Supporting Information

Structure Analysis at the Nanoscale: Closed WS_2 Nanoboxes through a Cascade of Topotactic and Epitactic Processes

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Table S1: Measurement and refinement parameters of the x-ray diffraction pattern of the precursor

Diffractometer	Siemens D5000
Sample preparation	Fine powder fixed between two stripes of Scotch™ tape
Measuring mode	Transmission
Wavelength /Å	1.540596
Measuring range	$10 \leq 2\Theta\ /° \leq 90$; $0.71 \leq Q\ /Å^{-1} \leq 5.77$
Temperature /K	298K
Profile Fit	Rietveld refinement according to reported crystal structure models
Background / Parameters	Chebyshev / 15
Profile function	Fundamental Parameters Approach
Program	TOPAS Academic V5
Total No. of Parameters	31
R_{exp}	4.10
R_{wp}	10.53
GoF	2.57
Weighted Durbin Watson	0.59
Phase I	**WO_3, *mP*32**
Space group	$P2_1/c$
Cell parameters /Å	a = 7.3071(5), b = 7.5397(7), c = 10.5485(10), β = 133.17(1)°
Crystallite size / nm	> 100nm, crystalize size broadening below instrumental broadening
Fraction /%wt	60.2(4)
Strain	0.31(1)
Preferred Orientation	1.17, (0 2 0)
Phase II	**$WO_3 \times {}^1/_3\ H_2O$, *oF*52**
Space group	*Fmm*2
Cell parameters /Å	a = 7.3320(5), b = 12.6007(10), c 7.6973(9)
Crystallite size / nm	95(2), crystalize size broadening in same order of magnitude as instrumental broadening
Fraction /%wt	39.8(4)
Preferred Orientation	1.21(1), (0 0 2)

Table S2. Measurement and refinement parameters of the x-ray diffraction pattern of the product after sulfidization

Diffractometer	Siemens D5000
Sample preparation	Fine powder fixed between two stripes of Scotch™ tape
Measuring mode	Transmission
Wavelength /Å	1.540596
Measuring range	$10 \leq 2\Theta\ /° \leq 90$; $0.71 \leq Q\ /Å^{-1} \leq 5.77$
Temperature /K	298K
Profile Fit	Rietveld refinement according to reported crystal structure models
Background / Parameters	Chebyshev / 15
Profile function	Fundamental Parameters Approach
Program	TOPAS Academic V5
Total No. of Parameters	23
R_{exp}	4.1
R_{wp}	11.1
GoF	2.72
Weighted Durbin Watson	0.27
Phase I	**WS_2, *hP*6**
Space group	$P6_3/mmc$
Cell parameters /Å	a = 3.151(1), c = 2 × 6.327(7)*
Crystallite size / nm	10(1)*
Fraction /%wt	41(1)
Preferred Orientation	1.15(1), (0 0 2)
Phase II	**WS_2, *hR*9**
Space group	*R*3*m* (hexagonal setting)
Cell parameters /Å	a = 3.151(1), c = 3 × 6.327(7)*
Crystallite size / nm	10(1)*
Fraction /%wt	59(1)
Preferred Orientation	1.15(1)*, (0 0 3)

* values refined constraint to each other

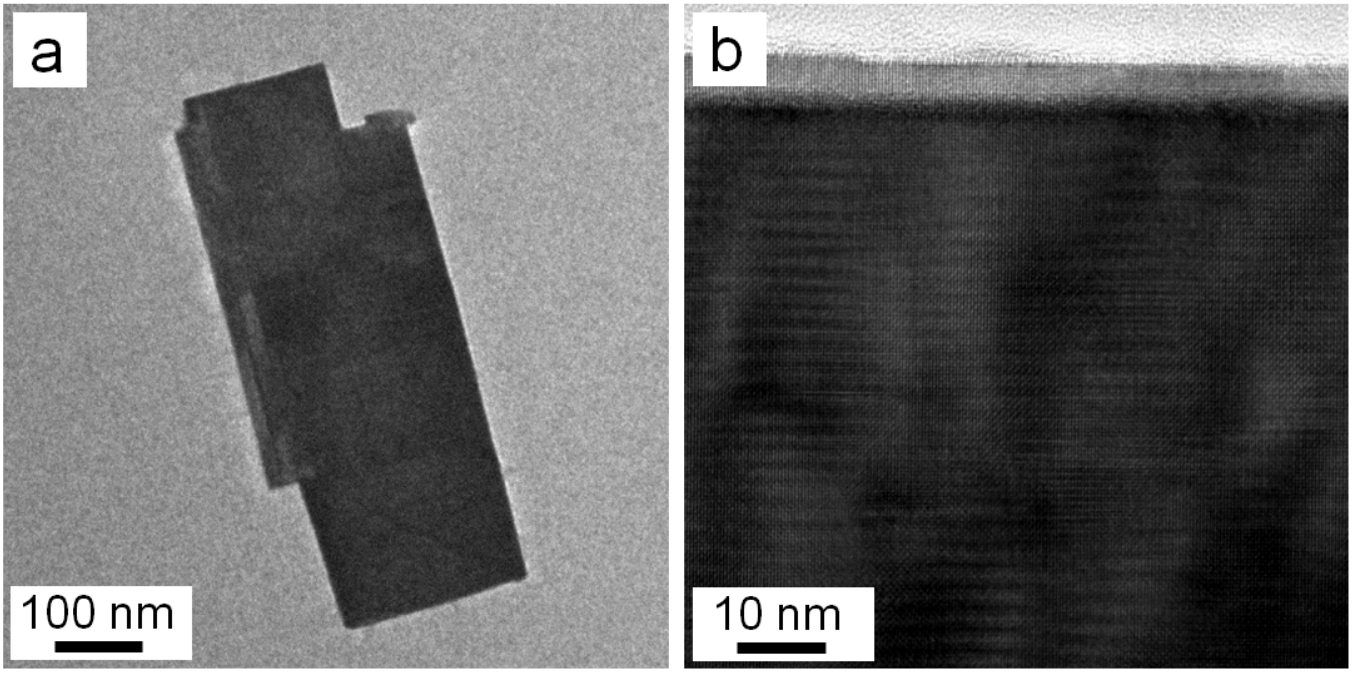

Figure S1. TEM overimage of two rods of WO_3 / $WO_3 \times ⅓\ H_2O$ precursor. (b) HRTEM showing disorder in WO_3 / $WO_3 \times ⅓\ H_2O$.

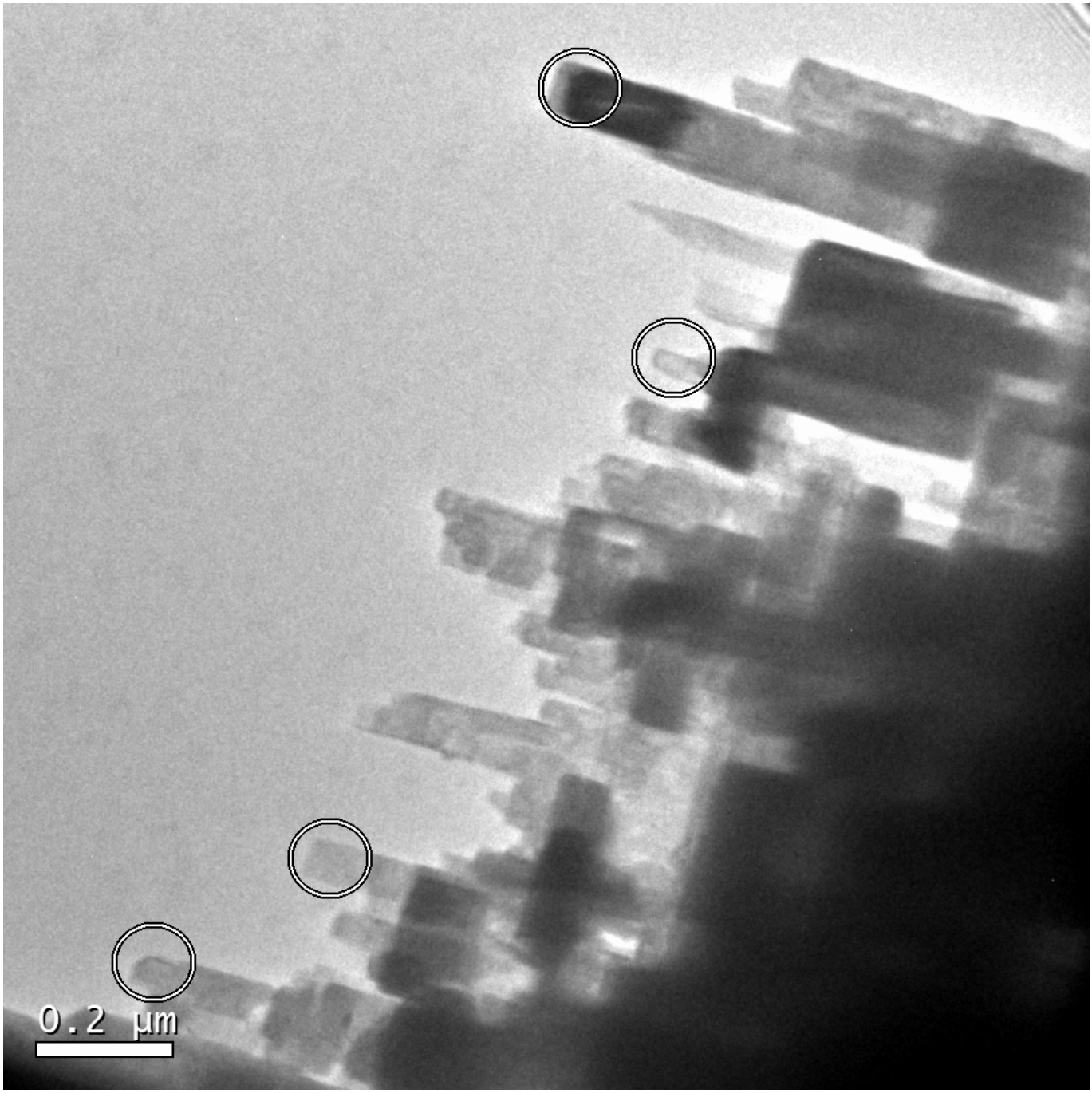

Figure S2. TEM overview image of the sulfidized WS_2 product