smaller than that of its precursor (P100). This is different from data obtained from the carbon without surface oxide (fig. 2). It suggests that the surface oxide can prevent lithium from being intercalated into carbon. However, this disordered carbon showed the same ability to release lithium when V_{wr} was above 0.3 V.

The above-described affects of carbon's oxide surfaces on the carbon's performance as anode of lithium-ion battery are summarized in figure 4. It appears that the surface oxides prevented the solvent from entering the carbon structure. It also prevented lithium from entering the soft carbon product, but not the P100 precursor.

The affects of surface oxide on the carbon's performance as anode in lithium-ion battery appear to be complicated and critical. Further study is needed to identify the physical and chemical properties of the surface oxide which can improve the reversible capacity and cycle life, but at the same time reduce the irreversible capacity of the carbon anode.

Experiments were also made to test the rate of lithium intercalation-deintercalation for the 180 °C vacuum heated sample as well as its precursor. In this set of experiments, carbon materials fully intercalated at 10 mA/g were deintercalated at several higher rates. And carbon materials fully deintercalated at 10 mA/g were intercalated at several higher rates. Figures 5 and 6 show, for the disordered soft carbon and its precursor, respectively, the V_{wr} as functions of the intercalated quantity at several different intercalation and deintercalation rates. It can be seen that both the disordered soft carbon and its precursor performed well at high rates of intercalation (80 mA/g) and deintercalation (2000 mA/g).

CONCLUSION

Disordering of carbon atoms in P-100 graphitized carbon fibers was accomplished. Both the P-100 precursor and the disordered soft carbon product were electrochemically tested for their performance as anode in lithium ion battery.

The disordering of the graphitized carbon fiber P-100 was made by treating the fiber with the process of fluorination and then slow defluorination in 450 °C bromoform environments, followed by 1000 °C nitrogen heating and room temperature air storage.

Immediately after 540 °C heating in nitrogen to remove the oxygen in this product, the sample was electrochemically tested and was found to have larger reversible and irreversible capacity to intercalated lithium than their precursor. Data suggests that the sample and its precursor has the same ability to intercalate lithium when $V_{wr} < 0.3V$, which is the only possible voltage range for graphite to intercalate lithium. The reason for the higher lithium storage capacity in the disordered carbon was the creation of new lithium sites which were active when $V_{wr} > 0.3$ V. If the carbon was heated in 180 °C vacuum before half cell preparation, the surface oxygen was not totally removed. In that case the surface may prevent solvent from entering the carbon structure and therefore help increasing the cycle life of the carbon when it is used as the anode material in the lithium-ion battery. The effect of surface oxide on the carbon's lithium storage capacity is not conclusive. It reduced the capacity of lithium intercalation (when $V_{wr} < 0.3V$) in the soft carbon tested in this research, but increased the capacity in its precursor (P-100). Both this sample and its precursor can have a very high rate of intercalation and deintercalation. It can intercalate at 200 mA/g, and deintercalate at a rate of 2000 mA/g without significant capacity loss.

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TABLE I.—PROPERTIES OF THE PRECURSOR AND THE PRODUCT OF THE FOLLOWING REACTIONS

$$P100 \xrightarrow[\text{Fluorination}]{} CF_{0.68} \xrightarrow[\text{Bromoform}]{450\ ^\circ C} \xrightarrow[\text{Nitrogen}]{1000\ ^\circ C} C(\text{Soft carbon})$$

	Diameter, μm	Resistivity, μΩ-cm	X-ray diffraction (002) peak		
			Position, A	Width at half maximum	Intensity
Carbon from slow defluorination of $CF_{0.68}$ in $CHBr_3$	11.3	900	3.35	1.4°	1
P100	10.0	390	3.37	0.454°	100

TABLE II.—INTERCALATION AND DEINTERCALATION CAPACITIES FOR THE CARBON PRODUCTS PRETREATED AT 540 °C NITROGEN (C1 AND C3 DESCRIBED IN FIGURE 1)

(a) C1 (Soft carbon fibers)

Cycle number	1	2	3	4	5	6
Intercalation	487	293	286	288	276	273
Deintercalation	280	274	270	268	265	260

(b) C3 (Graphitized carbon fibers)

Cycle number	1	2	3	4	5	6
Intercalation	249	226	223	224	221	221
Deintercalation	223	220	221	219	217	217

TABLE III.—INTERCALATION AND DEINTERCALATION CAPACITIES FOR THE CARBON PRODUCTS PRETREATED AT 180 °C VACUUM (C2 AND C4 DESCRIBED IN FIGURE 1)

(a) Soft Carbon Fibers

Cycle number		1	2	3	4
Intercalation	Capacity (mAHr/g)	210	213	207	197
	Current (mA/g)	10	3-6	8	10
Deintercalation	Capacity (mAHr/g)	208	208	196*	197*
	Current (mA/g)	13	10	50	30

(b) Graphitized Carbon Fibers

Cycle number		1	2	3	4
Intercalation	Capacity (mAHr/g)	257	242	245	194
	Current (mA/g)	10	28	10	200
Deintercalation	Capacity (mAHr/g)	245	241	244	194
	Current (mA/g)	10	40	9	40

TABLE IV.—INTERCALATION AND DEINTERCALATION CAPACITIES AT HIGH CURRENT DENSITIES FOR CARBON PRODUCTS PRETREATED AT 180 °C VACUUM (C2 AND C4 DESCRIBED IN FIGURE 1)

(a) C2 (Soft Carbon Fibers)

Intercalation	Capacity (mAHr/g)	197	190	177	161	105
	Current (mA/g)	10	50	80	200	350
Deintercalation	Capacity (mAHr/g)	197	196	--	184	163
	Current (mA/g)	30	50	--	500	2000

(b) C4 (Graphitized Carbon Fibers)

Intercalation	Capacity (mAHr/g)	245	232	194	79
	Current (mA/g)	10	80	200	350
Deintercalation	Capacity (mAHr/g)	244	241	235	232
	Current (mA/g)	9.1	40	500	2000

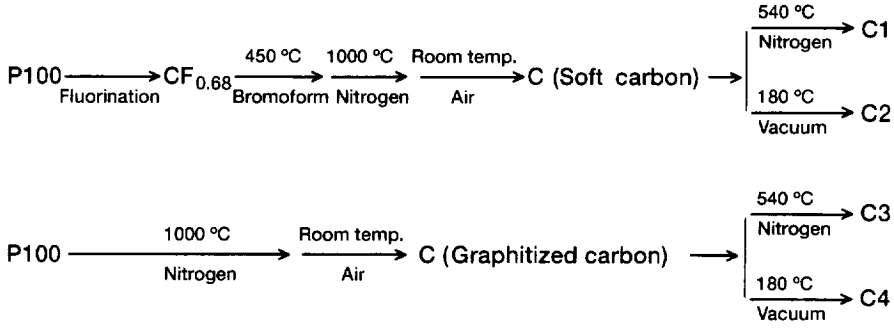


Figure 1.—Preparation of carbon samples electrochemically tested in this study.

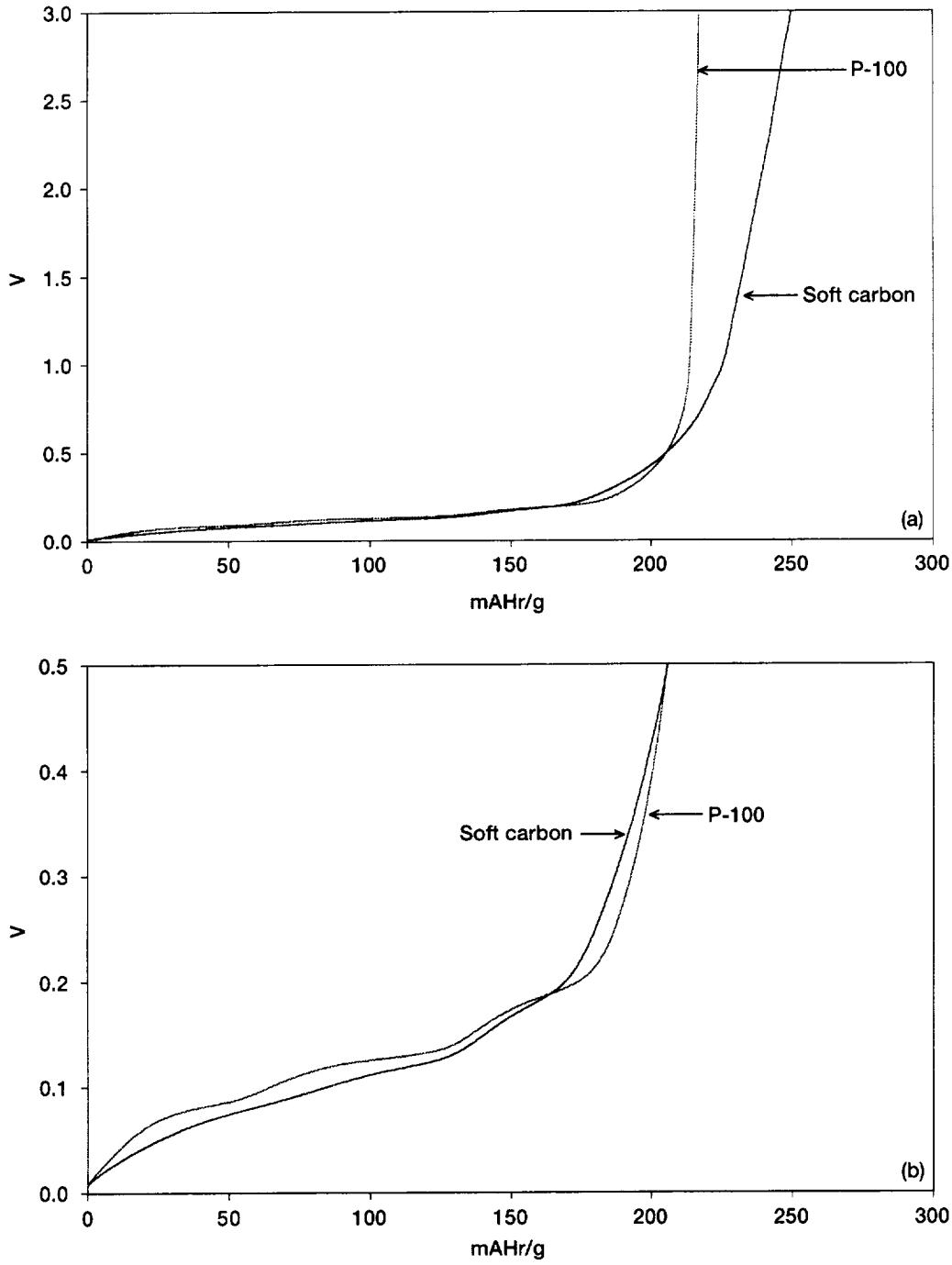
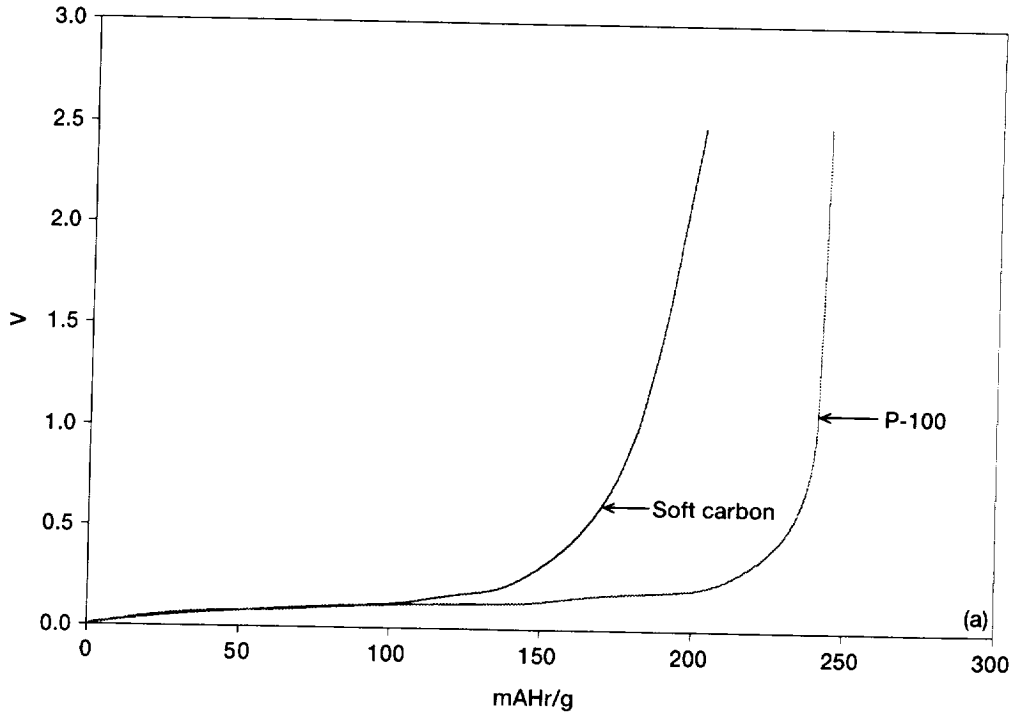


Figure 2.—Electrochemical deintercalation of lithium from carbon pretreated at 540 °C nitrogen (C1 (soft carbon) and C3 (P-100) described in Figure 1). (a) The entire voltage range (0 to 3 V). (b) The lower voltage range only (<0.5V). The half cell (C/saturated LiI-50/50 (vol %) EC and DMC/Li) was operated at 10 mA/g. The plateaus indicate deintercalation.



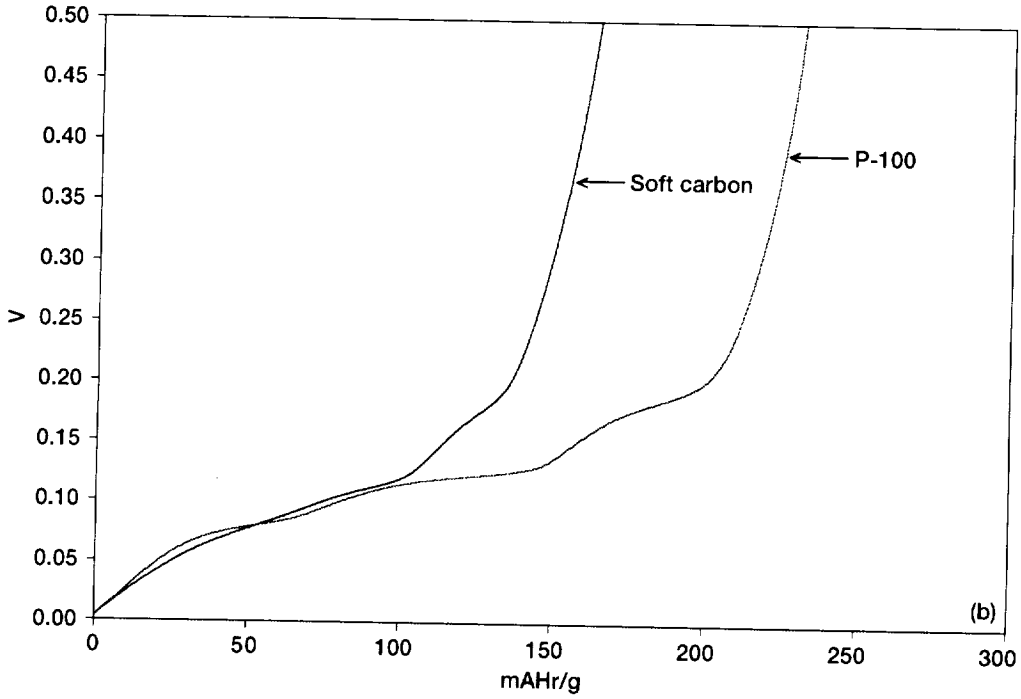


Figure 3.—Electrochemical deintercalation of lithium from carbon pretreated at 180 °C vacuum (C2 (soft carbon) and C4 (P-100) described in Figure 1). (a) The entire voltage range (0 to 3 V). (b) the lower voltage range only (<0.5V). The half cell (C/saturated LiI-50/50 (vol %) EC and DMC/Li) was operated at 10 mA/g. The plateaus indicate deintercalation.

	P100	Slowly defluorinated $CF_{0.68}$
Capacity	↓	↑
Cycle life	↓	↓

Figure 4.—Effect of removing surface oxide (C and O) on the performance of carbon materials as anode in lithium-ion battery.

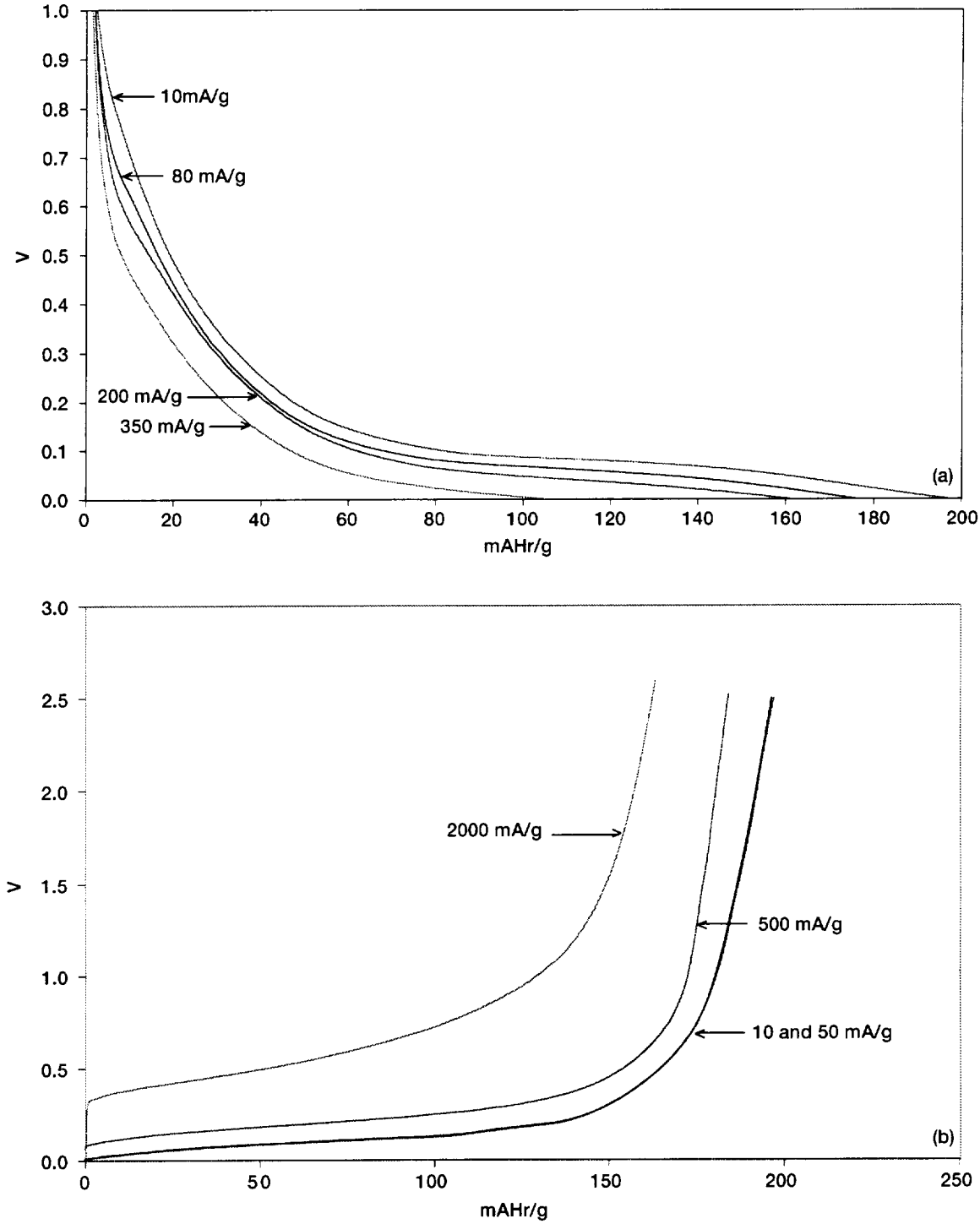


Figure 5.—Electrochemical behavior as functions of current density in a C/saturated LiI-50/50 (vol %) EC and DMC/ Li half cell. The carbon was a soft carbon pretreated at 180 °C vacuum. (C2 described in Figure 1). (a) intercalation of a slowy (10mA/g) deintercalated carbon. (b) deintercalation of a slowy (10mA/g) intercalated carbon.

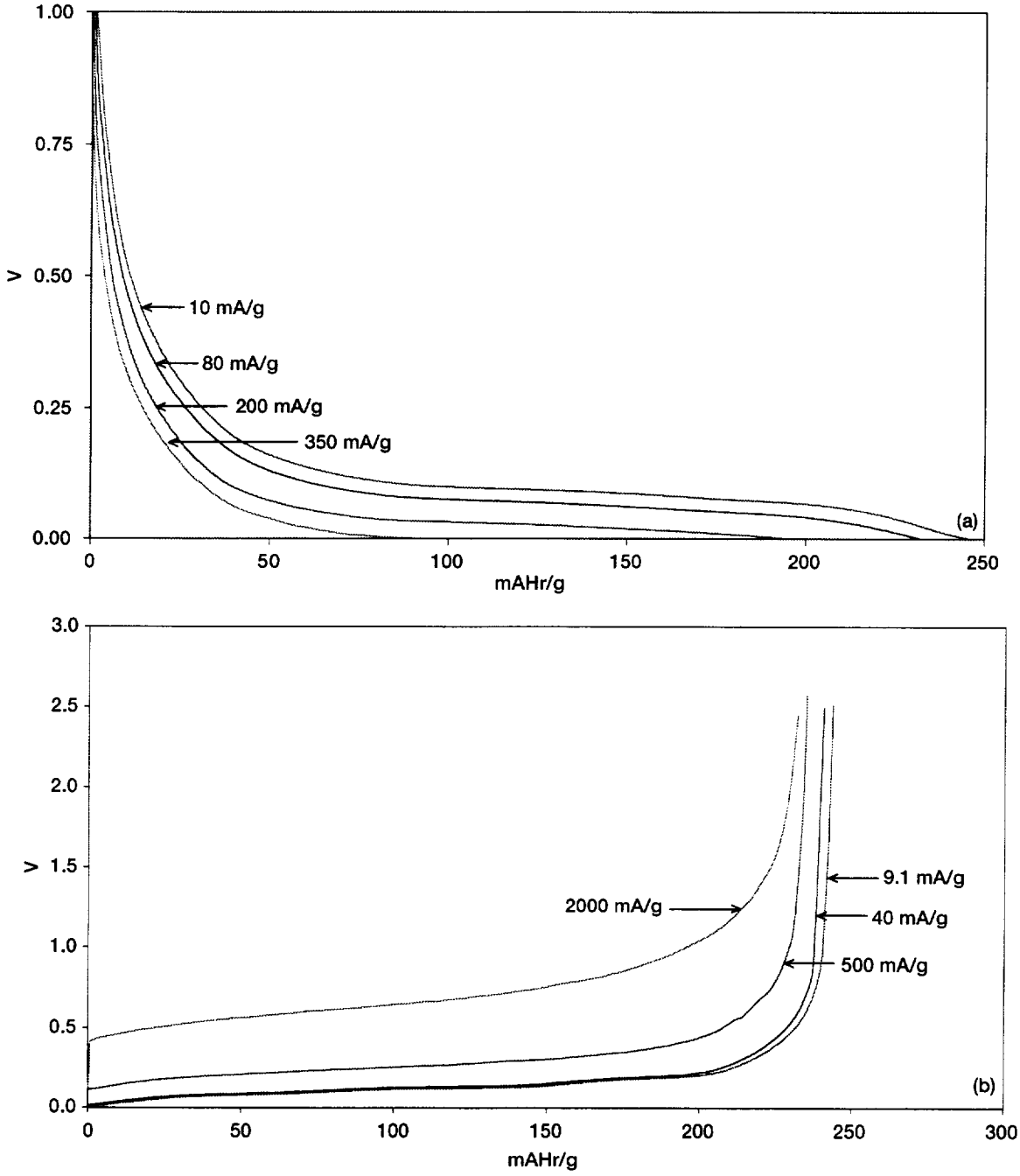


Figure 6.—Electrochemical behavior as functions of current density in a C/saturated LiI-50/50 (vol %) EC and DMC/ Li half cell. The carbon was P-100 pretreated at 180 °C vacuum. (C4 described in Figure 1). (a) intercalation of a slowy (10mA/g) deintercalated carbon. (b) deintercalation of a slowy (10mA/g) intercalated carbon.

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13. ABSTRACT ***(Maximum 200 words)***

The effects of carbon structure and surface oxygen on the carbon's performance as the anode in lithium-ion battery were studied. Two carbon materials were used for the electrochemical tests: soft carbon made from defluorination of graphite fluoride, and the carbon precursor from which the graphite fluoride was made. In this research the precursor was graphitized carbon fiber P-100. It was first fluorinated to form $CF_{(1.08)}$, then defluorinated slowly at 350 to 450 °C in bromoform, and finally heated in 1000 °C nitrogen before exposed to room temperature air, producing disordered soft carbon having basic surface oxides. This process caused very little carbon loss. The electrochemical test involved cycles of lithium intercalation and deintercalation using C/saturated LiI-50/50 (vol %) EC and DMC/Li half cell. The cycling test had four major results. (1) The presence of a basic oxide surface may prevent solvent from entering the carbon structure and therefore prolong the carbon's cycle life for lithium intercalation-deintercalation. (2) The disordered soft carbon can store lithium through two different mechanisms. One of them is lithium intercalation, which gives the disordered carbon an electrochemical behavior similar to its more ordered graphitic precursor. The other is unknown in its chemistry, but is responsible for the high-voltage portion (>0.3V) of the charge-discharge curve. (3) Under certain conditions, the disordered carbon can store more lithium than its precursor. (4) These sample and its precursor can intercalate at 200 mA/g, and deintercalate at a rate of 2000 mA/g without significant capacity loss.

14. SUBJECT TERMS

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