Electronic Supplementary Information

NIOBIUM CARBIDE NANOFIBERS AS A VERSATILE PRECURSOR FOR HIGH POWER SUPERCAPACITOR AND HIGH ENERGY BATTERY ELECTRODES

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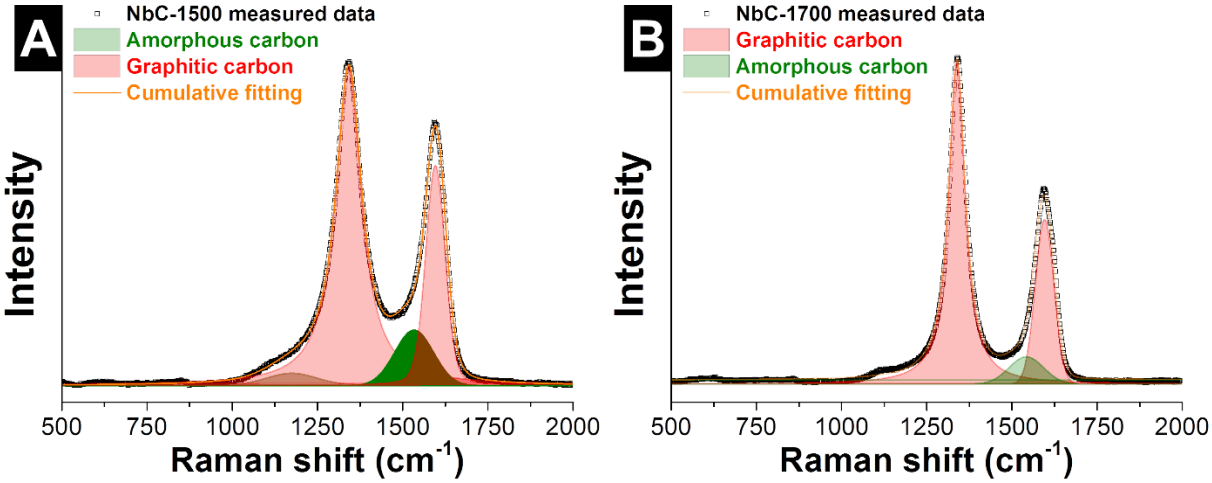

Figure S1: Peak fitting (four Voigt peaks) results for Raman spectra of (A) NbC/C composite fibers synthesized at 1500 °C and (B) 1700 °C.

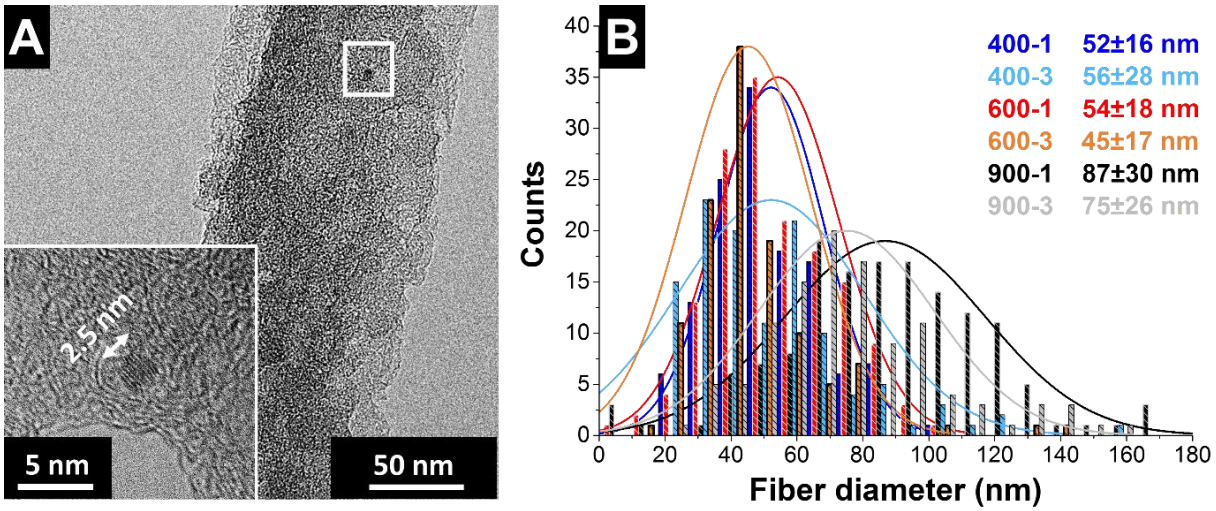

Figure S2: (A) TEM micrograph of NbC-CDC fibers synthesized at 400 °C and 3 h, where some residual NbC crystals are observed in the inset. (B) Fiber diameter distribution of NbC-CDC nanofibers with different synthesis temperatures (400, 600, or 900 °C) and chlorine gas treatment durations (1 h or 3 h).

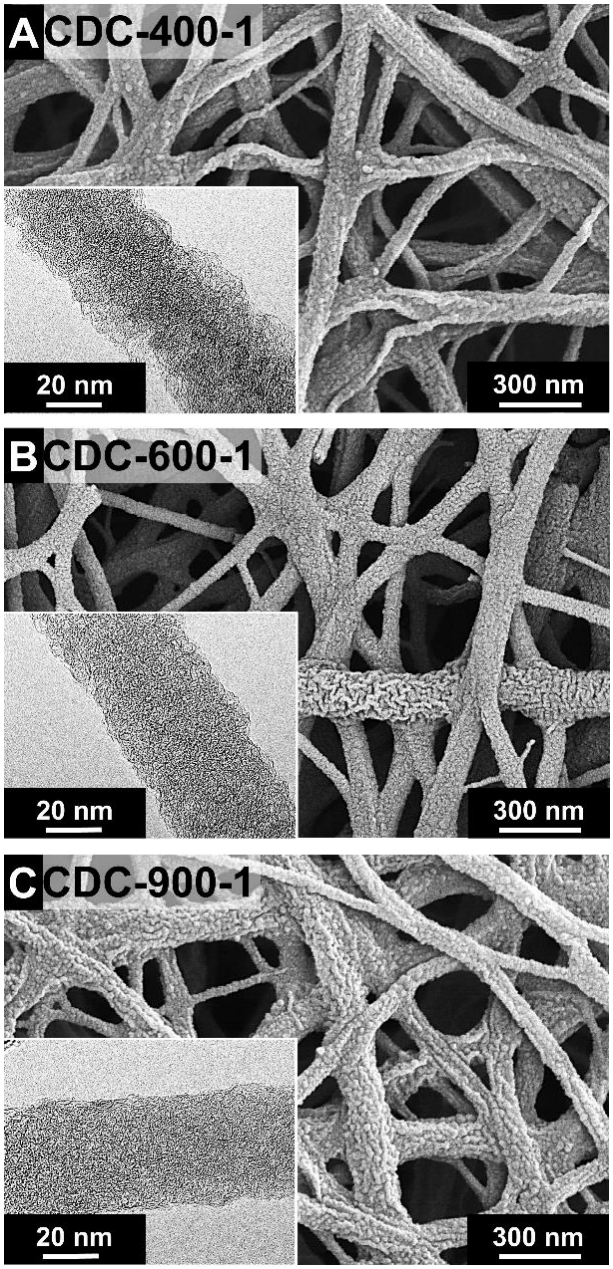

Figure S3: SEM and TEM micrographs (inset) of NbC-CDC fibers synthesized by chlorine gas treatment for 1 h at 400 °C (A), 600 °C (B) and 900 °C (C).

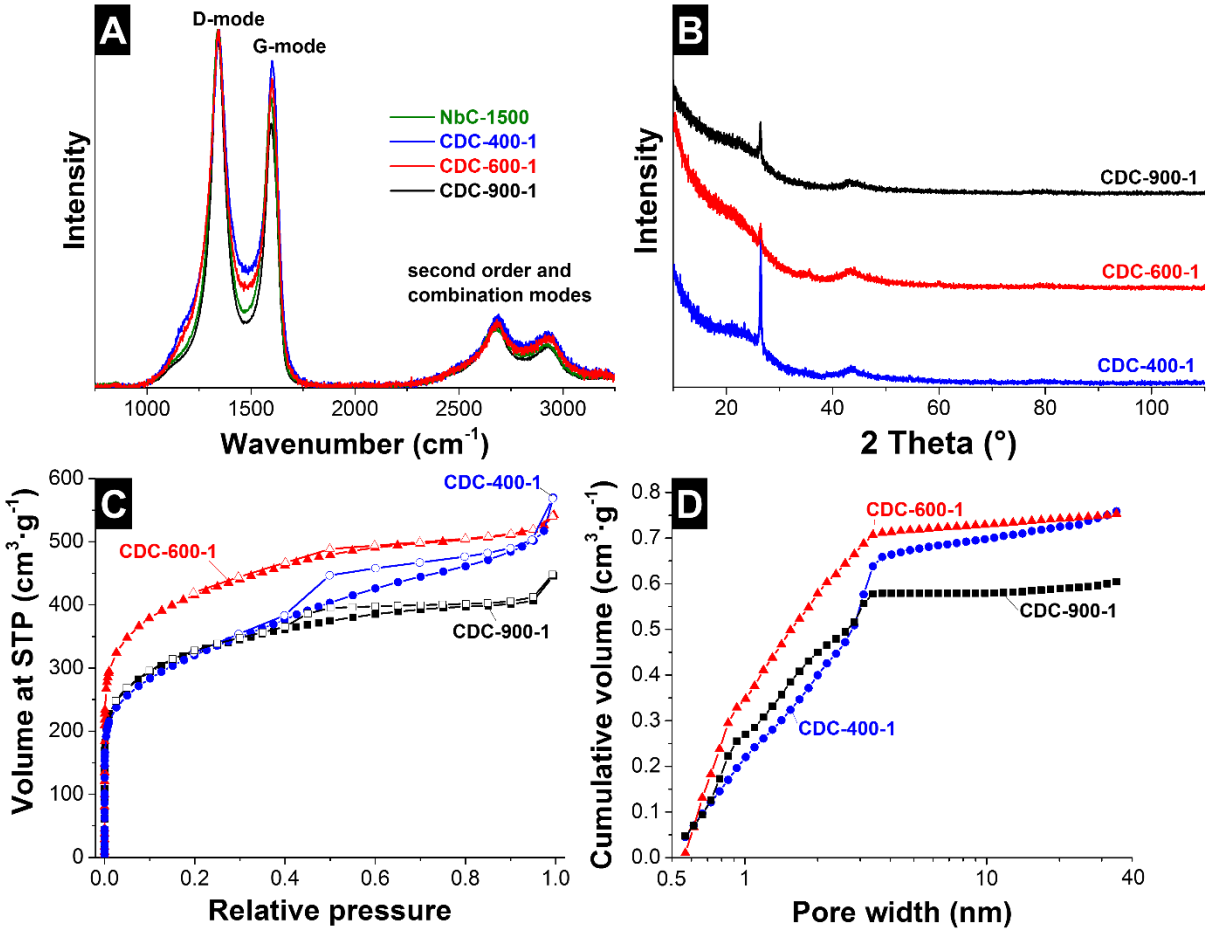

Figure S4: Structural characterization of NbC-CDC fibers synthetized by chlorine treatment for 1 h: (A) Raman spectra and (B) XRD diffractograms. (C) Nitrogen gas sorption isotherms at -196 °C (STP: standard temperature pressure, and (D) corresponding cumulative pore volume distribution obtained from QSDFT data analysis.

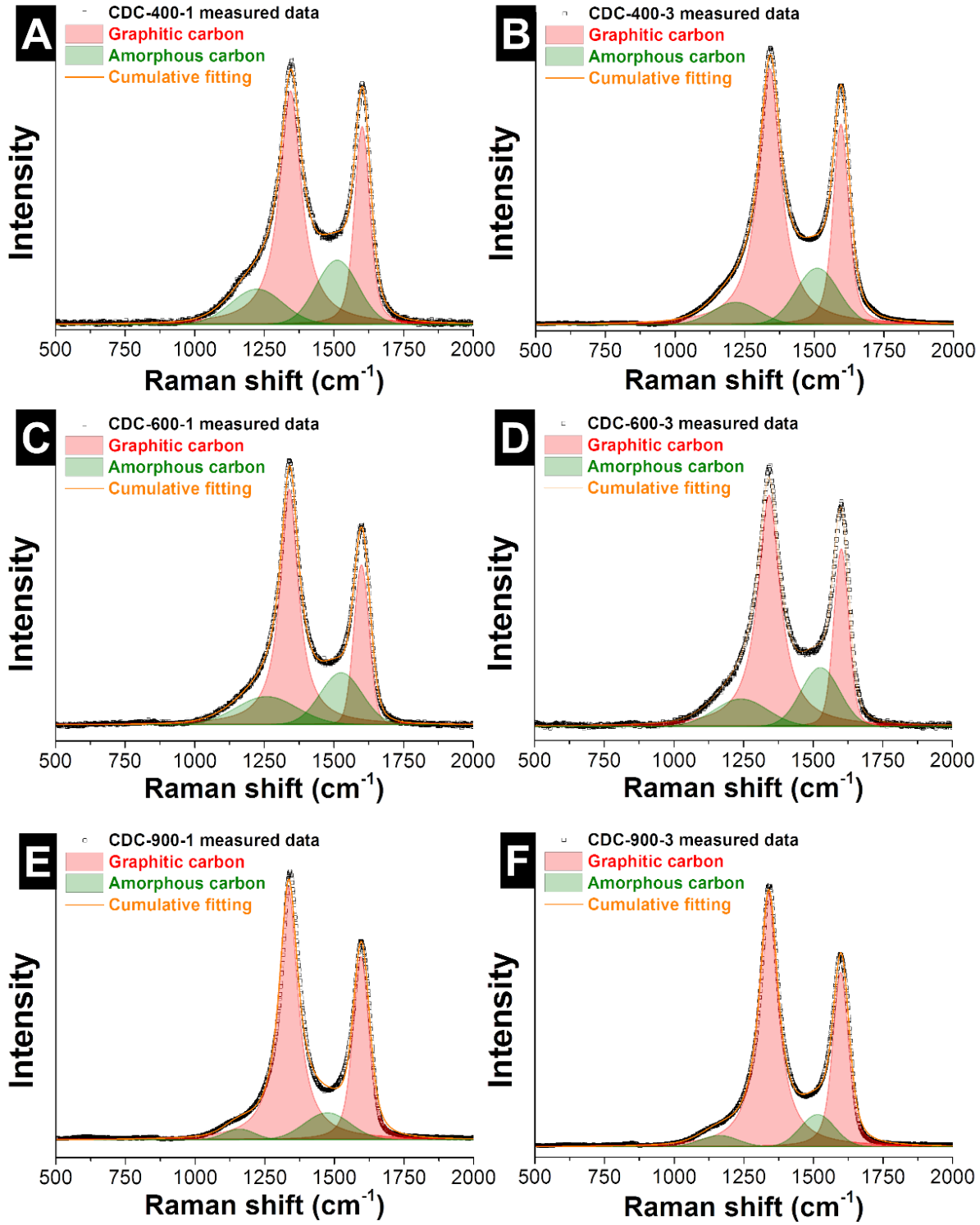

Figure S5: Results of four Voigt peak fitting for Raman spectra of NbC-CDC fibers synthesized. Samples after thermal chlorine gas treatment at (A-B) 400 °C, (C-D) 600 °C, and (E-F) 900 °C for (A,C,E) 1 h or (B,D,F) 3 h.

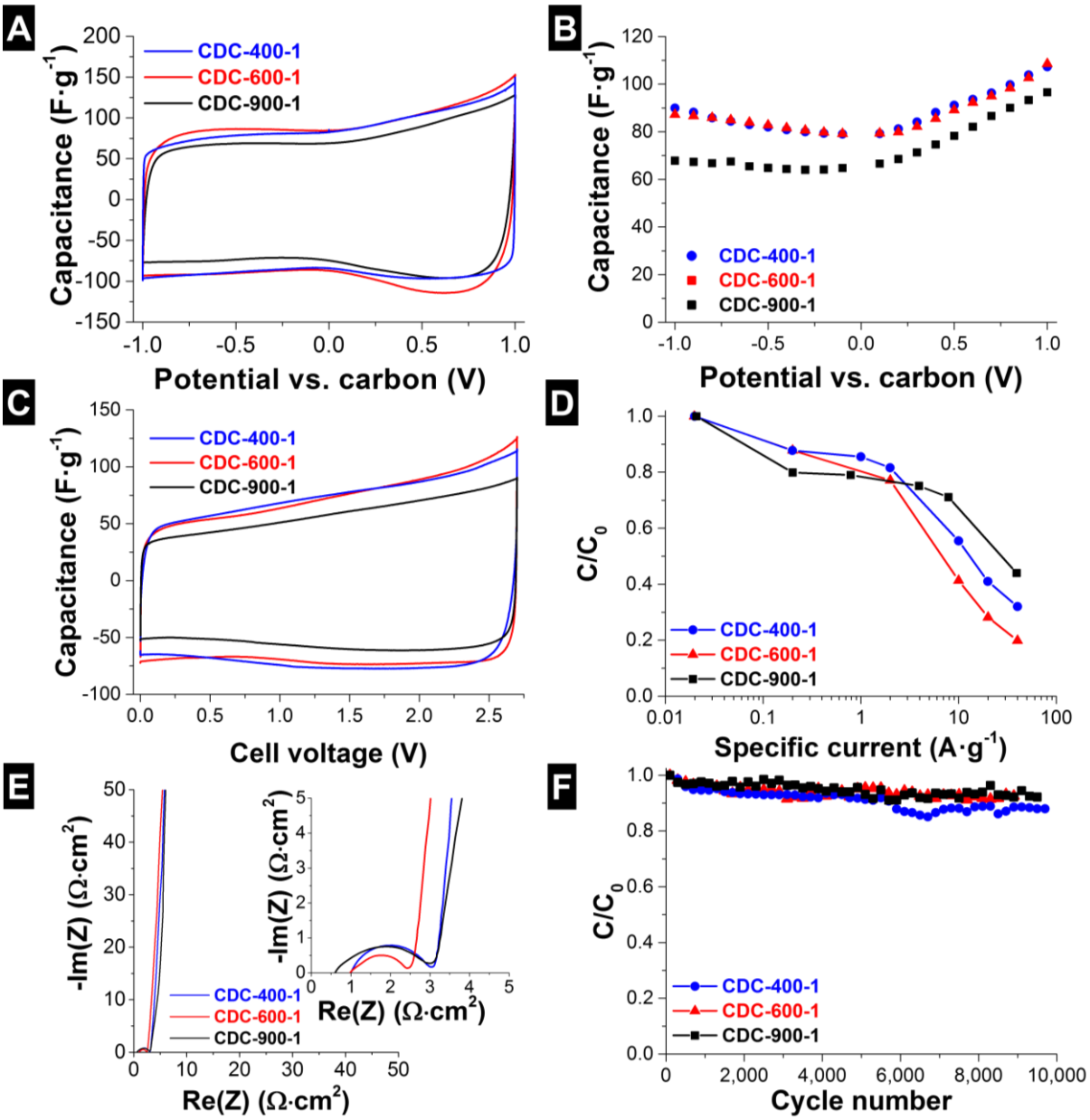

Figure S6: Electrochemical characterization of chlorine treated samples for 1 h in 1 M TEA-BF_4 in ACN. Half-cell measurements with (A) cyclic voltammetry at 10 mV·s^{-1} and (B) GCPL at 2 A·g^{-1}. Characterization for full-cell with (C) cyclic voltammetry at 10 mV·s^{-1}, (D) galvanostatic charge/discharge power handling up to 2.5 V cell voltage between 0.02-40 A·g^{-1}, (E) EIS at 0 V, and (F) galvanostatic charge/discharge cycling stability up to 2.5 V at 1 A·g^{-1} for 10,000 cycles.

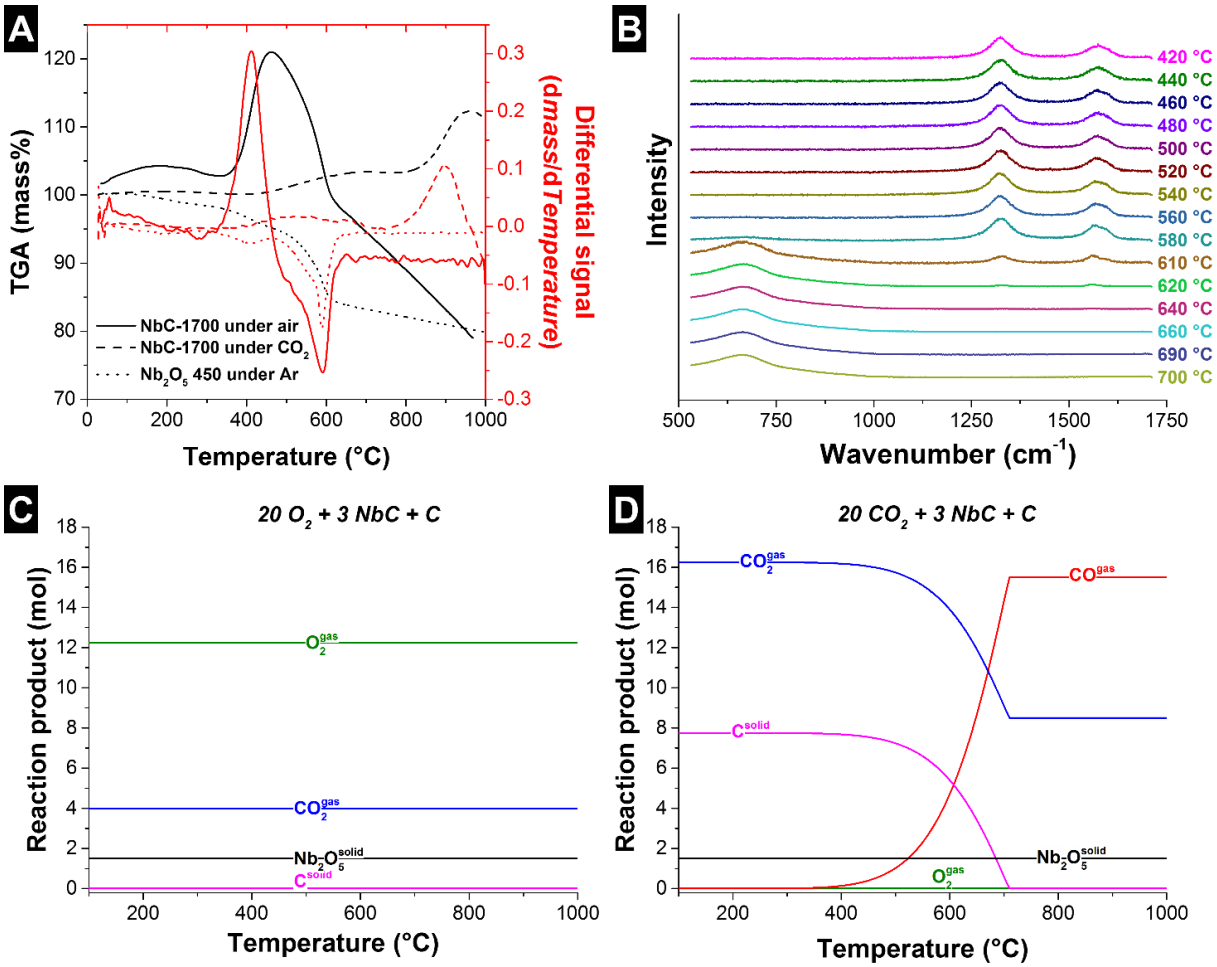

Figure S7: (A) TGA and DTG of NbC-1700 fibers under air and CO_2 at 5 °C·min^{-1}, and TGA and DTG of Nb_2O_5-450 fibers under Argon. (B) In situ Raman spectra of NbC-1700 fibers under treatment in air at 5 °C·min^{-1}. Thermodynamic calculations with FACTSAGE of reactions products from NbC/C (C) in air (simplified as oxygen) and (D) in CO_2.

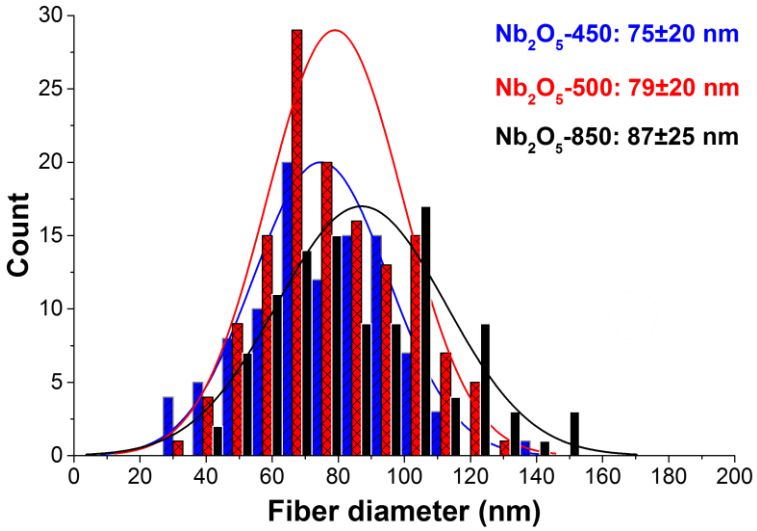

Figure S8: Fiber diameter distribution of Nb_2O_5/C hybrid fibers.

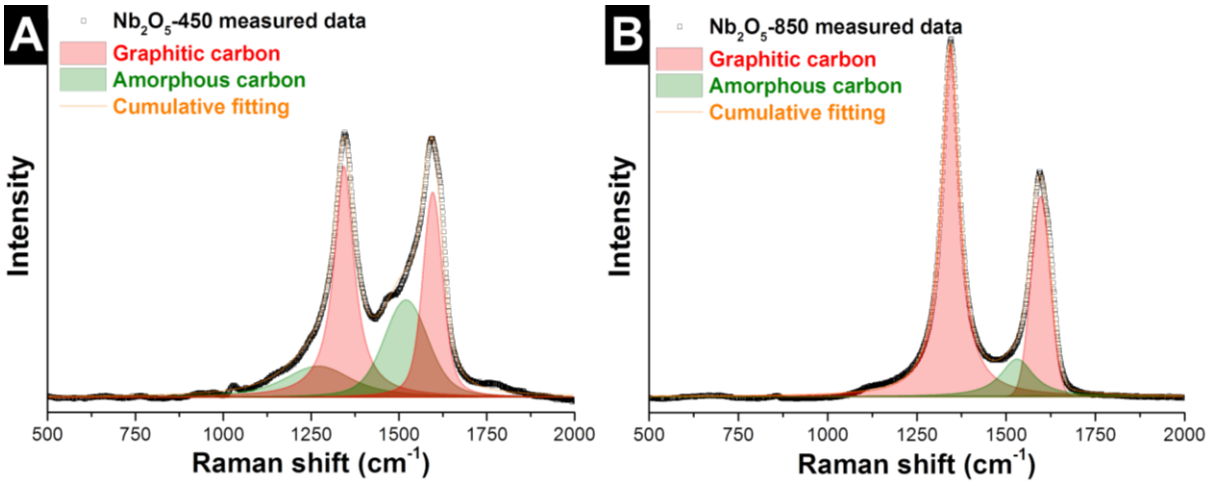

Figure S9: Four Voigt peak fitting results of Raman spectra of (A) Nb_2O_5-450 and (B) Nb_2O_5-850.

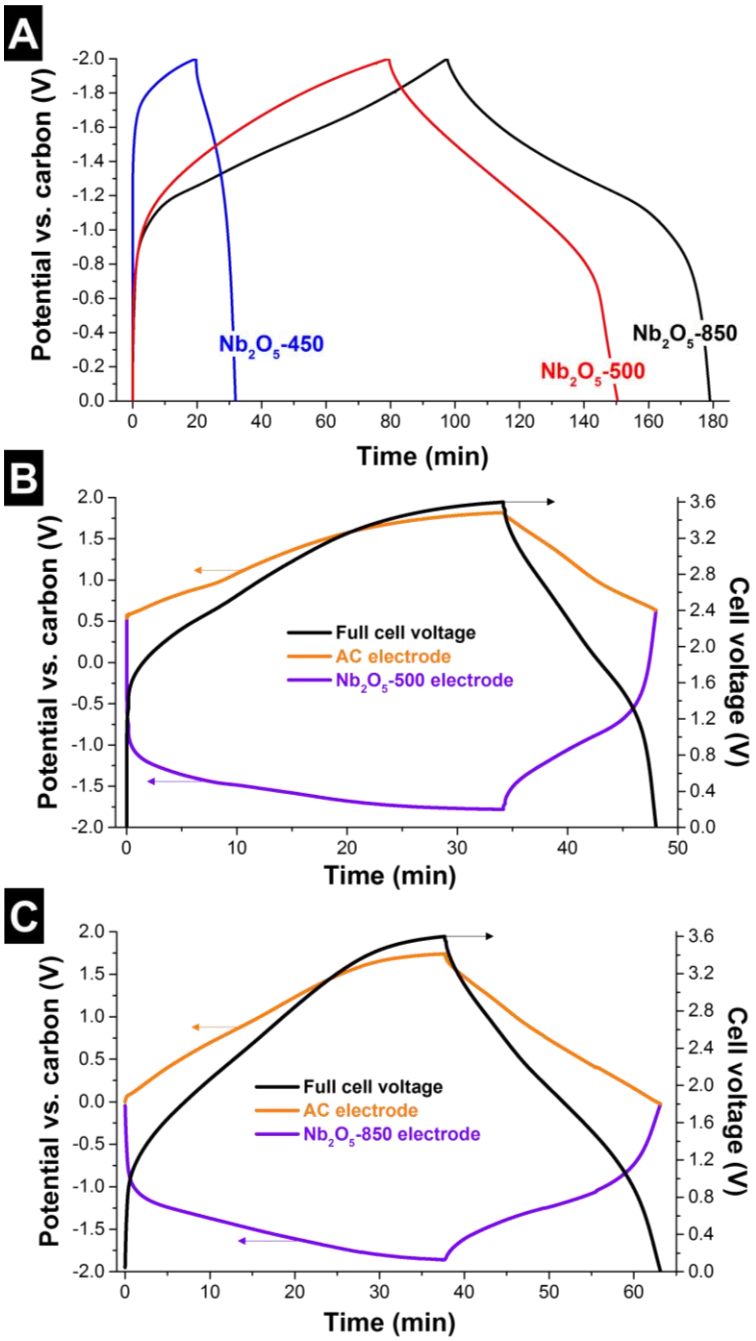

Figure S10: (A) Electrochemical characterization of Nb_2O_5/C hybrid fibers for half-cell configuration by GCPL at 0.1 $A \cdot g^{-1}$ in $LiClO_4$ 1 M in EC/DMC (B) Full-cell measurements with a spectator reference electrode to monitor the potential development at the positive and negative electrode separately. The cell voltage is increased to 3.6 V at 0.1 $A \cdot g^{-1}$, while observing the potential of the negative electrode (Nb_2O_5 electrode) and the positive electrode (AC electrode) through an AC reference electrode.

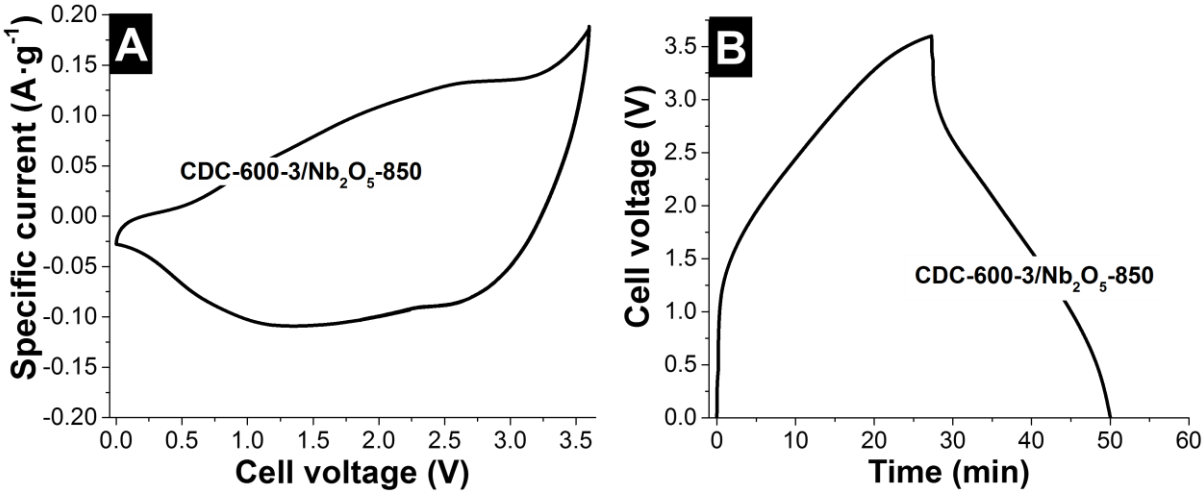

Figure S11: (A) Electrochemical characterization of CDC-600-3/Nb_2O_5-850 hybrid full-cell configuration by cyclic voltammetry at 1 mV·s^{-1}, (B) and by GCPL to 3.6 V cell voltage at 0.1 A·g^{-1} in $LiClO_4$ 1 M in EC/DMC. The cell used a Nb_2O_5-850 negative electrode and a CDC-600-3 positive electrode.

Table S1: EDX results of NbC-CDCs fibers and measured mass change after chlorine gas treatment.

Sample	Mass change	C (mass%)	O (mass%)	Nb (mass%)	Cl (mass%)
NbC-1500	-	24.8±0.6	2.3±0.5	73.9±0.9	
CDC-400-1	-75%	96.8±0.2	2.8±0.2	0.2±0.0	0.2±0.0
CDC-400-3	-78%	96.1±0.2	2.6±0.2	0.6±0.1	0.7±0.1
CDC-600-1	-74%	98.6±0.2	0.8±0.3	0.1±0.0	0.5±0.1
CDC-600-3	-74%	97.9±0.3	1.7±0.2	0.2±0.0	0.2±0.1
CDC-900-1	-79%	98.0±0.3	1.2±0.1	0.1±0.0	0.7±0.1
CDC-900-3	-72%	98.3±0.4	0.9±0.2	0.3±0.0	0.5±0.1

Table S2: Peak fitting of Raman spectra from NbC-CDCs fibers.

Sample	D-mode (cm^{-1})	G-mode (cm^{-1})	I_D/I_G-ratio	FWHM (cm^{-1}) D-mode	FWHM (cm^{-1}) G-mode
NbC-1500	1342	1598	2.1	80.2±0.6	68.0±0.8
CDC-400-1	1345	1603	2.1	96.2±0.8	66.0±0.7
CDC-400-3	1343	1603	2.3	98.4±0.6	67.0±1.0
CDC-600-1	1341	1601	2.7	90.2±3.6	65.2±0.9
CDC-600-3	1340	1597	2.6	87.1±6.7	64.2±0.9
CDC-900-1	1335	1597	2.0	82.8±3.6	69.9±1.4
CDC-900-3	1335	1596	2.1	81.6±0.4	68.2±0.7

Table S3: Peak fitting of Raman spectra from Nb_2O_5/C fibers.

Sample	D-mode (cm^{-1})	G-mode (cm^{-1})	I_D/I_G-ratio	FWHM (cm^{-1}) D-mode	FWHM (cm^{-1}) G-mode
NbC-1700	1340	1598	1.9	59.2±0.1	65.4±0.0
Nb_2O_5-450	1346	1599	1.5	69.7±6.5	67.2±1.2
Nb_2O_5-500	1354	1584	-	-	-
Nb_2O_5-850	1346	1599	2.5	60.9±0.2	64.1±0.9