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# Thermal stability and phase transitions of TMDs investigated by in situ XRD

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## Introduction

Transition metal dichalcogenides (TMDs) have emerged as key materials for advanced technological applications. However, a critical gap persists in understanding their real-time structural evolution under extreme operational environments.

This work investigates the microstructural evolution and degradation of various TMDs ($MoS_2$, $TiS_2$, and $In_2Se_3$) using in situ high temperature X-ray diffraction (HT-XRD) under dynamic vacuum. The study provides a real-time map of thermal expansion, phase transitions, and the eventual reduction of these compounds at temperatures reaching up to 1100 °C.

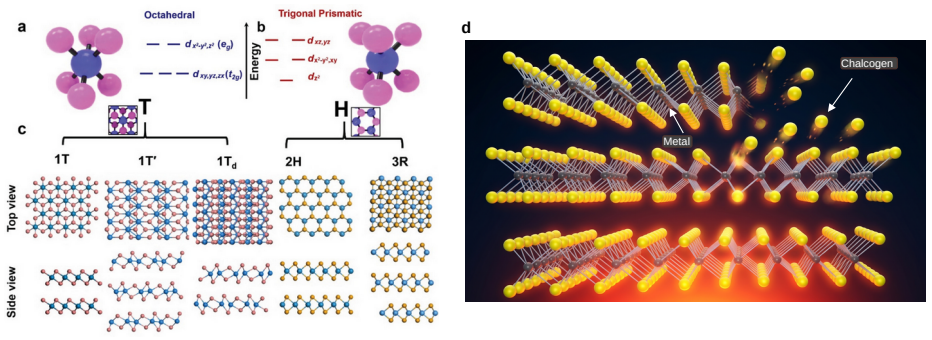



**Figure 1.** (a-c) Possible structures found in TMDs. (d) Illustration of the phase transition and the loss of chalcogen atoms due to heating of the material.

## Objective

To investigate the thermal stability, phase transitions, and structural degradation mechanisms of various TMDs under high dynamic vacuum (up to 1100 °C) via in situ X-ray diffraction, mapping the kinetic competition between thermal expansion, residual oxidation, and sublimation.

## Methodology

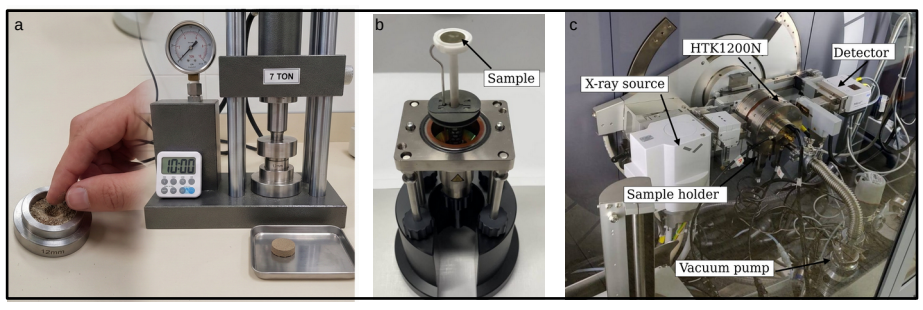



**Figure 2.** (a) Pressing of powdered TMDs to obtain a disk with a flat surface. (b) Securing the disk to the sample holder. (c) HTK1200N coupled to a diffractometer, together with a turbomolecular pump.

## Results and discussion

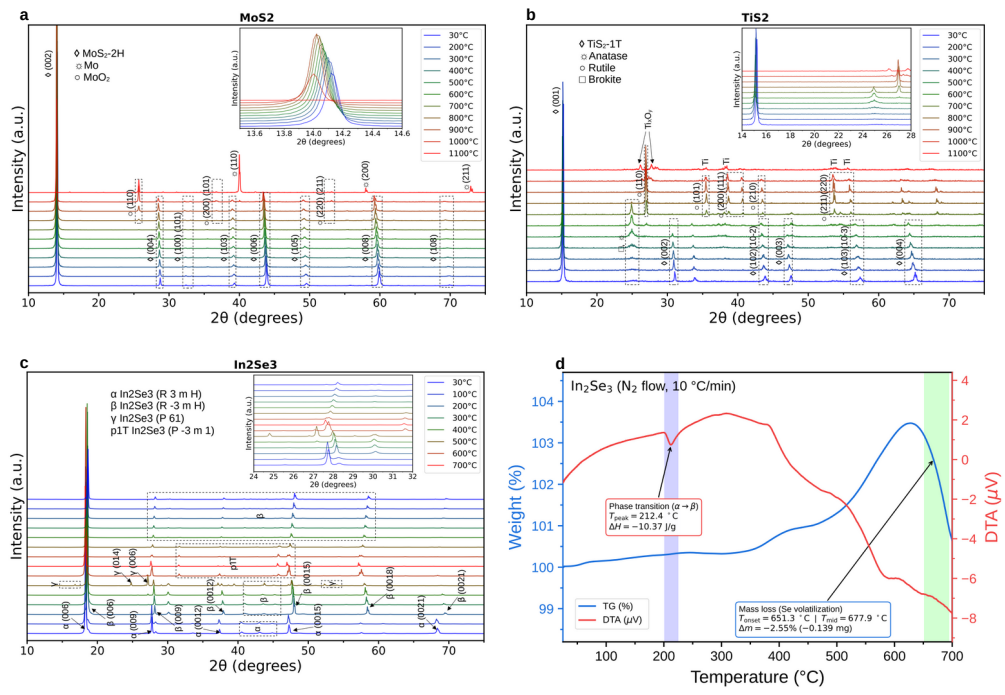



**Figure 3.** In situ thermal measurements. (a–c) HT-XRD under dynamic vacuum for MoS2, TiS2, and In2Se3, (d) Simultaneous TG-DTA curves for In2Se3.

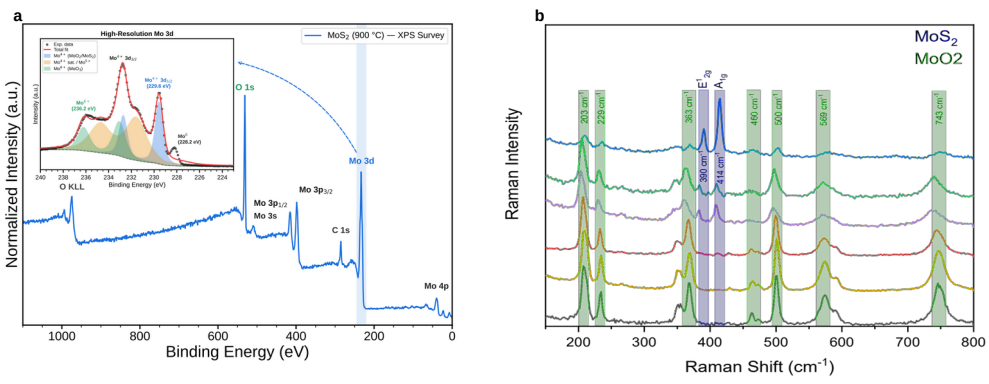



**Figure 4**. Ex situ post-treatment characterization at ambient conditions for MoS2 heated to 900°C. (a) XPS spectrum and (b) Raman spectra.

**Table 1.** Summary of thermal phase transitions, oxidation, and degradation pathways

| TMD | Phase Transitions | Oxidation Events | Degradation |
|---|---|---|---|
| $MoS_2$(2H) | -- | 900°C | 1100°C (Mo) |
| $TiS_2$(1T) | -- | 500°C | 700°C ($TiO_2$) |
| $In_2Se_3$(α) | 200°C (β)→ 500°C (γ)→ 600°C (p1T)→ ↓400°C (β) | -- | 800°C |

## Conclusions

- High-temperature stability under dynamic vacuum is governed by a competition between chalcogen volatization and residual oxygen reactivity.
- $In_2Se_3$ undergoes a well-defined polymorphic sequence (α→β→γ →p1T) and phase reversibility upon cooling.
- Combining HT-XRD and secondary techniques provides a robust framework to decouple intrinsic thermal transitions.

## References and Acknowledgments

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