APPLICATION NOTE

No. AN41170

In-Situ measurements on ARL EQUINOX 3500 using HTK16N

Authors: Dr. Simon Welzmiller, Dr. Henry Pilliere, Thermo Fisher Scientific

Introduction

The most commonly used technique to investigate the crystallographic structure of matter is X-ray diffraction, especially in in-situ experiments. Reactions or phase transitions can appear when materials are exposed to non-ambient conditions like high/low temperature which could lead to significant changes in the crystallographic structure.

The real-time detector technology of the Thermo Scientific™ ARL™ EQUINOX product line is well suited to perform rapid XRD measurements to study structural phase transitions of materials at high temperature. The Anton Paar HTK16N strip heater chamber can perform experiments up to 1600°C which, coupled with a simultaneous full pattern XRD capability, allows to follow dynamic studies at high temperature much faster and in real time.

This application note presents an example of such a study with a Thermo Scientific™ ARL™ EQUINOX 3500 coupled with a HTK16K chamber to illustrate the capabilities of this setup.

Instrument

The ARL EQUINOX product family represent a portfolio of XRD instruments from simple, easy to use bench-top systems for routine analysis to more advanced floor-standing, research grade systems.

The Thermo Scientific™ ARL™ EQUINOX 3000/3500 instrument series are floor-standing, research-grade X-ray diffractometers which employ a 3 kW high voltage generator using standard sealed tubes (Co, Cu or Mo). Both, high resolution Ge (111) monochromators as well as mirror optics (focal / parabolic) for high flux are available.

The ARL EQUINOX 3000/3500 (c.f. Figure 1) provides very fast data collection compared to other diffractometers due to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time. Therefore, it is well suited for in-situ measurements as it is possible to follow sample changes in real time especially if mirror optics are used (c.f. Figure 2).

Figure 1: ARL EQUINOX 3500 diffraction system

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Experimental

An ARL EQUINOX 3500 with Cu Kα radiation and focal mirror optics was used to measure the phase transitions of α-Quartz (RT - 1200°C; 20°C/min; 10 sec acquisition time) and $RbNO_3$ (160°C – 168°C / 215°C – 223°C / 287 – 295°C; 10°C/min; 60 sec acquisition time).

Results

Due to the CPS detector technology an overview measurement in reflection of α-Quartz from room temperature to 1200°C is possible in below 2 h. Phase transitions from α-Quartz to α-Quartz (~600°C) and Tridymite (~1150°C) are clearly visible. (c.f. Figure 3) Due to the speed of measurement there are some deviations from literature values.

For more precise determination of the transition temperature a heating program with a slower heating ramp is required. $RbNO_3$ exhibits three phase transitions between RT and 300°C. In order to determine precise transition temperatures three measurement steps with slow heating ramps of 10°C per minute were used (c.f. Figure 4). The transition temperatures are in good comparison to the literature values (164°C / 220°C / 291°C) indicated by the white lines in Figure 4.

Conclusion

The ARL EQUINOX instrument line is perfectly suited for in-situ studies due the unique CPS real time detection technology. Measurements with high dynamics (e.g., for overviews or fast sample changes) are easily possible (c.f. measurement of quartz) are possible as well as precise measurements using slow heating ramps and several steps (c.f. measurement of $RbNO_3$).

Figure 2: HTK16 installed on ARL EQUINOX 3500 instrument

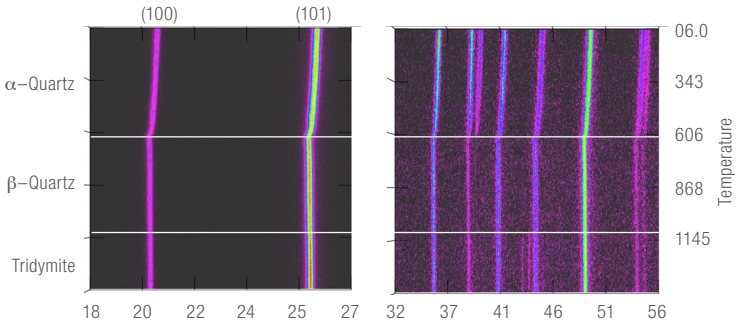

Figure 3: Measurement of α-Quartz from RT -1200°C (Phase transitions indicated by white lines); zoom into (100) and (101) reflections left side

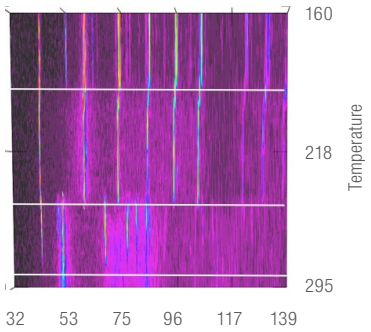

Figure 4: Measurement of $RbNO_3$ between 160°C and 295°C

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