# Raw data files for Synthesis of Hard Carbon-TiN/TiC Composites by Reacting Cellulose with $TiCl_4$ Followed by Carbothermal Nitridation/Reduction

Dataset DOI: 10.5258/SOTON/D0892

ReadMe Author: Andrew Hector and Hang Cheng, University of Southampton

Paper Authors: Hang Cheng, Nuria Garcia-Araez, Andrew L. Hector and Samantha Soulé

Journal: Inorganic Chemistry

Paper DOI:10.1021/acs.inorgchem.9b00116.

This dataset contains: Raw data used to produce figures in the manuscript and the supplementray information.

The figures and related data sets are as follows:

**Fig 3 The XRD patterns of HC-TiN (a) and HC-TiC (b) composites**.

1-1 XRD patterns data for HC-TiN composites (degrees 2θ then counts in each pair of columns).

1-2 XRD patterns data for HC-TiC composites (degrees 2θ then counts in each pair of columns).

**Fig 5 Raman spectra of HC-TiN (a) and HC-TiC (b) composites.**

2-1 Raman spectra data for HC-TiN composite (wavenumber $cm^{-1}$ then counts in each pair of columns).

2-2 Raman spectra data for HC-TiC composites (wavenumber $cm^{-1}$ then counts in each pair of columns).

**Fig 6 High resolution XPS spectra of the C 1s, O 1s, N 1s and Ti 2p regions for HC-TiN and HC-TiC composites with 15.15 *wt%* TiN and 8.65 *wt%* TiC, respectively.**

3-1 C 1s spectra data for HC-TiN composites (binding energy eV then counts for data followed by fit components).

3-2 C 1s spectra data for HC-TiC composites (binding energy eV then counts for data followed by fit components).

3-3 O 1s spectra data for HC-TiN composites (binding energy eV then counts for data followed by fit components).

3-4 O 1s spectra data for HC-TiC composites (binding energy eV then counts for data followed by fit components).

3-5 Ti 2p spectra data for HC-TiN composites (binding energy eV then counts for data followed by fit components).

3-6 Ti 2p spectra data for HC-TiC composites (binding energy eV then counts for data followed by fit components).

3-7 N 1s spectra data for HC-TiN composites (binding energy eV then counts for data followed by fit components).

**Fig 7 $N_2$ adsorption and desorption profile of HC-TiN (a) and HC-TiC (b).**

4-1 Isotherm data for HC-TiN composites (relative pressure then volume absorbed $cm^3\ g^{-1}$ in each pair of columns).

4-2 Isotherm data for HC-TiC composites (relative pressure then volume absorbed $cm^3\ g^{-1}$ in each pair of columns).

**Fig 8 The first cycle charge-discharge profiles of HC-$N_2$ and HC-TiN compositions as shown (a) and cycling performance of HC-$N_2$ and the 15.15 *wt%* HC-TiN composite (b) between 2.0 and 0.005 V (vs. $Na^+$/Na) at 50 mA $g^{-1}$.**

5-1 Charge-discharge profile data for HC-TiN composites (specific capacity mA h $g^{-1}$ then potential vs $Na/Na^+$ V in each pair of columns).

5-2 Cycling performance for HC-TiN composite over 50 cycles (columns contain cycle number, hard carbon oxidation capacity mA h $g^{-1}$, HC-TiN oxidation capacity mA h $g^{-1}$, hard carbon coulombic efficiency % and HC-TiN coulombic efficiency %).

**Fig 9 Nyquist plots of HC and 15.15 *wt%* HC-TiN composites before and after 50 cycles between 2.0 and 0.005 V vs. $Na^+$/Na at 50 mA $g^{-1}$.**

6 Nyquist plot data for HC-TiN composites (Z' Ω then –Z" Ω in each pair of columns).

**Fig 10 *Ex situ* grazing incidence XRD patterns of HC-TiN composite-based electrodes at various stages of cycling when reducing to 5 mV and oxidizing back to 2 V vs. $Na^+$/Na.**

7 *Ex situ* grazing incidence XRD patterns of HC-TiN composite-based electrodes (degrees 2θ then counts in each pair of columns).

**Fig 11 (a) Charge-discharge profiles of HC-Ar and HC-TiC composites electrodes between 2.0 and 0.005 V (vs. $Na^+$/Na) at 50 mA/g, (b) cyclic performance of HC-TiC composites in 50 cycles.**

8-1 Charge-discharge profile data for HC-TiC composites (specific capacity mA h $g^{-1}$ then potential vs $Na/Na^+$ V in each pair of columns).

8-2 Cycling performance for HC-TiC composite over 50 cycles (columns contain cycle number, hard carbon oxidation capacity mA h $g^{-1}$, HC-TiC oxidation capacity mA h $g^{-1}$, hard carbon coulombic efficiency % and HC-TiC coulombic efficiency %).

**Fig S1 TGA analysis of 15.15 *wt%* HC-TiN (a) and 8.65 *wt%* HC-TiC composites (b) under argon-oxygen mixture.**

9-1 TGA analysis data for HC-TiN composites (temperature °C then mass %).

9-2 TGA analysis data for HC-TiC composites (temperature °C then mass %).

**Fig S6 Curve fitting of three peaks for (left) 15.15 *wt%* HC-TiN composite and (right) 8.65 *wt%* HC-TiC composite in Raman spectra.**

10-1 Curve fitting of Raman spectrum for HC-TiN composites (wavenumber $cm^{-1}$ then counts in each pair of columns: data, baseline, D, D3 and G).

10-2 Curve fitting of Raman spectrum for HC-TiC composites (wavenumber $cm^{-1}$ then counts in each pair of columns: data, baseline, D, D3 and G).

**Fig S7 Raman spectrum of commercial $TiO_2$ (Sigma-Aldrich, anatase, ≥ 99%) with Raman shift of 200-1000 $cm^{-1}$.**

11 Raman spectrum data for $TiO_2$ (wavenumber $cm^{-1}$ then counts).

**Fig S8 XPS survey spectra of (left) 15.15 *wt%* HC-TiN composite and (right) 8.65 *wt%* HC-TiC composite.**

12-1 XPS survey spectra data for HC-TiN composites (binding energy eV then counts).

12-2 XPS survey spectra data for HC-TiC composites (binding energy eV then counts).

**Fig S9 XPS deconvolution spectra of C 1s, and N 1s of HC-$N_2$ and HC-Ar.**

13-1 C 1s spectra data for HC-$N_2$ (binding energy eV then counts for data followed by fit components).

13-2 C 1s spectra data for HC-Ar (binding energy eV then counts for data followed by fit components).

13-3 N 1s spectra data for HC-$N_2$ (binding energy eV then counts).

13-4 N 1s spectra data for HC-Ar (binding energy eV then counts).

**Fig S10 Pore size distribution of HC-TiN (a) and HC-TiC (b), with loadings as shown. Data obtained by NLDFT method.**

14-1 Pore size distribution data for HC-TiN composites (pore width nm then incremental pore volume $cm^3$ $g^{-1}$ in each pair of columns).

14-2 Pore size distribution data for HC-TiC composites (pore width nm then incremental pore volume $cm^3$ $g^{-1}$ in each pair of columns).

**Fig S11 The first cycle of dQ/dV vs potential curve obtained from HC and HC-TiN composites.**

15 The first cycle of dQ/dV vs potential curve data for HC-TiN composites (reduction potential V, reduction dQ/dV mA h $V^{-1}$, oxidation potential V, oxidation dQ/dV mA h $V^{-1}$ in each set of 4 columns)

**Fig S12 Cycling performance of hard carbon and 15.15 *wt%* HC-TiN composites electrodes (different samples to those shown in Fig. 8) between 2.0 and 0.005 V (vs. $Na^+$/Na) at 50 mA $g^{-1}$.**

16 Cycling performance for HC-TiN composites over 50 cycles (cycle number, HC oxidation specific capacity and HC-TiN oxidation specific capacity).

**Fig. S13 XRD refinement of 15.15 *wt%* HC-TiN electrode before cycling by using GSAS package.**

17 Fitted XRD data for HC-TiN ink on a copper foil current collector before cycling (2θ ° then counts: data, fit, background and difference).

Date of data collection: 2017-2019

Information about geographic location of data collection: University of Southampton, U.K. and Harwell XPS, Chilton, UK



Date that the file was created: April 2019