

Supporting Information for

Structural evolution by heat treatment of soft and hard carbons as Li storage materials. A joint NMR/XRD/TEM/Raman study.

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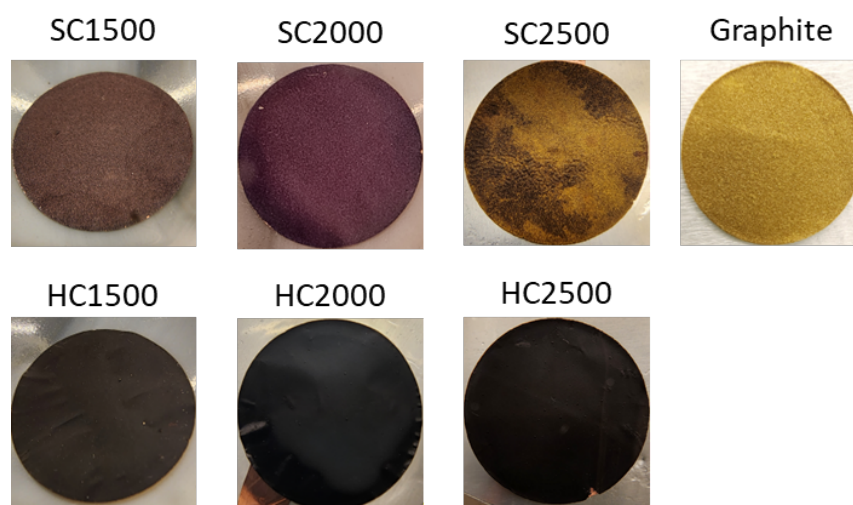


Fig. S1. Color of fully lithiated carbon electrodes.

Table S1. Elemental composition obtained from EDX analysis.

	Elemental composition (EDX)	
	C / wt%	O / wt%
SCA	99.9	0.14
SC2500	99.9	0.045
Graphite	99.9	0.051
HCA	99.9	0.11
HC2500	99.9	0.067

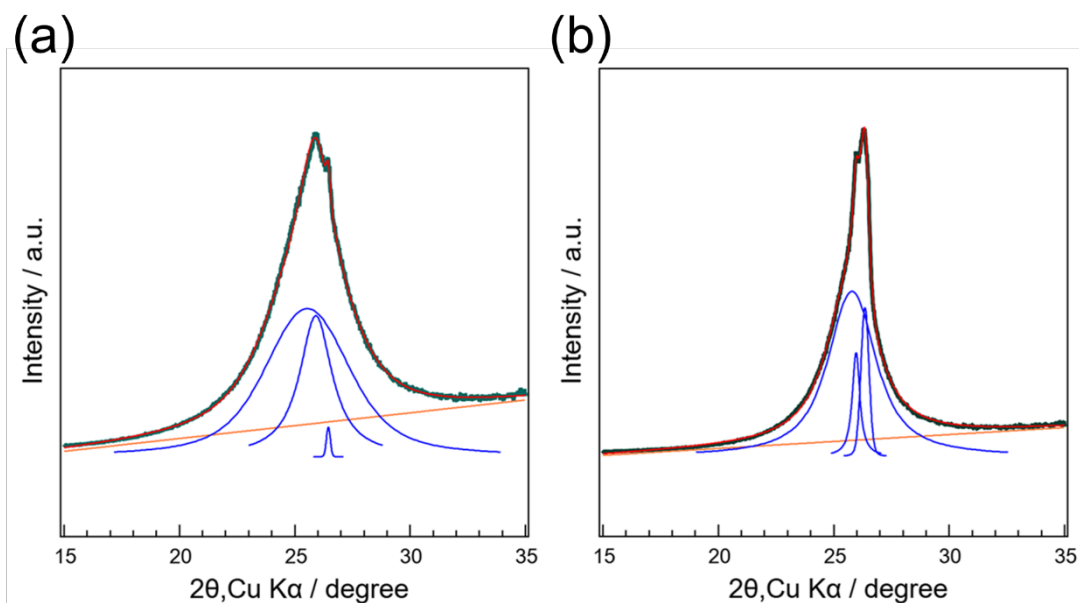


Fig. S2 Deconvolution of (002) X-ray diffraction peaks of (a) HC2000 and (b) HC2500 (green lines: experimental, orange solid lines: background, blue solid lines: components, red solid lines: components summed).

Table S2. Numerical analysis results of XRD profiles and the Raman spectra of carbon samples.

	2θ / degree (002)	d ₀₀₂ / nm	Lc (002) / nm	Layer numbers	La (110) / nm	A _G /A _{D1}
HCA	25.22	0.353	1.2	3.4	-	0.31
HC1500	25.46	0.350	1.6	4.5	-	0.35
	25.55	0.349	1.9	5.4	-	
HC2000	25.91	0.344	4.7	14	-	0.61
	26.46	0.337	41	121	-	
	25.79	0.345	3.3	9.5	-	
HC2500	25.97	0.343	21	60	-	1.50
	26.35	0.338	20	58	-	
SCA	25.83	0.345	2.9	8.4	-	0.43
SC1500	25.88	0.344	5.9	17	-	0.98
SC2000	26.06	0.342	22	66	14	1.64
SC2500	26.42	0.337	34	101	52	4.73
Graphite	26.48	0.337	35	105	51	1.66

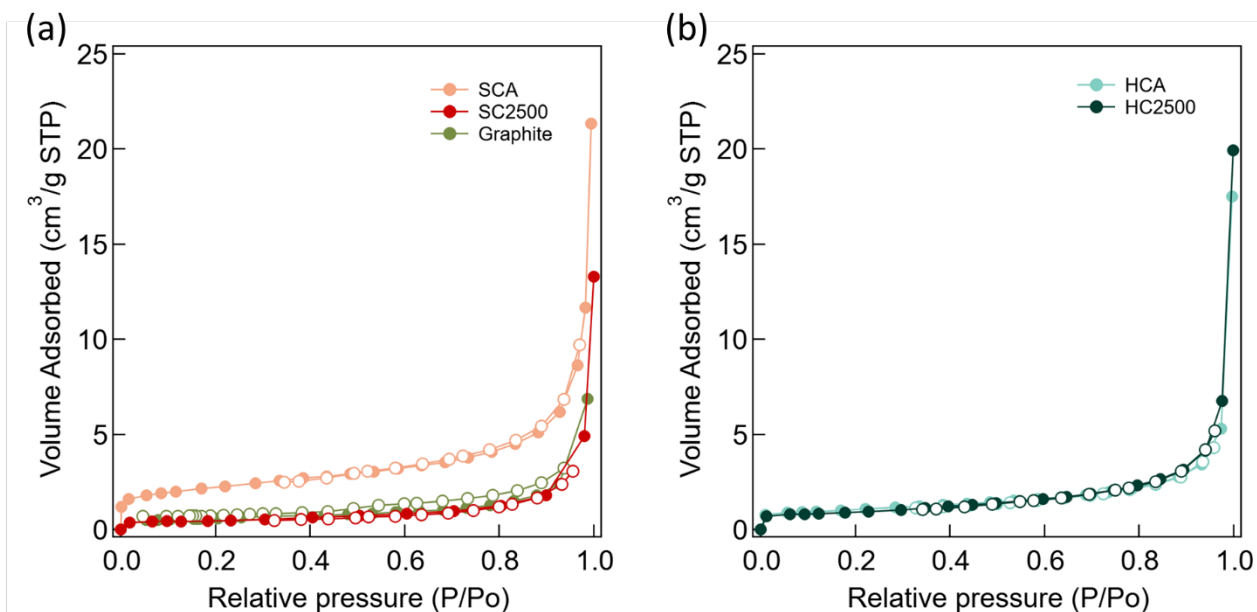


Fig. S3. N₂ adsorption (closed marks) and desorption (open marks) isotherms at 77K on (a) soft carbons and graphite, and (b) hard carbons.

Table S3. The lithiation capacity and mass loading of active material for each carbon electrode, numbers extracted from the observations presented in Fig. 4.

	Capacity / mAh g ⁻¹	Active material mass / mg
SCA	311	8.46
SC1500	243	5.49
SC2000	198	8.73
SC2500	479	6.84
Graphite	386	7.38
HCA	408	9.72
HC1500	179	7.56
HC2000	152	8.55
HC2500	177	7.02

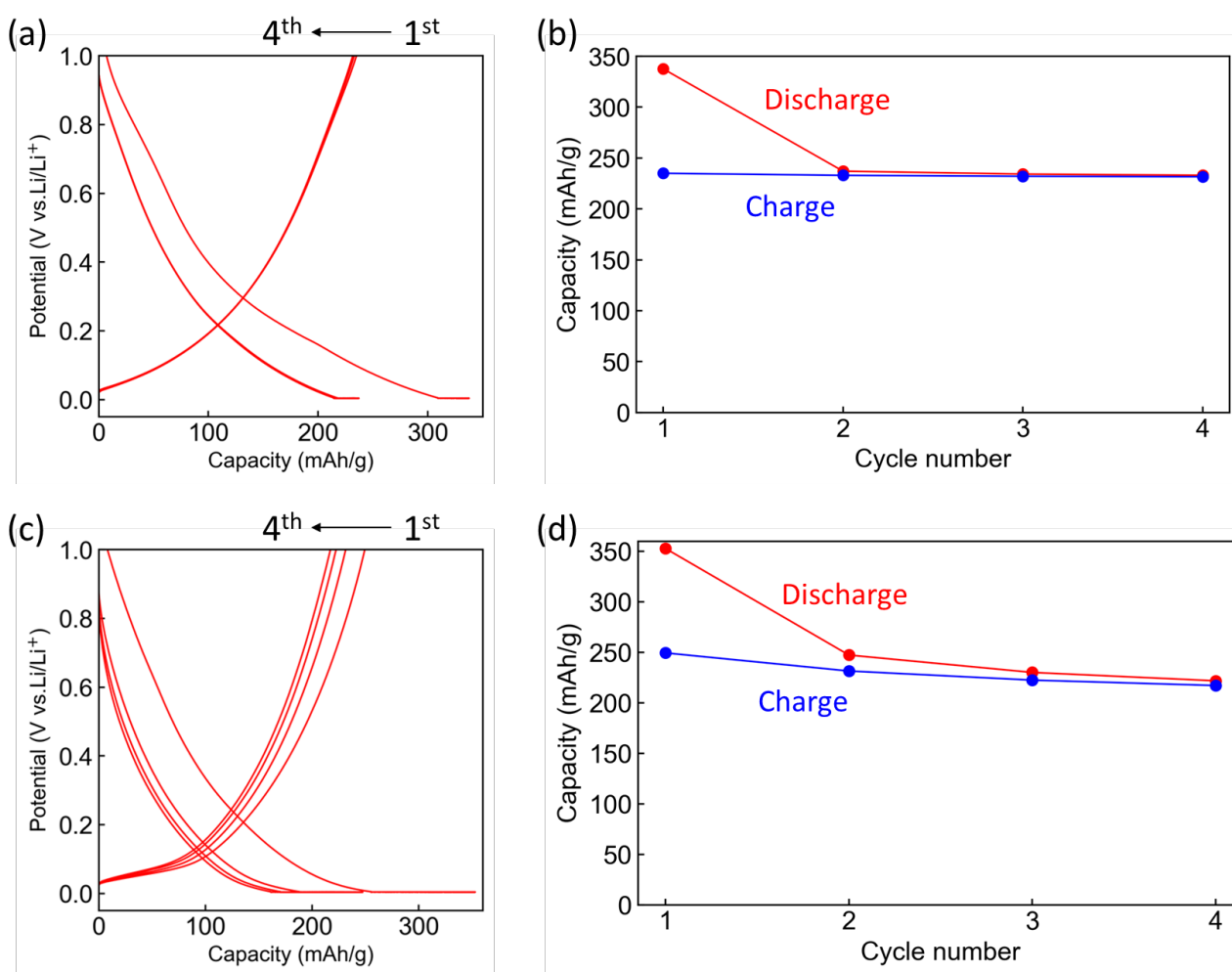


Fig. S4. Charge/discharge curves of (a) SCA, and (c) HCA and cycling performance of (b) SCA, and (d) HCA. The charge process was performed at constant current (100 mA/g, ~ 1.0 V vs. Li/Li⁺), while the discharge process was conducted at constant current (100 mA/g, ~ 0.005 V vs. Li/Li⁺) followed by constant voltage (0.005 V vs. Li/Li⁺, cut off current density: 10 mA/g).

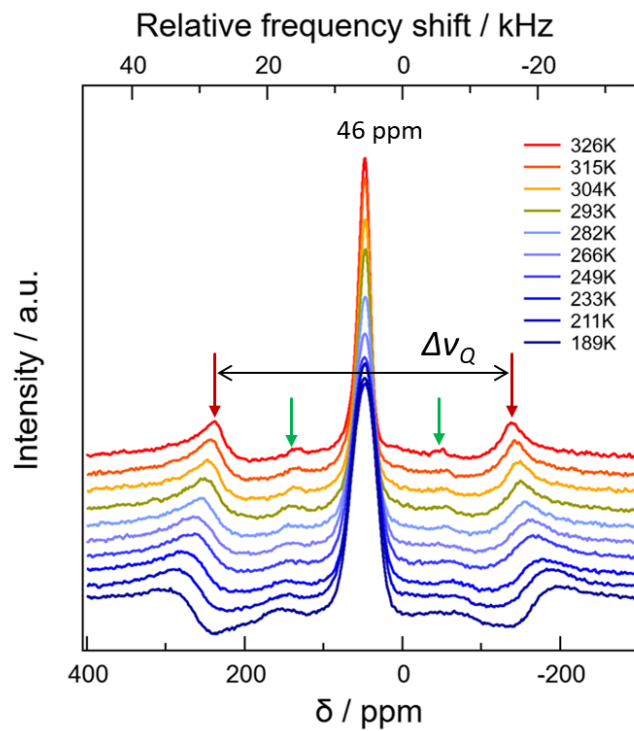


Fig. S5. Temperature-dependent ^7Li NMR spectra of fully lithiated graphite (LiC_6).

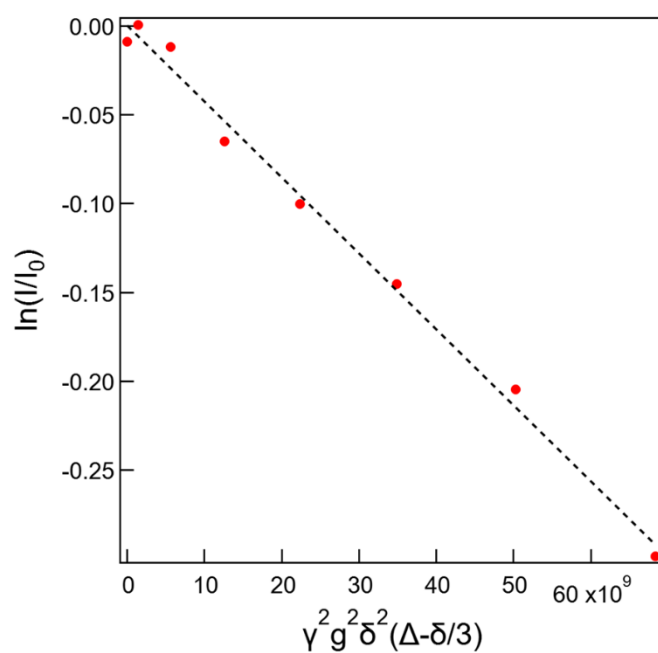


Fig. S6. The diffusional decay of the ^7Li NMR spectra of SCA at 343 K; the dashed line is the fit of the Stejskal-Tanner expression¹ to the data.

Table S4. The ^7Li spin-lattice relaxation time (T_1 / s) of fully lithiated carbon samples measured in the temperature region 189-326K.

Temperature (K)	HCA ($\delta=24\sim64$ ppm)	SCA ($\delta=23$ ppm)	HC2500 ($\delta=17$ ppm)	HC2500 ($\delta=47$ ppm)	SC2500 ($\delta=13$ ppm)	SC2500 ($\delta=49$ ppm)	Graphite ($\delta=46$ ppm)
189	0.826	2.31	2.66	-	-	3.32	3.09
211	0.713	1.46	2.41	-	-	3.01	2.84
216	0.688	-	-	-	-	-	-
233	0.569	1.07	1.98	2.27	-	2.58	2.54
249	0.439	0.743	1.38	1.73	-	2.41	2.43
266	0.374	0.562	0.908	1.41	2.23	2.05	2.15
282	0.270	0.429	0.701	1.21	1.92	1.94	2.07
293	0.188	0.321	0.589	0.872	1.53	1.81	1.84
304	0.180	0.277	0.493	0.943	1.41	1.77	1.83
315	0.150	0.230	0.479	0.749	1.27	1.62	1.88
326	0.142	0.200	0.430	0.702	0.931	1.69	1.68

References

- 1 E. O. Stejskal and J. E. Tanner, *J. Chem. Phys.*, 1965, **42**, 288–292.