ESI for

Chalcogenoether complexes of Nb(V) thio- and seleno-halides as single source precursors for low pressure chemical vapour deposition of NbS_2 and $NbSe_2$ thin films

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Table S1 X-ray crystallographic data

Compound	$[NbSCl_3(SMe_2)]$	$[NbSCl_3(MeSCH_2CH_2SMe)]$
Formula	$C_2H_6Cl_3NbS_2$	$C_4H_{10}Cl_3NbS_3$
Formula weight	293.45	353.56
Crystal system	Monoclinic	Monoclinic
Space group (No)	$P2_1$ (4)	$P2_1$ (4)
a/ Å	7.2513(5)	7.2010(3)
b/ Å	13.0295(6)	11.3494(4)
c/ Å	9.8715(7)	7.8693(5)
α/ deg	90	90
β/ deg	108.075(7)	115.502(7)
γ/ deg	90	90
U/ $Å^3$	886.6(1)	580.47(6)
Z	4	2
μ(Mo-Kα)/mm^{-1}	2.641	2.023
F(000)	568	348
Total no. reflections	7662	5010
Unique reflections	3468	2251
R_{int}	0.034	0.016
No. of parameters, restraints	149, 1	102, 2
R_1[b] [Io>2σ(Io)]	0.036	0.018
R_1 (all data)	0.073	0.018
wR_2[b] [Io >2σ(Io)]	0.042	0.042
wR_2 (all data)	0.075	0.043

[a] Common items: temperature =100 K; wavelength (Mo-K_α) = 0.71073 Å; θ(max)= 27.5°.

[b] $R_1 = \Sigma||F_o|-|F_c||/\Sigma|F\sigma|$, $wR_2=[\Sigma\omega(F_o^2-F_c^2)^2/\Sigma\omega F_o^4]^{1/2}$

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Table S1 cont.

Compound	$[NbSCl_3(^iPrSCH_2CH_2S^iPr)]$	$[NbSCl_3\{MeSe(CH_2)_3SeMe]$
Formula	$C_8H_{18}Cl_3NbS_3$	$C_5H_{12}Cl_3NbSSe_2$
Formula weight	409.66	461.39
Crystal system	Monoclinic	Tetragonal
Space group (No)	$P2_1/c$ (14)	$I4_1/a$ (88)
a/ Å	8.7791(2)	10.0061(1)
b/ Å	9.1658(2)	10.0061(1)
c/ Å	19.6825(5)	26.1100(8)
α/ deg	90	90
β/ deg	98.753(2)	90
γ/ deg	90	90
U/ $Å^3$	1565.36(6)	2614.2(1)
Z	4	8
μ(Mo-K_α)/mm^{-1}	1.652	7.216
F(000)	824	1744
Total no. reflections	14369	11648
Unique reflections	5181	2242
R_{int}	0.032	0.045
No. of parameters, restraints	140, 0	60, 0
R_1 [Io>2σ(Io)]	0.038	0.030
R_1 (all data)	0.050	0.042
wR_2 [Io >2σ(Io)]	0.084	0.063
wR_2 (all data)	0.091	0.068

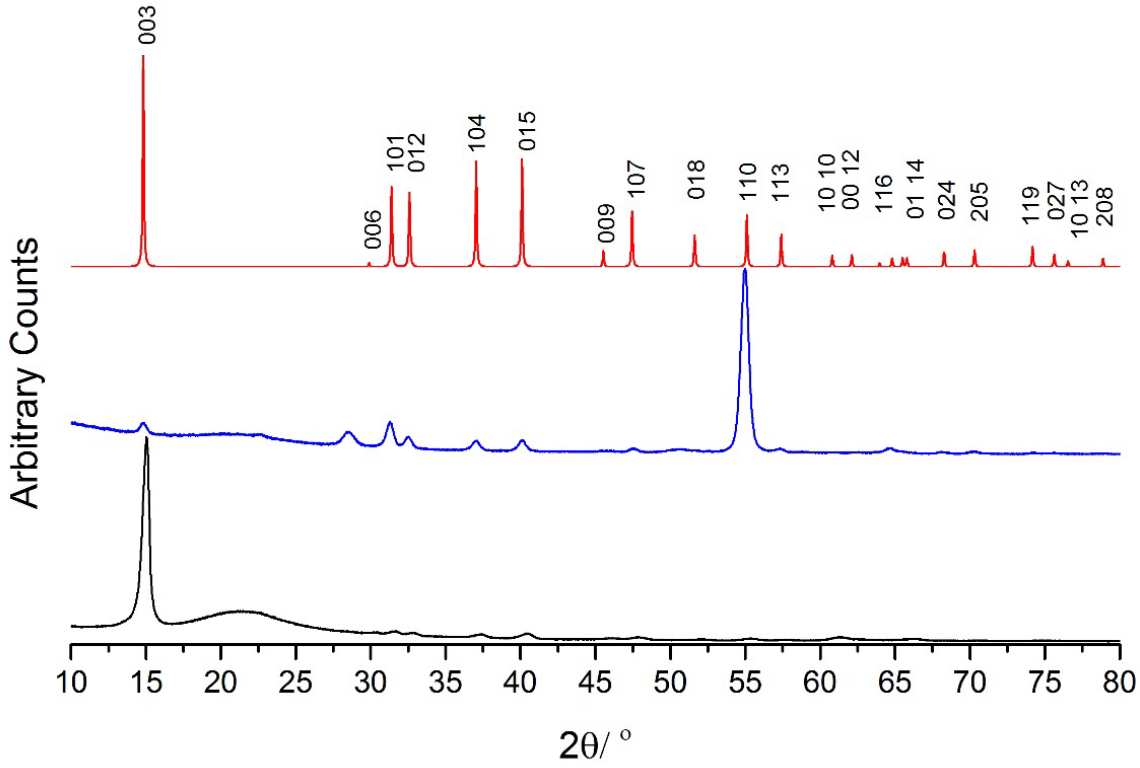

Fig. S1 In plane XRD (blue), grazing incidence (incidence angle = 1°) XRD (black) from the NbS_2 thin film deposited by LPCVD using [$NbSCl_3${$^nBuS(CH_2)_3S^nBu$}] at 700 °C; simulated XRD pattern for bulk NbS_2 from ref. 26 (red). The broad feature at $2\theta \sim 22°$ is from the SiO_2 substrate.

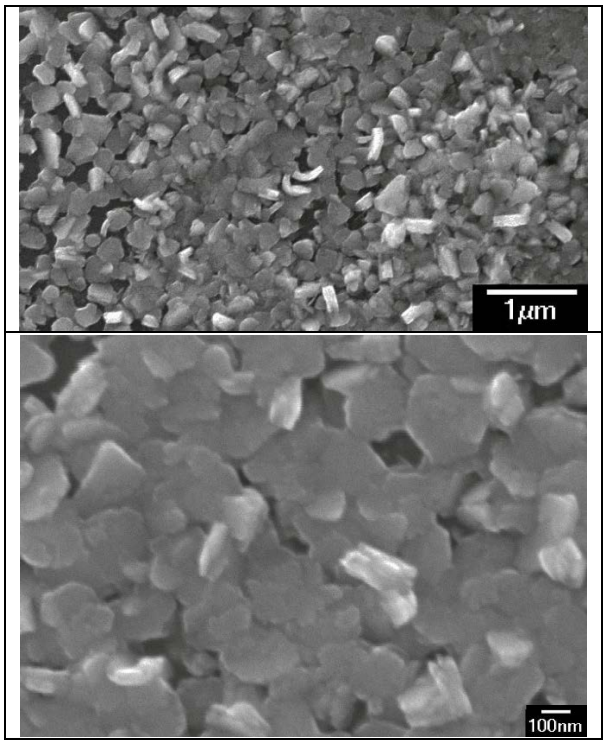

Fig. S2 SEM image of NbS_2 thin film deposited from [$NbSCl_3${$^nBuS(CH_2)_3S^nBu$}]

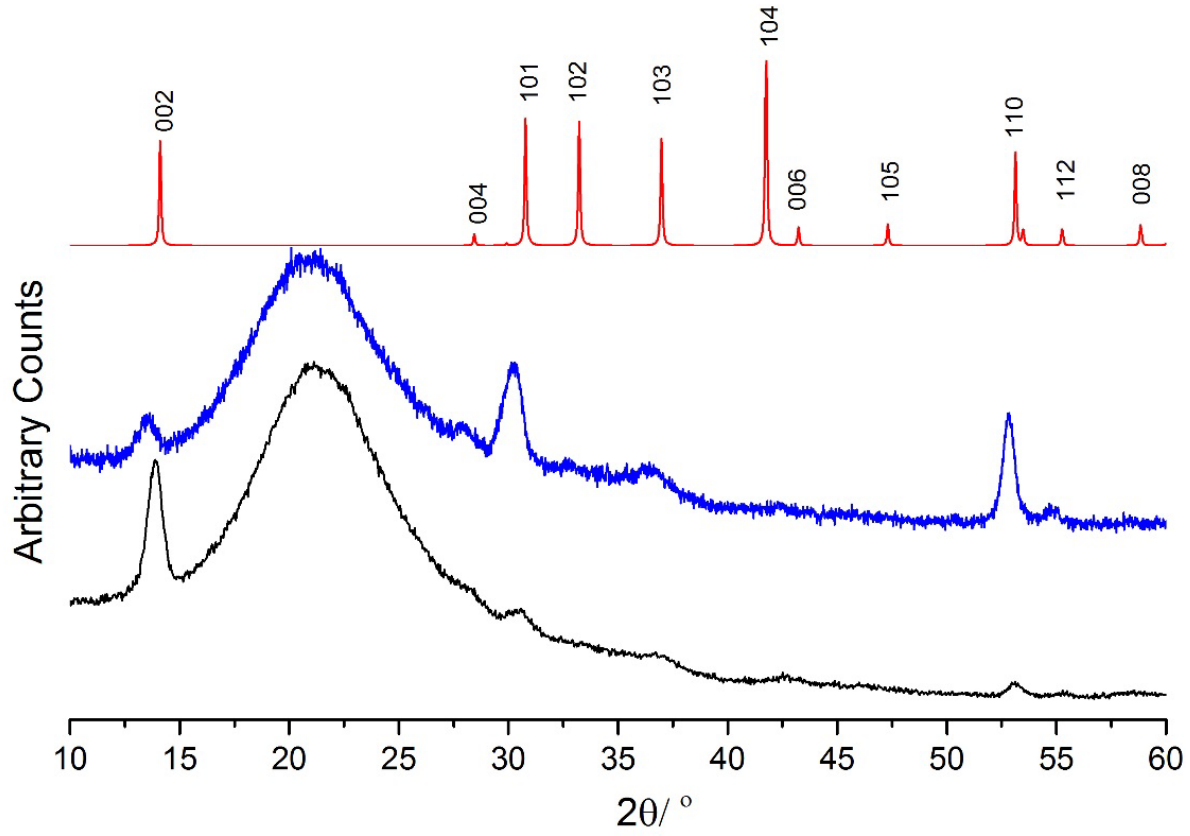

Fig. S3 In plane XRD (blue), grazing incidence (incidence angle = 1°) XRD (black) from the $NbSe_2$ thin film deposited by LPCVD using [$NbSe_2Cl_3(Se^nBu_2)$] at 650 °C; simulated XRD pattern from bulk $NbSe_2$ from ref. 27 (red). The broad feature at 2θ ~ 22° is from the SiO_2 substrate.

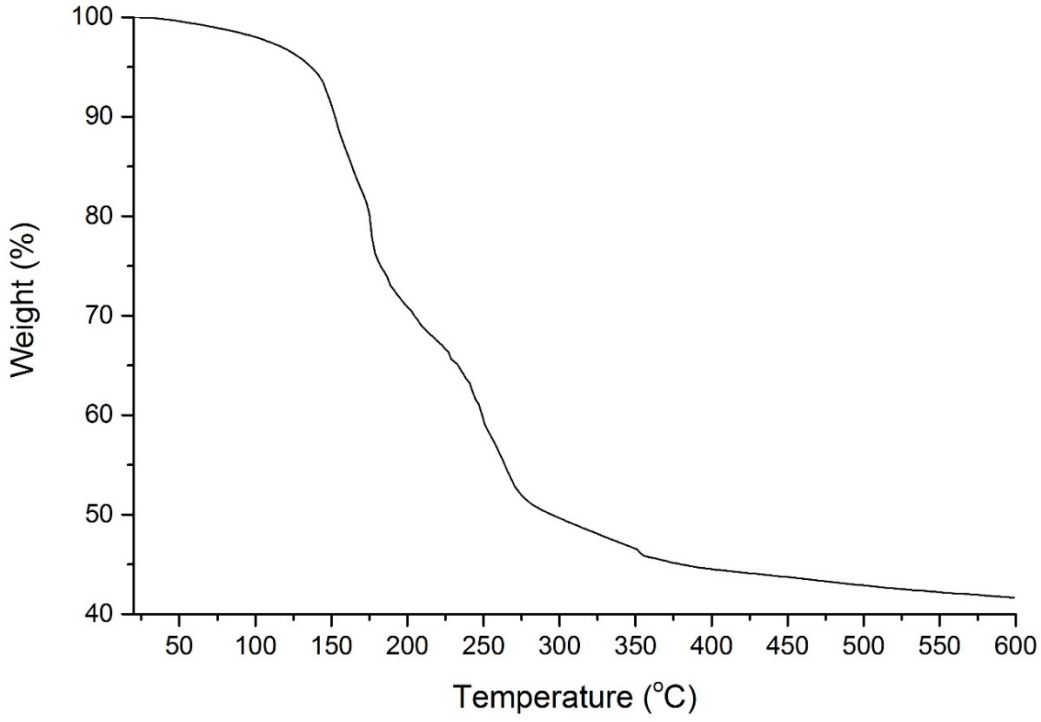

Fig. S4 TGA of [$NbSCl_3(S^nBu_2)$]

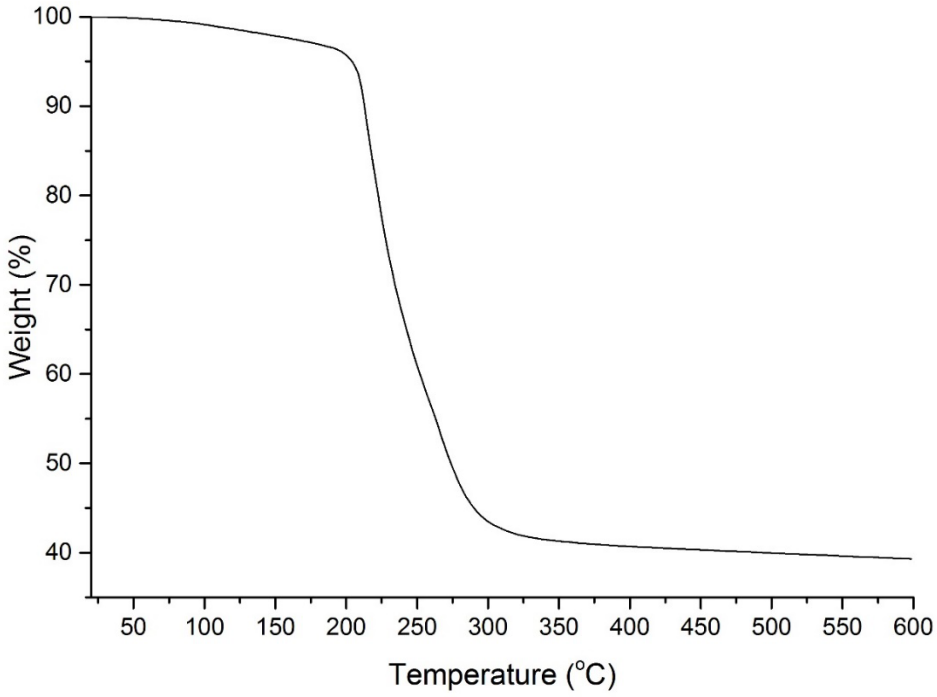

Fig. S5 TGA of [$NbSCl_3\{^nBuS(CH_2)_3S^nBu\}$]

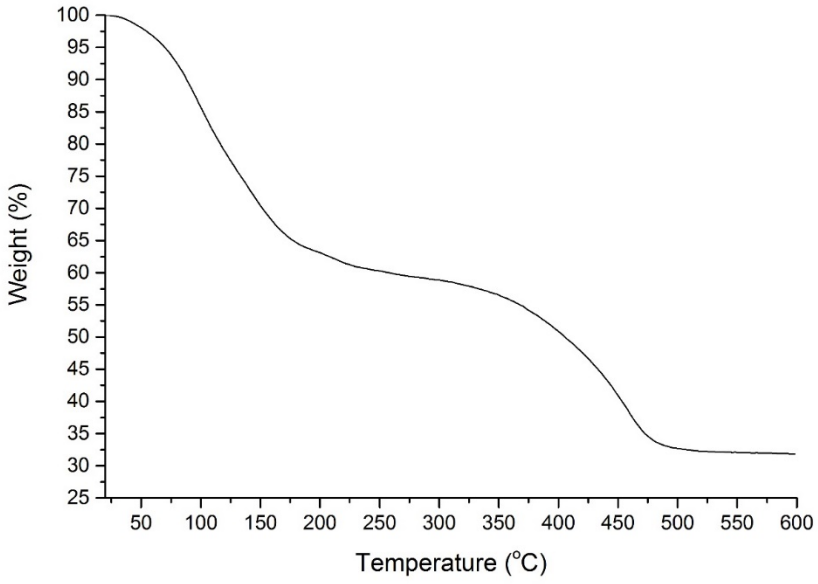

Fig. S6 TGA of [$NbSe_2Cl_3(Se^nBu_2)$]