Supplementary Material

In situ formation of nanoparticles on carbon nanofiber surface using ceramic intercalating agents

Alex A. Burnstine-Townley[1,2], Sajia Afrin[1,2], Yuen Yee Li Sip[1,3], David Fox[1,2], Lei Zhai*[,1,2,3]

1 NanoScience and Technology Center, University of Central Florida, Florida 32816, United States of America
2 Department of Chemistry, University of Central Florida, Florida 32816, United States of America
3 Department of Materials Science and Engineering
* Correspondence: lzhai@ucf.edu

1. Characterization Instrumentation

X-ray diffraction (XRD) patterns were recorded using a PANalytical Empyrean X-ray diffractometer to determine the crystal phases present and their lattice spacing. X-ray Photoelectron Spectroscopy (XPS) spectra were recorded using a PHI 5400 to identify the chemical identity and states of the atoms. Scanning Electron Microscopy (SEM) images were collected by Zeiss Ultra-55 FEG with Noran System 7 energy dispersive X-ray (EDX) analysis. These images show morphology as well as spatial position atoms and domains. Transmission Electron Microscopy (TEM) images were collected by Tecnai F30 with EDX mapping analysis to identify nanoscale morphology.

2. Chemicals

Polyacrylonitrile (PAN) (average, Mw = 150,000) and N, N- dimethylformamide (DMF) were purchased from Sigma-Aldrich. Nickel chloride hexahydrate ($NiCl_2 \cdot 6H_2O$) was purchased from Fisher Scientific. Ceraset PSZ 20 – (EF: $SiNC_{1.4}H_{5.4}$) was purchased from Kion Corporation. The reagents were used as received without any further experimental purification.

3. Characterization Results

8:1:4 PAN:silazane:$NiCl_2$ fibers pyrolyzed at 1000°C

The XRD diffractogram (Figure 2d) shows the formation of various phases, including cubic metallic nickel at 44, 52, and 76° *2θ*, in addition to orthorhombic Ni_2Si with peaks at 32, 39, 42.4, 45.8, 48.9, 53.5, and 68.2° *2θ*.[1] An additional peak at 47.2° is attributed to $Ni_{31}Si_{12}$.[2] XPS further confirms the presence of these species (Figure S2), with Ni $2p_{3/2}$ peak at 854.24 eV and $2p_{1/2}$ at 871.82 eV, indicative of Si-Ni bonds.[3] XPS data also shows characteristic peaks at 283.3 eV for the C 1s peak representing the C-Si bond and 101.9 eV for the Si 2p corresponding to the Si-C bond. Other peaks include the Si-N bond at 102.9 eV, Si-O at 103.5 eV and C-N/O at 285.0 eV.[4,5]

8:4 PAN:$NiCl_2$ fibers pyrolyzed at 1000°C

XPS shows the metallic Ni $2p_{3/2}$ peak at 853.60 eV (Fig. S5), shifted from 854.24 eV (Figure S2) that is associated with the Ni-Si bond. Corroborating shift of the Ni $2p_{1/2}$ peak was also observed from 871.82 eV to 871.30 eV when silazane was excluded.

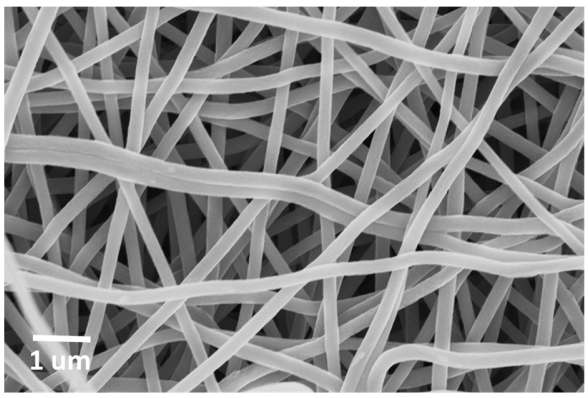

Figure S1: SEM image of 8:1:4 PAN:silazane:$NiCl_2$, stabilized at 260°C.

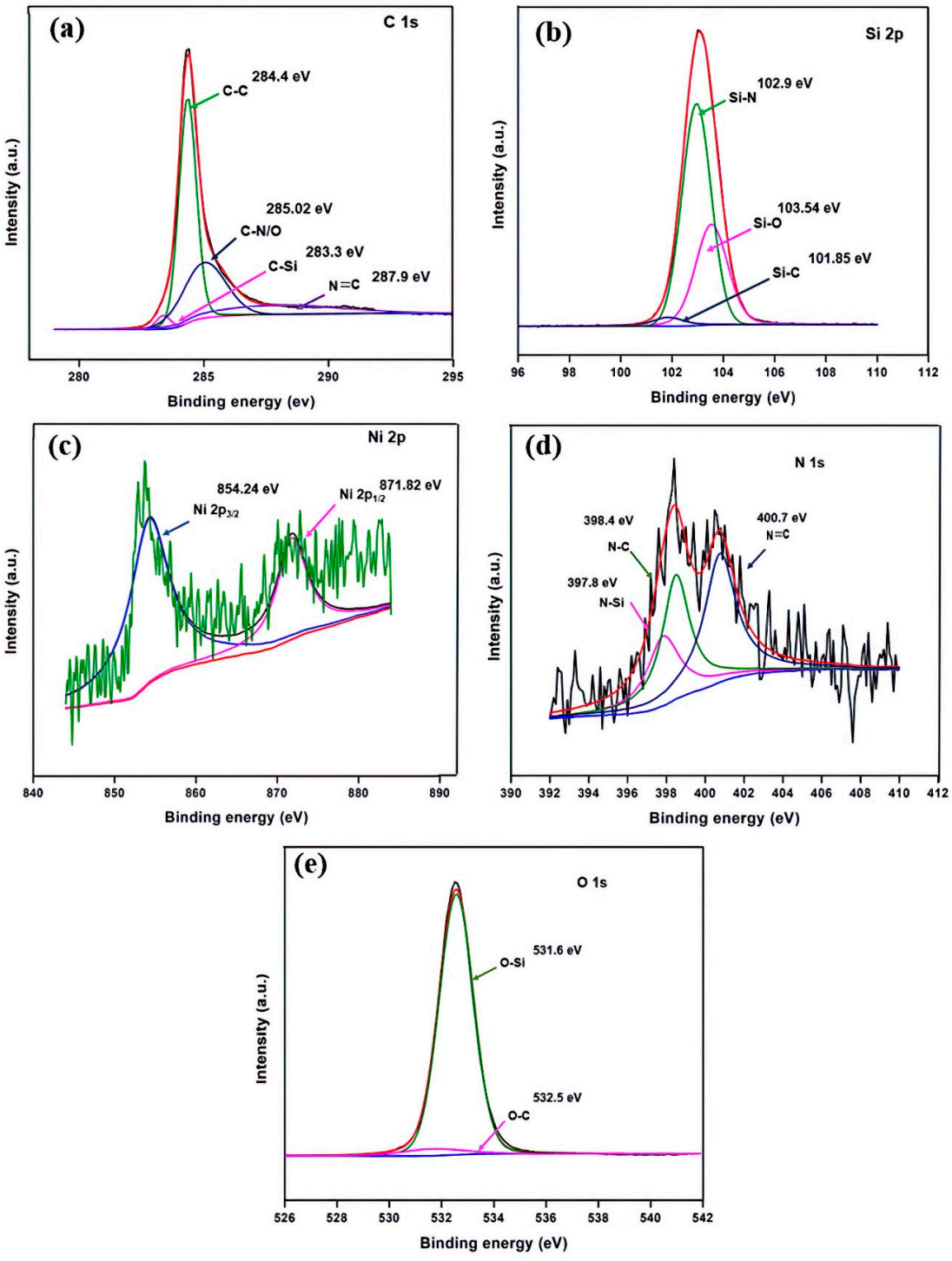

Figure S2: XPS spectra of 8:1:4 PAN:silazane:$NiCl_2$, pyrolyzed at 1000°C. (a) C 1s, (b) Si 2p, (c) Ni 2p, (d) N 1s, (e) O 1s.

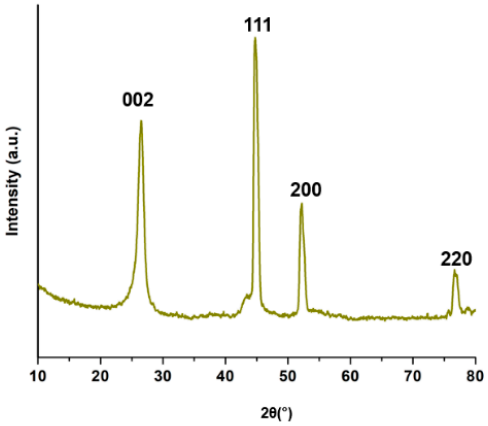

Figure S3: XRD pattern of 8:4 PAN:$NiCl_2$, pyrolyzed at 1000°C.

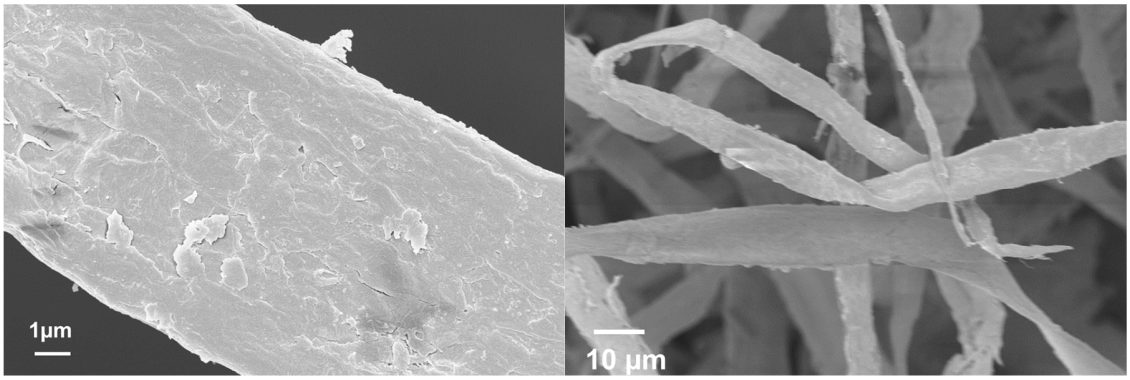

Figure S4: SEM images of 8:4 PAN:$NiCl_2$, pyrolyzed at 1000°C.

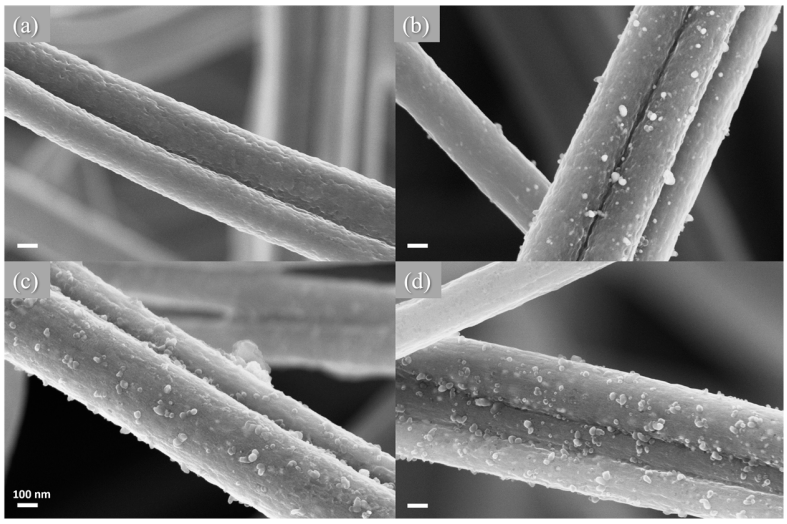

Figure S5: SEM images of 8:1:4 PAN:silazane:$NiCl_2$, pyrolyzed at temperature (a) 600°C, (b) 700°C, (c) 800°C, (d) 900°C.