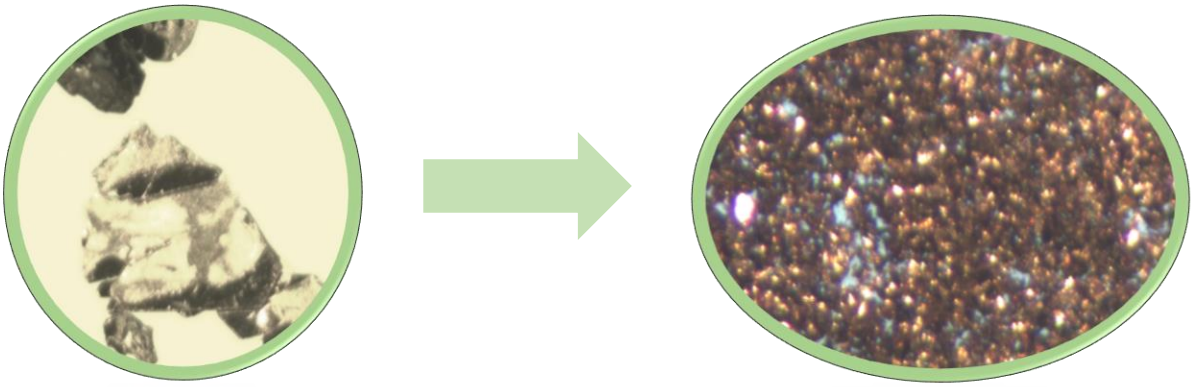
Indexing

Introduction

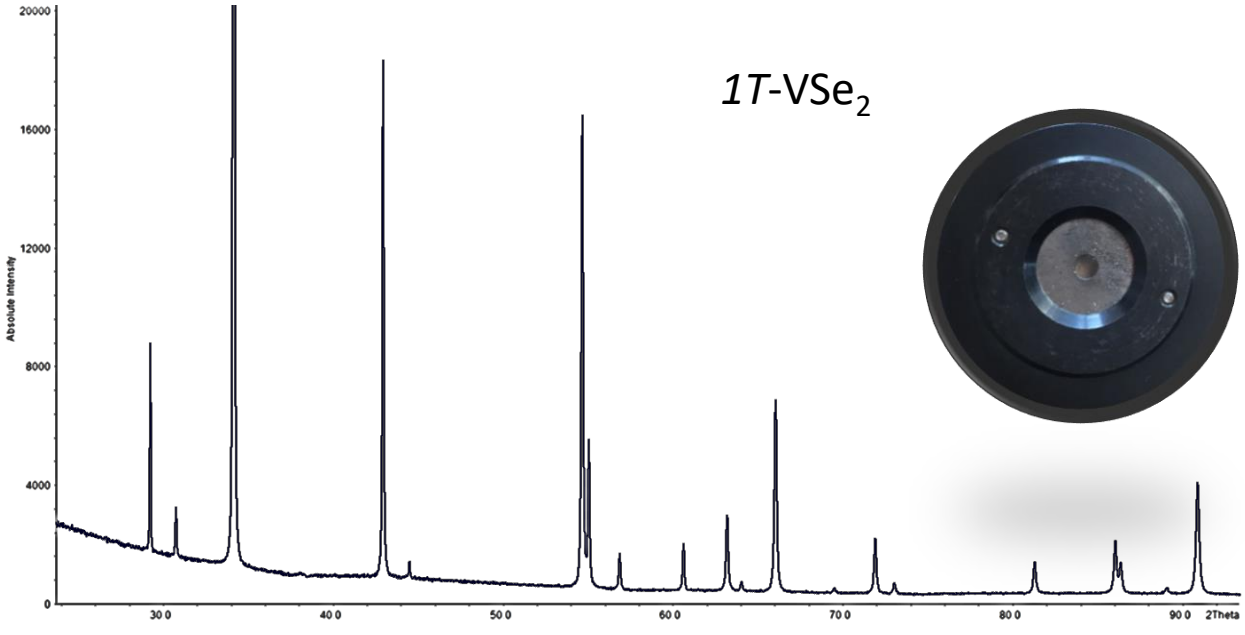
Several sample batches of *1T*-VSe_2 crystals were grown by chemical vapour transport (CVT) under varying growth temperatures, T_g, in the range of 550-700˚C. In order to inspect changes in the composition as a function of temperature, the determination of unit cell data for each batch was necessary. *1T*-VSe_2 belongs to the group of layered transition metal dichalcogenides (TMDs) which form crystal sheets rather than single crystals, making it impossible to carry out characterisation by single crystal XRD. This meant that PXRD was the method of choice for this problem.

Experimental details

Crystals were reduced in size to form fine particles with the use of a razor blade, and placed between two acetate foils in a transmission sample holder in preparation for PXRD analysis.



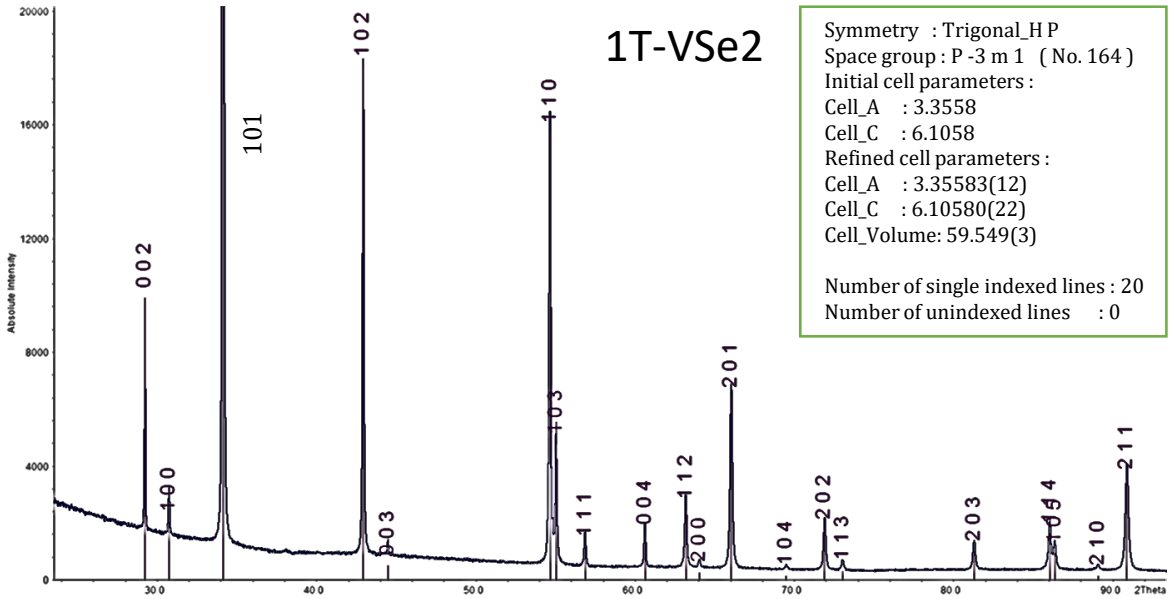
- Scan Mode: Transmission
- Diffraction: curved Ge (111) monochromated Cu-Kα_1 radiation (λ = 1.54060 Å)
- Generator: 40kV, 40mA
- Detector : Multi-Mythen (two DECTRIS 1K (1280 strips) detectors) / Moving/ Fixed Omega
- Scan range: 20˚- 93˚ in 2 Theta / 600sec times 3 measurement



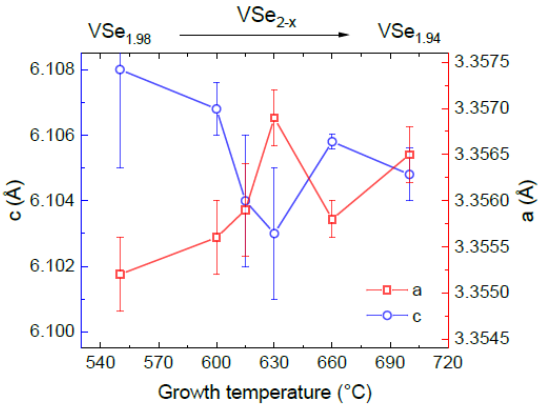
Indexing Procedure

This requirted the use of WinXPOW software [1]

- GRAPHICS peaks are assigned to the raw data
- Profile parameters and intensities from the measured PXRD pattern were determined through PATTERN FITTING
- The TREOR and DICVOL [2-3] indexing routines were used on the series of *1T*-VSe2 samples to INDEX&REFINE the data, with space group information being taken from the ICCD-PDF2 data base of previously published VSe2 phases.



Conclusion



Lattice parameters of $1T$-VSe_2 single crystals obtained from powder x-ray diffraction (PXRD). The left and right plot axes show the c- and a-axis parameters of the $P\bar{3}m1$ unit cell respectively. The top-axis labels shows a comparison with the overall variation of crystal stoichiometry (Se/V ratio) obtained from laboratory XPS measurements.

We found that the phase transition is suppressed in crystals grown at higher temperatures, which we suspected was due to defects/doping. Determination of unit cell characteristics could be used to support this hypothesis. Data from PXRD analysis helped us to eliminate possible defect scenarios e.g. interstitials between layers which would increase the c-axis

We found that VSe_2 has increased Se vacancies at higher temperature as the cause of the doping.

[1] WINXPOW: Powder Diffraction Software version 3.6.0.1 (Feb2018) STOE&Cie GmbH, Germany

[2] *1 P.-E. Werner, L.Eriksson, M. Westdahl: J. Appl. Cryst.* ***18****, 367 (1985)*

[3] *A. Boultif, D. Louër: J. Appl. Cryst.* ***37****, 724 (2004)*

[4] C. J. Sayers, L. S. Farrar, S. J. Bending, M. Cattelan, A. J. H. Jones, N. A. Fox, G. Kociok-Köhn, K. Koshmak, J. Laverock, L. Pasquali, E. Da Como; Physical Review Materials, **4**, 025002 (2020) 1-9