Supplemental Information: Polytypism and Superconductivity in the NbS_2 System

Catherine Witteveen,[1,2] Karolina Gornicka,[3,4] Johan Chang,[2] Martin Månsson,[5] Tomasz Klimczuk,[3,4] and Dr. Fabian O. von Rohr[1,2,*]

[1] *Department of Chemistry, University of Zurich, CH-8057 Zurich, Switzerland*
[2] *Department of Physics, University of Zurich, CH-8057 Zurich, Switzerland*
[3] *Department of Solid State Physics, Gdansk University of Technology,80-233 Gdansk, Poland*
[4] *Advanced Materials Centre, Gdansk University of Technology, ul. Narutowicza 11/12, 80-233 Gdansk, Poland*
[5] *Department of Applied Physics, KTH Royal Institute of Technology, Roslagstullsbacken 21, SE-106 91 Stockholm, Sweden*

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I. SYSTEMATIC ERRORS

Preferred orientation of particles arises, when there is a strong preference in the particles to orient themselves in nonrandom direction. This happens, when the shape of the particle is non-spherical. In the case of layered materials, their plate-like particles will preferably orient themselves with their a-b plane parallel to the surface. In PXRD, this preferred orientation creates a systematic error in the observed intensities of the diffraction peaks. Depending on the used geometry of the PXRD instrument, the sample's preferred orientation will lie parallel (reflection geometry) or perpendicular (transmission geometry) to the incoming beam at low angles, resulting in a systematic error in the {00l}, see 1a), or {hk0} reflections, see 1b), respectively. A comparison of PXRD patterns of 2H-NbS_2 resulting from both instrument geometries is given in figure 1.

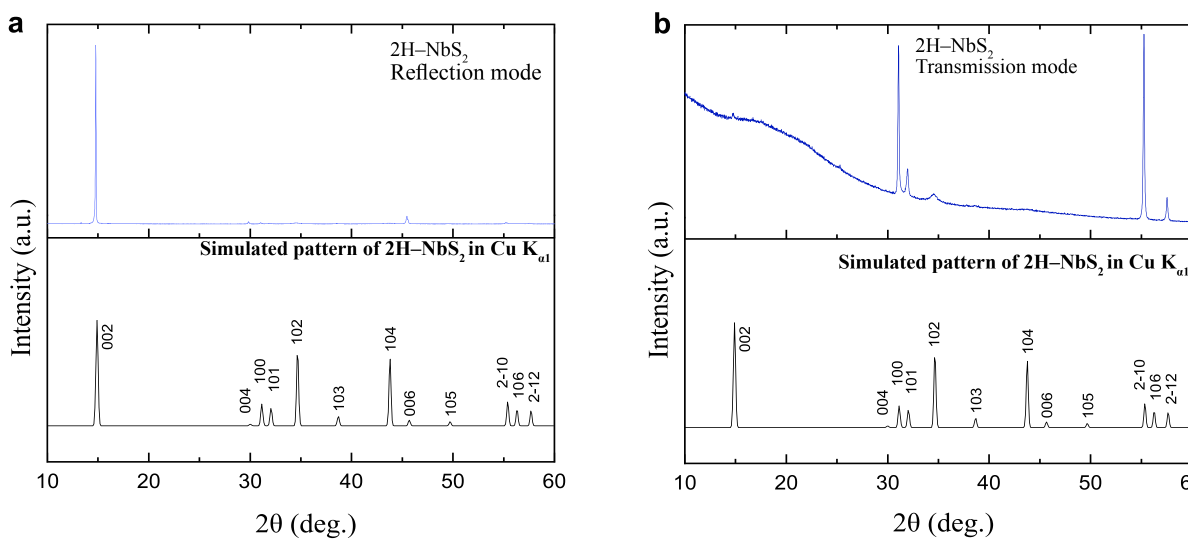

FIG. 1. The layered nature of 2H-NbS_2 resulted in very strong systematic errors. a) In reflection mode, especially the {00l} reflections are observable. b) In transmission mode a systematic error occurs for the {hk0} reflections. In the black is the simulated PXRD pattern obtained from the ICSD.

II. POWDER X-RAY DIFFRACTION DATA

PXRDs of the synthesized samples are shown in Figure 2. The gradual change with temperature from amorphous or nano-crystalline to crystalline samples is well observed in the PXRD patterns of the samples at a fixed sulfur content.

* fabian.vonrohr@uzh.ch

Crystallinity is defined for samples with the FWHM being lower than 2θ=0.21°for the reflection at $2\theta \approx 31°$, which corresponds to either the (100) reflection of 2H-NbS_2, or the (101) reflection of 3R-NbS_2 polytype. 2 samples were oxidized during the process of synthesis, which is represented by empty patterns.

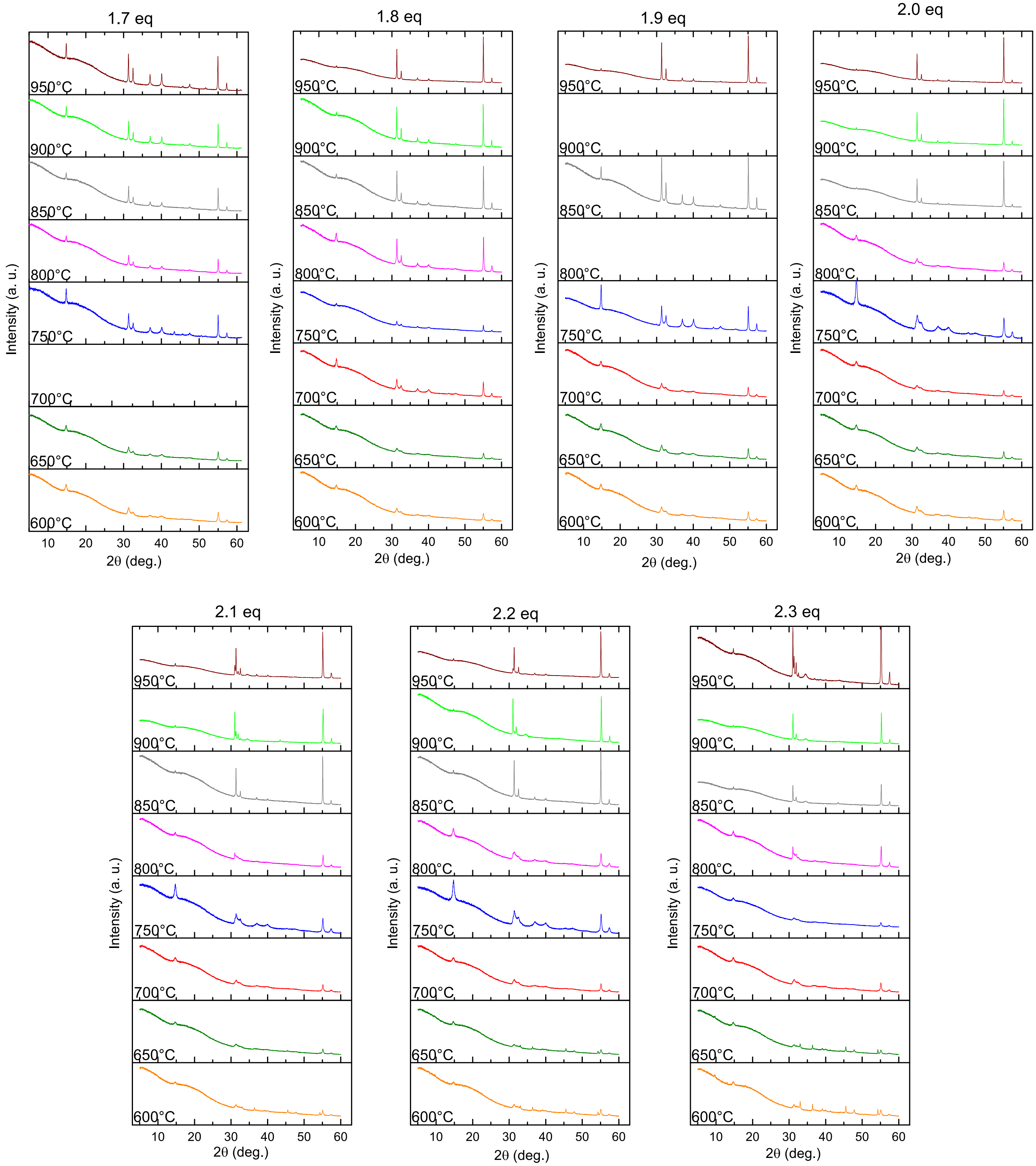

FIG. 2. All PXRD patterns of the series of samples synthesized, using Cu $K_{\alpha 1}$ radiation over a 2θ range of 10 °to 60 °for the varying eq of sulfur. The overall trend shows more crystalline samples at higher temperatures of synthesis. Empty boxes denote oxidized samples during the process of synthesis.

III. SEM ANALYSIS

SEM images taken for samples synthesized with 2.3 eq of sulfur at various temperatures. Nicely observable is the formation of the plately structure including layers at the synthesis temperatures of 850 and 950 °C, whereas at lower temperatures no distinct shape is formed.

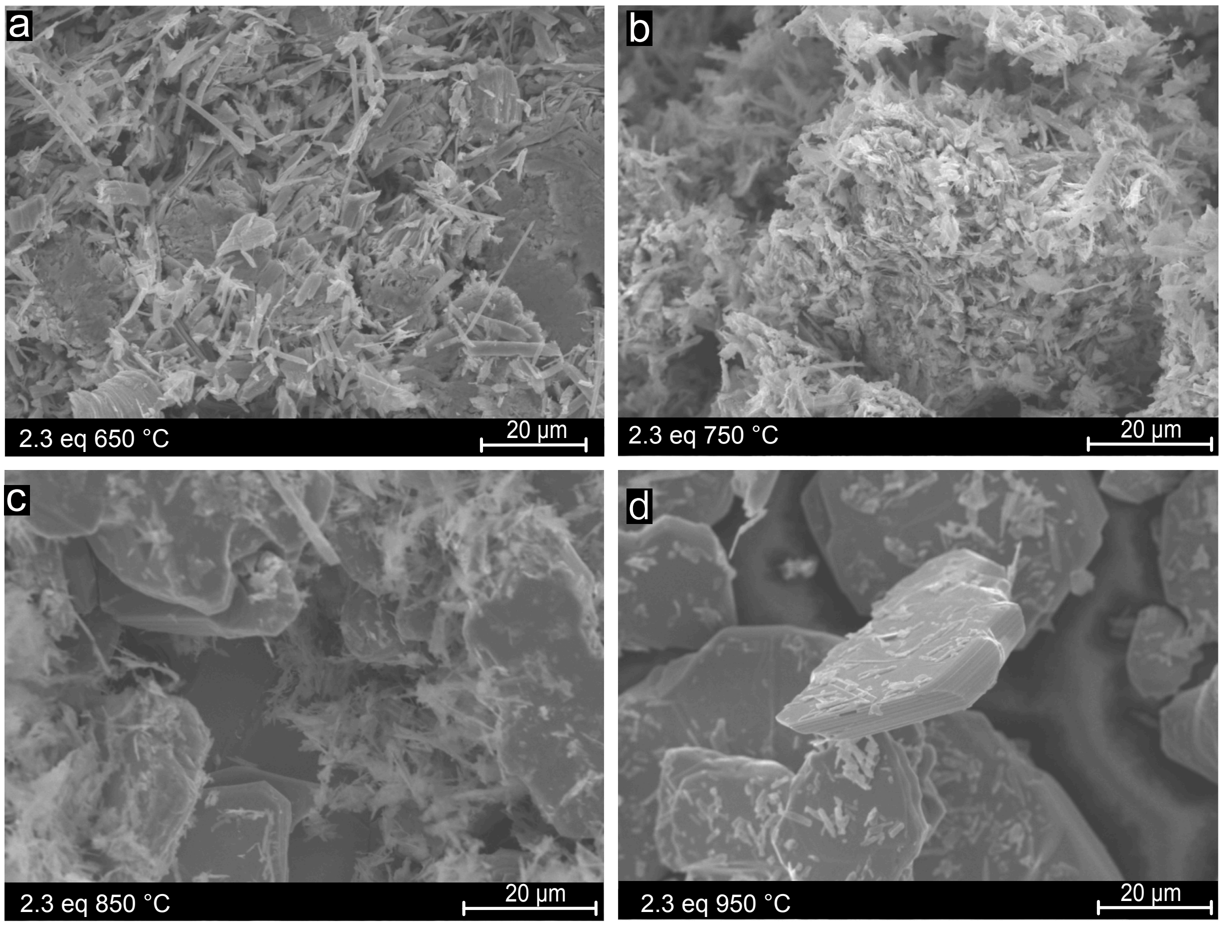

FIG. 3. SEM images of $NbS_{2.3}$ synthesized at various temperatures.

IV. SPECIFIC HEAT DATA FOR 2H-NBS$_2$

The Temperature dependence of the specific heat at magnetic fields from 0 T to 0.5 T in 0.1 T steps is shown in Figure 4. The sharp anomaly corresponding to the thermodynamic transition in 2H-NbS$_2$ from the normal state to the superconducting state is clearly lowered with higher magnetic fields, indicating the suppression of the superconducting state.

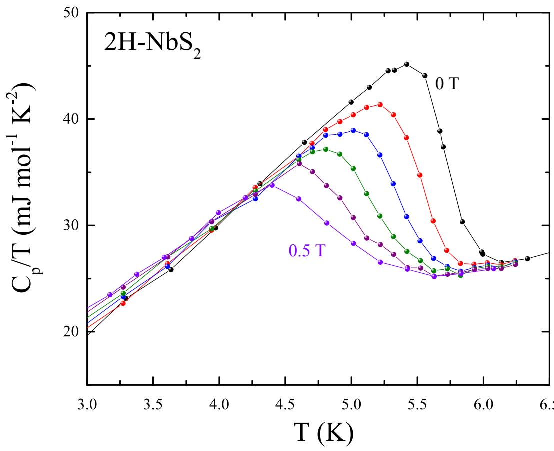

FIG. 4. Temperature dependence of the specific heat at magnetic fields from 0 T to 0.5 T in 0.1 T steps for phase pure 2H-NbS$_2$.

V. EDX ANALYSIS

The chemical composition of two selected samples was determined by EDX as 1.89 for a sample synthesized at 900 °C with 2.0 eq of sulfur, see Figure 5a) and as 1.92 for a sample synthesized at 900 °C with 2.3 eq of sulfur, see Figure 5b). That the nominal compositions does not deviate much from each other goes hand in hand with the observation of unreacted sulfur sticking to the quartz glass walls of the ampoules after quenching.

a NbS$_{2.0}$ elemental comp.: 1.89

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Sulfur	16	655	46.14	39.51	65.43	2.75	5.96
Niobium	41	616	70.63	60.49	34.57	4.33	6.13
		Sum	116.77	100.00	100.00		

b NbS$_{2.3}$ elemental comp.: 1.92

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
Sulfur	16	2394	29.89	39.90	65.80	1.33	4.46
Niobium	41	2291	45.02	60.10	34.20	2.04	4.54
		Sum	74.91	100.00	100.00		

FIG. 5. Results of the elemental analysis by SEM-EDX resulted in a nominal sulfur composition of (a) 1.89 for NbS$_{2.0}$ synthesized at 900 °C and (b) 1.92 for NbS$_{2.3}$ synthesized at 900 °C.