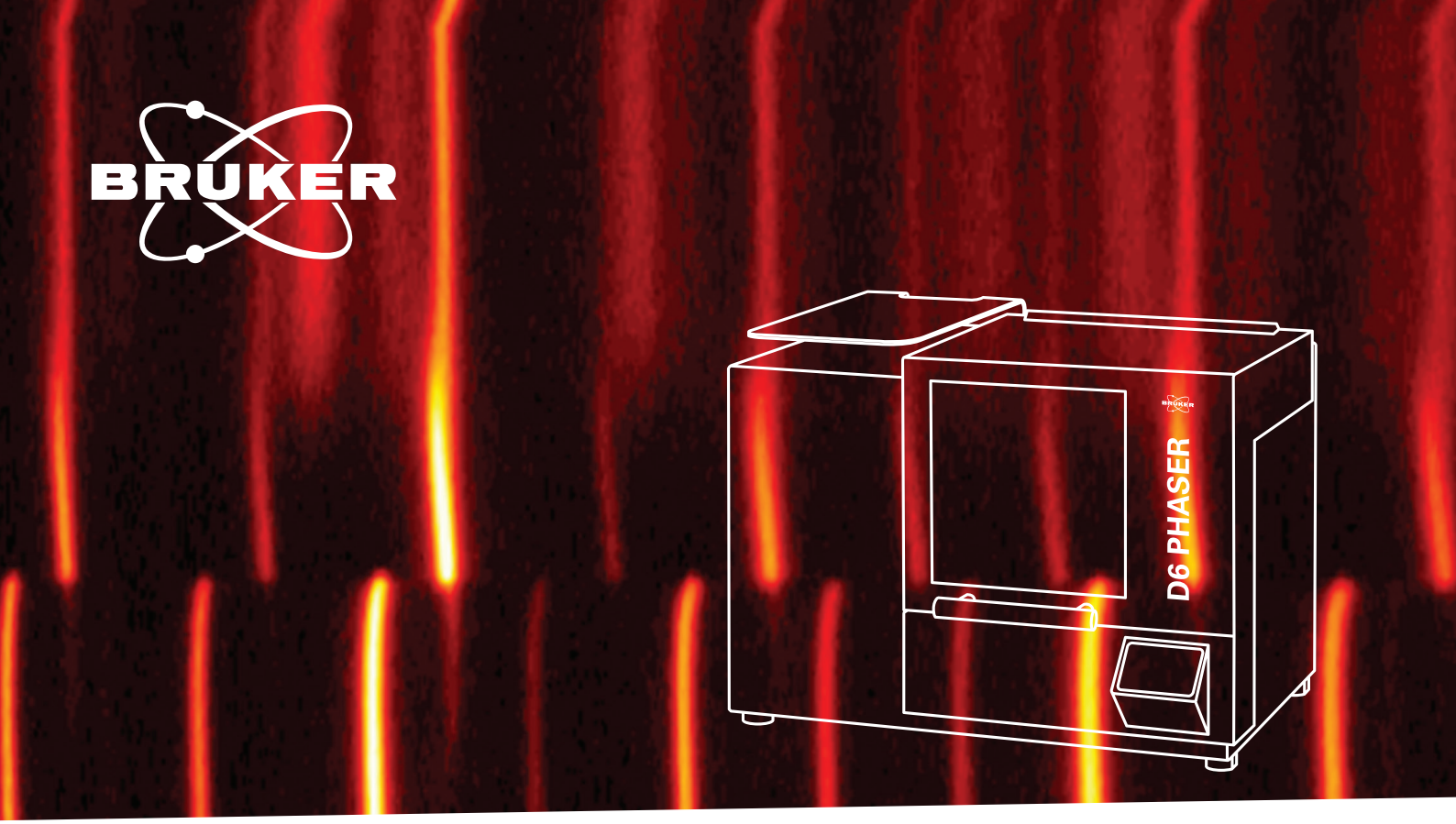

2D scan-view of phase transformations in Tungsten-phosphate, WPO_4.

X-RAY DIFFRACTION

D6 PHASER – Benchtop Non-ambient XRD

Application Report 37

Non-ambient X-ray powder diffraction (XRPD) is conveniently integrated into the D6 PHASER benchtop diffractometer. As the most powerful benchtop XRD on the market, the D6 PHASER is perfect for analyzing small sample volumes at fast data acquisition rates.This is achieved by combining up to 1200 W of power with a compact goniometer radius.

The system can accommodate Anton Paar's compact BTS 150/500 temperature stages. This allows users to measure samples from -10°C to 150°C and from room temperature to 500 degrees. The proprietary centric stage mount of the D6 PHASER allows the user to swap temperature stages with the other sample stages while maintaining the guaranteed angular accuracy of ±0.01°2Theta over the entire angular range. The temperature stages are seamlessly integrated into the D6 PHASER's DIFFRAC.SUITE measurement and evaluation software.

The BTS chambers are straightforward to use. Basic scans at set temperature can be conducted interactively from COMMANDER, while more sophisticated studies are fully supported by our "Plan - Measure - Analyze" workflow.

The WIZARD's Multi-Technique Table Editor (MTTE) combines sophisticated temperature profiles (consisting of set temperatures, hold times, cycles or ramps) with traditional scans or rapid still scans. Repetitive analysis can conveniently be assigned to push buttons in START JOBS.

Non-ambient XRPD studies that can be performed with the D6 PHASER include:

- Crystallization, melting, rec-crystallization
- Phase formation
- 1st and 2nd-order phase transformations
- Critical behavior analysis
- Thermal expansion and lattice parameter evolution
- Temperature induced structural variations or reconstructive changes at atomic scale

Figure 1 (above)
BTS 500 heating stage for the D6 PHASER

The vast amount of scan data generated during non-ambient experiments requires simple and automated data evaluation tools. Scans can be visualized with 2D (see cover illustration) and waterfall views in DIFFRAC.EVA, where the side view may show temperature or intensity profiles. Cluster analysis (Figure 2) helps to identify the most representative scans for phase identification. Furthermore, EVA provides profile analysis for the sequential fit of lattice parameters or individual peak properties.

Rietveld analysis in DIFFRAC.TOPAS reveals the atomic structure at high temperatures as well as profile and lattice parameter variations. While the batch mode can conveniently fit hundreds of individual scans, parametric refinements of the two-dimensional temperature-diffraction space unveil temperature related dependencies among the scans, such as e.g. the thermal expansion behavior of the lattice parameters.

The measurement data shown in this report were collected with a D6 PHASER 600 W, Cu radiation, no filter, 2.5° Soller collimators, primary fixed divergence slit 50 µm, scan range 20 to 90 °2Theta, time per scan 214 sec with the LYNXEYE XE-T detector in high-resolution mode. Temperature was varied with a BTS 500 from 30°C to 500°C at 5° increment.

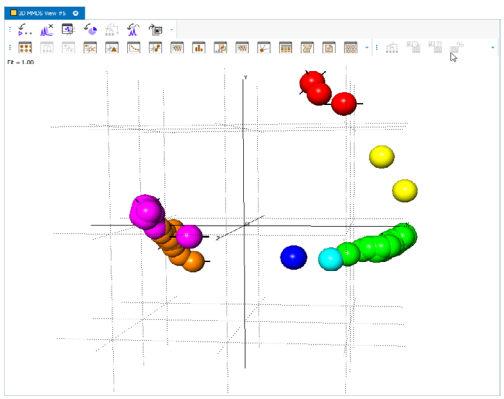

Figure 2
Cluster analysis MMDS view of temperature induced phase transformations in WPO_4.

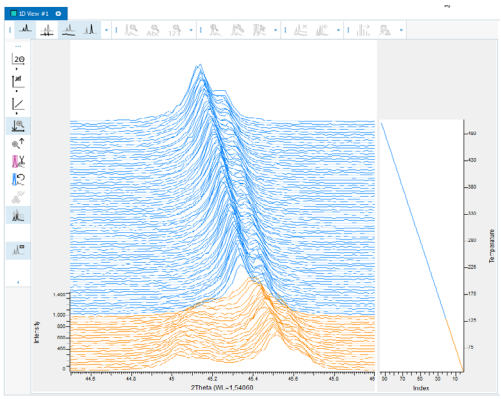

Figure 3
Stacked 1D scan-view and temperature profile (side view) for temperature induced peak splitting in perowskite. $BaTiO_3$.

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