Supplementary Information for

Exclusive Formation of SnO By Low Temperature Single-Source AACVD

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General Experimental Details

All manipulations were carried out using standard Schlenk line and glovebox techniques under an inert atmosphere of argon. NMR experiments were conducted in J. Youngs tap NMR tubes made up and sealed in a Glovebox. NMR spectra were recorded either on a Bruker AV-400 spectrometer at 100.6 MHz (^{13}C), Bruker AV-300 at 75.5 MHz (^{13}C), spectrometer or a Bruker AV-250 spectrometer at 62.9 MHz (^{13}C). The spectra were referenced relative to residual solvent resonances. Data quoted was recorded at 298 K. Elemental analysis was performed by Mr. Stephen Boyer at SACS, London Metropolitan University. Solvents were dried by passage through a commercial solvent purification system, under nitrogen and stored in ampoules over molecular sieves. D_8-toluene was purchased from Goss Scientific Instruments Ltd. and dried over molten potassium before distilling under nitrogen and storing over molecular sieves. $[Sn(NMe_2)_2]_2$ was prepared by a literature procedure[1] and *tert*-butyl isocyanate was purchased from Sigma-Aldrich.

TGA analysis of the complexes was performed at SAFC Hitech, Bromborough, UK, using a Shimadzu TGA-51 Thermogravimetric Analyser. Data points were collected every second at a ramp rate of 20 °C min^{-1} in a flowing (50 ml min^{-1}) N_2 stream. Thin films were deposited using either a cold wall or hot wall CVD conditions onto glass microscope slides or silicon wafers. The cold wall reactor (Electro-gas Systems Ltd, UK) consisted of a tubular quartz reactor containing a silicon carbide coated graphite susceptor. The temperature of the susceptor was monitored using a k-type thermocouple coupled with a proportional–integral–derivative controller (PID controller) and heated with a water cooled IR lamp mounted externally beneath the reaction tube. The hot wall system comprised a TSI 3076 Constant Output Atomiser using argon at 20 psi to generate the aerosol and act as carrier gas. The aerosol was passed through a quartz tube containing the substrate, heated by an Elite thermal Systems Ltd tube furnace. FE-SEM analysis of the films was undertaken on a JEOL 6301F. EDX analysis was performed using a JEOL 6480 LV SEM microscope. AFM analysis was performed using a Digital Instruments Nanoscope IIIa, with TAP300 tips in contact mode (tip radius <10 nm). Powder XRD of the films was performed on a Bruker D8 Advance powder diffractometer, using a Cu anode X-ray source (Kα wavelength = 1.5406 Å) at the University of Bath. X-ray photoelectron spectra were recorded on a Thermo Scientific K-Alpha instrument with a monochromated Al K_α X-ray source having an X-ray energy of 1486.6eV (thermo scientific Ltd, East Grinstead, UK) spectra were quantified using the CasaXPS software (CasaSoftware Ltd, Teignmouth, UK).

Preparation of Compound 1

To a solution of $Sn(NMe_2)_2$ (200mg, 0.97mmol) in toluene (5mL) was added ^{t}butyl isocyanate (192mg, 1.94mmol). The solution was stirred then left to evaporate in an argon atmosphere resulting in colourless crystals (390 mg, 0.96 mmol, 99%). M.p. 70 – 72 °C. ^{1}H NMR (300MHz, d^8 Tol); 1.37 (s, 9H, C-C*H*$_3$), 2.44 (s, 6H, N-C*H*$_3$). ^{13}C NMR (300MHz, d^8 Tol); 31.4 (C-*C*H$_3$), 41.4(N-*C*H$_3$), 53.0 (*C*-CH$_3$), 167.0 (N-*C* (O)-N). ^{119}Sn NMR (300MHz, d^8 Tol); δ -357. Elemental analysis: Expected for $C_{14}H_{30}N_4O_2Sn$ 41.51 %, H 7.46 %, N 13.83 %; Found C 41.47 %, H 7.54 %, N 13.69 %.

Figure S1: Thermogravimetric analysis of compound **1**

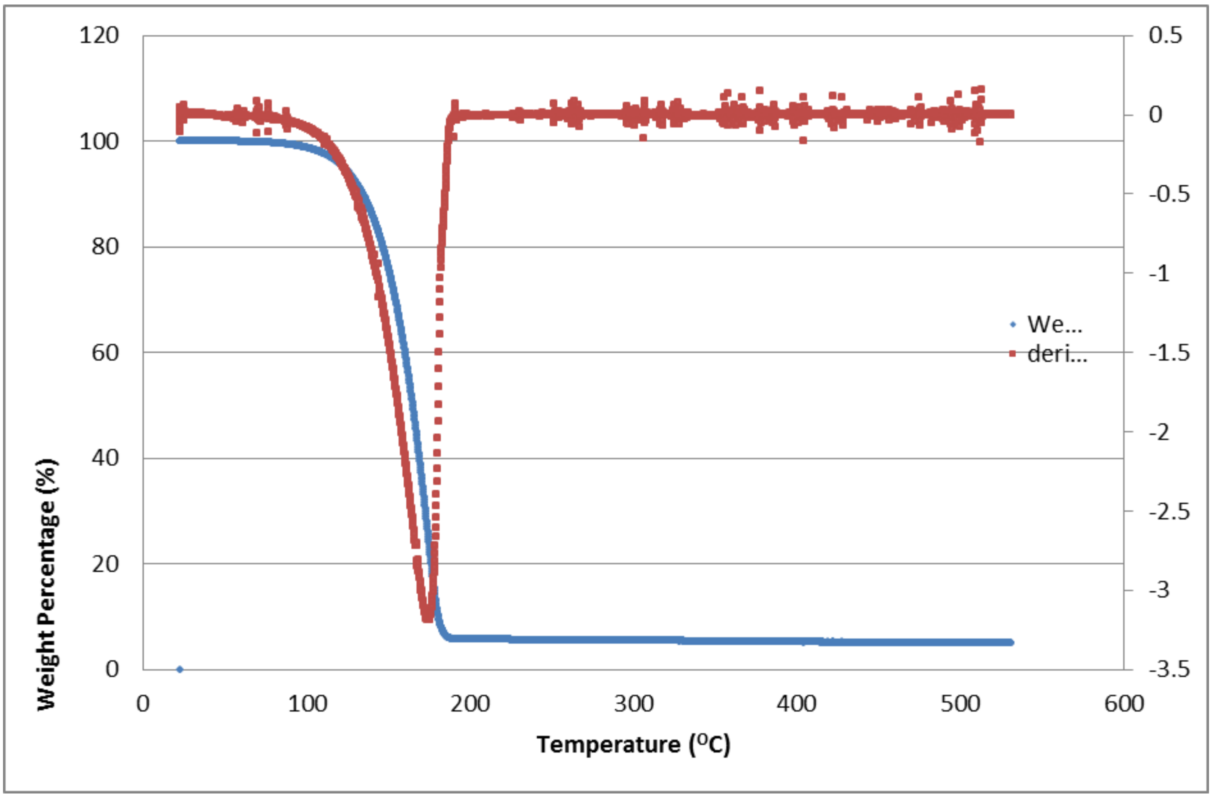


Figure S2: Raman spectrum of SnO deposited on silicon at 340 °C

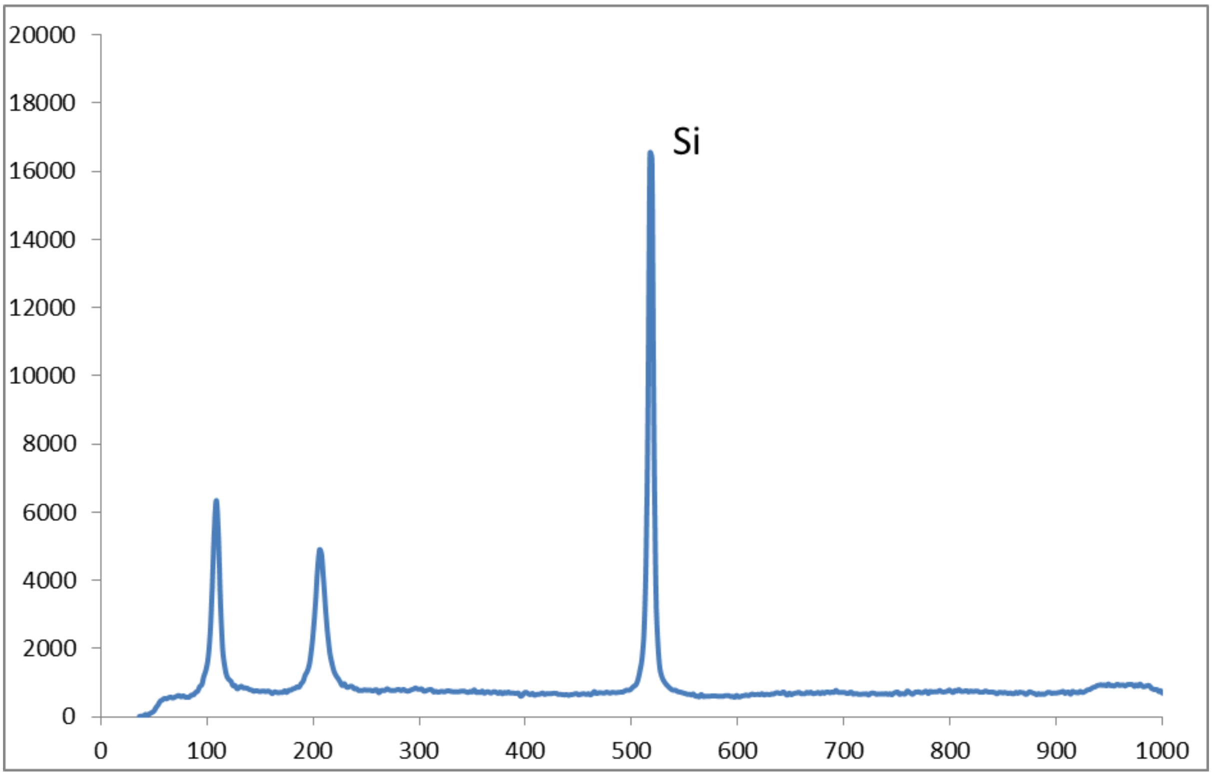
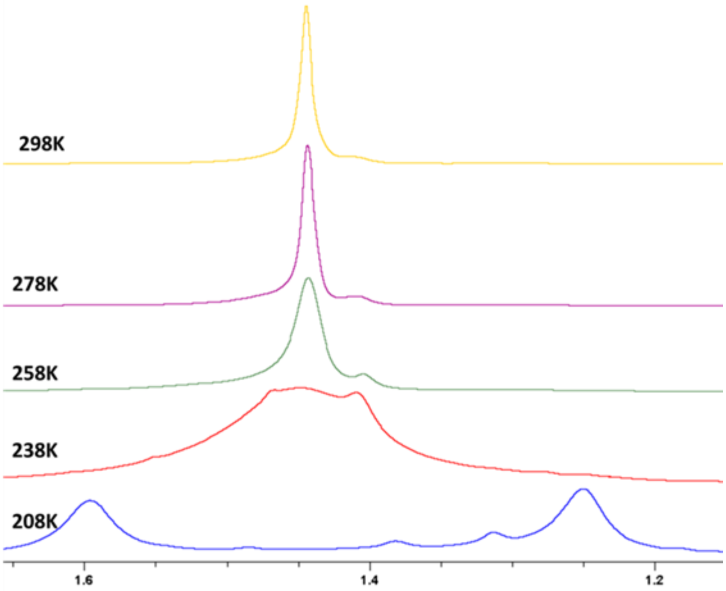


Figure S3: Variable Temperature ^{1}H NMR Analysis of Compound **1**



$\Delta G \cong RT_c[23 + \ln(T_c/\Delta\upsilon)$ where $\Delta\upsilon = 142$ Hz (maximum peak to peak separation at 200 K), T_c = coalescence temperature, 234 K for $\Delta G \cong 46$ kJ mol^{-1}.

Single Crystal X-ray Crystallography

Data for compound **1** were collected on a Nonius Kappa CCD diffractometer at 150(2) K using Mo-Kα radiation (λ = 0.71073 Å). Structure solution and refinement was performed using SHELX86[2] and SHELX97[3] software, respectively. Corrections for absorption were made in all cases. Data were processed using the Nonius Software.[4] Structure solution,[5] followed by full-matrix least squares refinement[3b] was performed using the WINGX-1.80 suite of programs throughout.[6] Hydrogen atoms were included at calculated positions.

X-ray diffraction data for **1**. $C_{14}H_{30}N_4O_2Sn$, M = 405.11, triclinic, P-1, a = 8.6700(3) Å, b = 9.4020(3) Å, c = 12.4160(3) Å, α = 79.878(2)°, β = 84.754(2)°, γ = 70.462(2)°, V = 938.39(5) Å^3, Z = 2, ρ = 1.434 g cm^{-3}, R_1 [I > 2σ(I)] = 0.0228, wR_2 [I > 2σ(I)] = 0.0539, R_1 [all data] = 0.0272, wR_2 [all data] = 0.0556, measured reflections = 14456, unique reflections = 5596, R_{int} = 0.0340.

References

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