DHS 900
HIGH TEMPERATURE INVESTIGATIONS WITH THE D8 DISCOVER WITH GADDS

Introduction

A number of different physical properties of crystalline materials vary with temperature. A classical example for this behaviour is the continuous or discontinuous change of the crystal lattice spacings. Typically these effects are studied in-situ by X-ray diffraction techniques.

Different types of low- and high temperature high vacuum chambers are used to obtain reliable data of changes in the lattice spacing as a function of the sample temperature. Because of the design, the weight and the stiffness of vacuum and cooling water connections, the chambers are typically mounted to the fixed base in the centre of a vertical theta/theta goniometer.

The new Anton Paar DHS 900 (dome heating stage) with its extraordinary low weight and pocket-sized design allows for the mounting of a high temperature chamber to an Eulerian cradle or XYZ-sample stage of a compact laboratory X-ray diffraction system. Furthermore, the dome-shaped design of the X-ray window of the DHS 900 allows access to the entire diffraction space of a sample investigated in reflection mode. This makes the DHS 900 the first choice for advanced material research applications with the D8 DISCOVER either for Material Research or with GADDS.

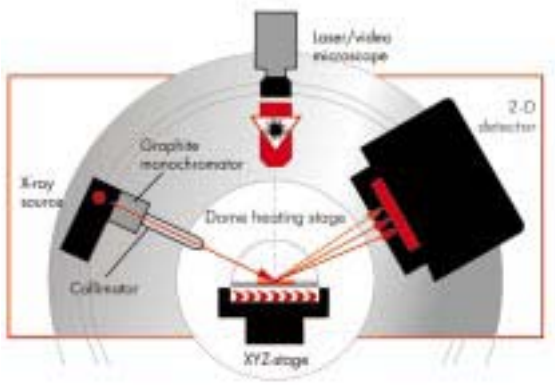

Fig. 1: Beam path schematics of the D8 DISCOVER with GADDS. The dome heating stage is mounted on the XYZ-sample stage. The laser/video microscope is used for the precise aligning of the sample in the X-ray beam with the dome removed.

The article reports measurements executed with the D8 DISCOVER with GADDS for Microdiffraction. NIST standard reference material 1976 Corundum was used for the investigations performed with Co-Kα radiation.

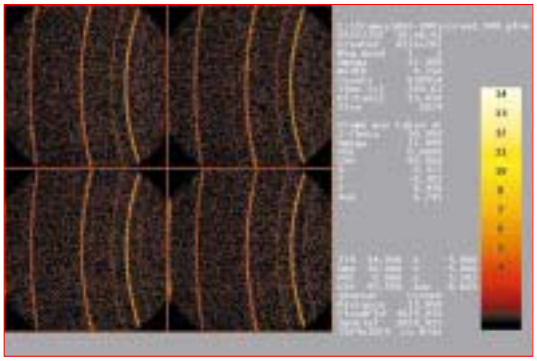

Fig. 2: 2-dimensional diffraction pattern of NIST SRM 1976 obtained at RT (upper left),500°C (upper right), 600°C (lower left) and 700°C (lower right), respectively. The measurements were performed using a 2-Theta setting of 50°. Each frame covers a 2-Theta range from 32.4° to 67.4°. The background scattering from the dome is substracted.

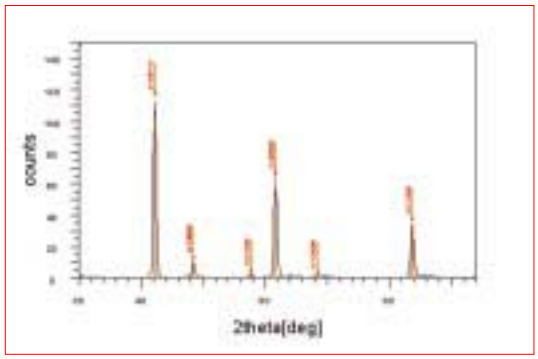

Fig. 3: Intensity versus 2-Theta plot obtained at RT. The plot was determined by chi-integration along the Debye rings. The background was subtracted. The red lines indicate the positions of the ICDD 46-1212 Corundum pattern.

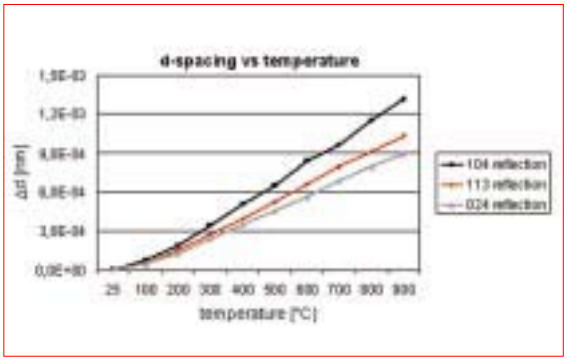

Fig. 4: d-spacing change of NIST 1976 Corundum sample with changing sample temperature. Black 104 reflection, red 113 reflection, and grey 024 reflection.

Instrumentation

- D8 DISCOVER with GADDS
- 1.8 kW spot focus Co X-ray tube
- flat graphite monochromator
- 500 µm collimator
- XYZ-sample stage
- Anton Paar DHS 900 heating stage
- area detector

Conclusion

The DHS 900 enables an expansion in the area of advanced material research applications for the D8 DISCOVER with GADDS. Its ease of use and handling, along with its extraordinary compact and lightweight design enables high-temperature work without removal of the dedicated sample stages from the goniometer (e.g. the XYZ or Eulerian cradle).

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