STOE
SINCE 1887

APPLICATION REPORT
STOE INSITU HT2

Measurements of a nano-crystalline Iron(III) oxide sample performed with a MYTHEN1K in simulated dual detector mode on a STOE STADI P COMBI Diffractometer using the STOE INSITU HT2

1. Experimental Setup

All measurements were performed on a STOE STADI P COMBI (Mo-$K_{\alpha 1}$ radiation 0.70930(9) Å) using the STOE INSITU HT2 high temperature reaction chamber. This chamber was designed to study solid state and solid state - gas reactions in capillaries in a temperature range between RT and 1600°C on a vertically mounted transmission diffractometer, e.g. the STOE STADI P or STADI MP.

The high temperature chamber consists of a cylindrical double walled, water cooled body with an entrance collimator for the primary beam and an exit window with 90° opening for the diffracted X-rays (covered with Kapton® foil). The heating element consists of a coiled graphite rod, which is clamped between the lid and base plate and contacted by a thermocouple directly. To reduce effects of preferred orientation the sample can be oscillated by a motor. The STOE IN SITU HT2 is fully computer-controlled in the newest WinX^{Pow} software version.

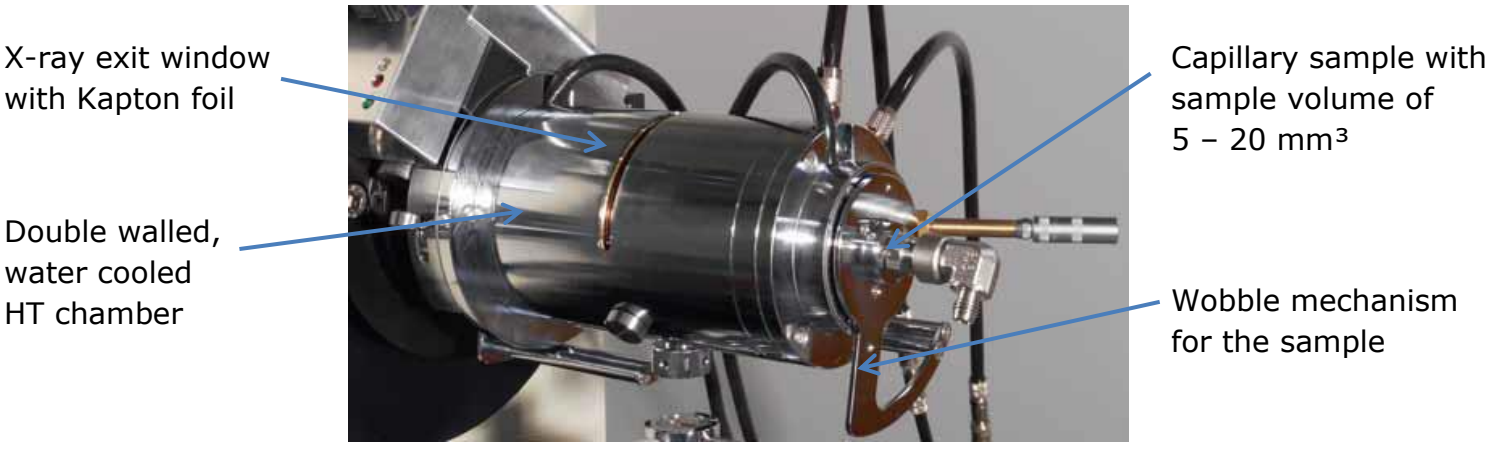

Figure 1: STOE INSITU HT2 high temperature reaction chamber installed on a vertical STOE STADI P transmission diffractometer using focused pure Molybdenum $K_{\alpha 1}$ radiation.

2. Results

a. Measurements with Mo-$K_{\alpha 1}$ in the HT2 in-situ measurement cell under ambient conditions

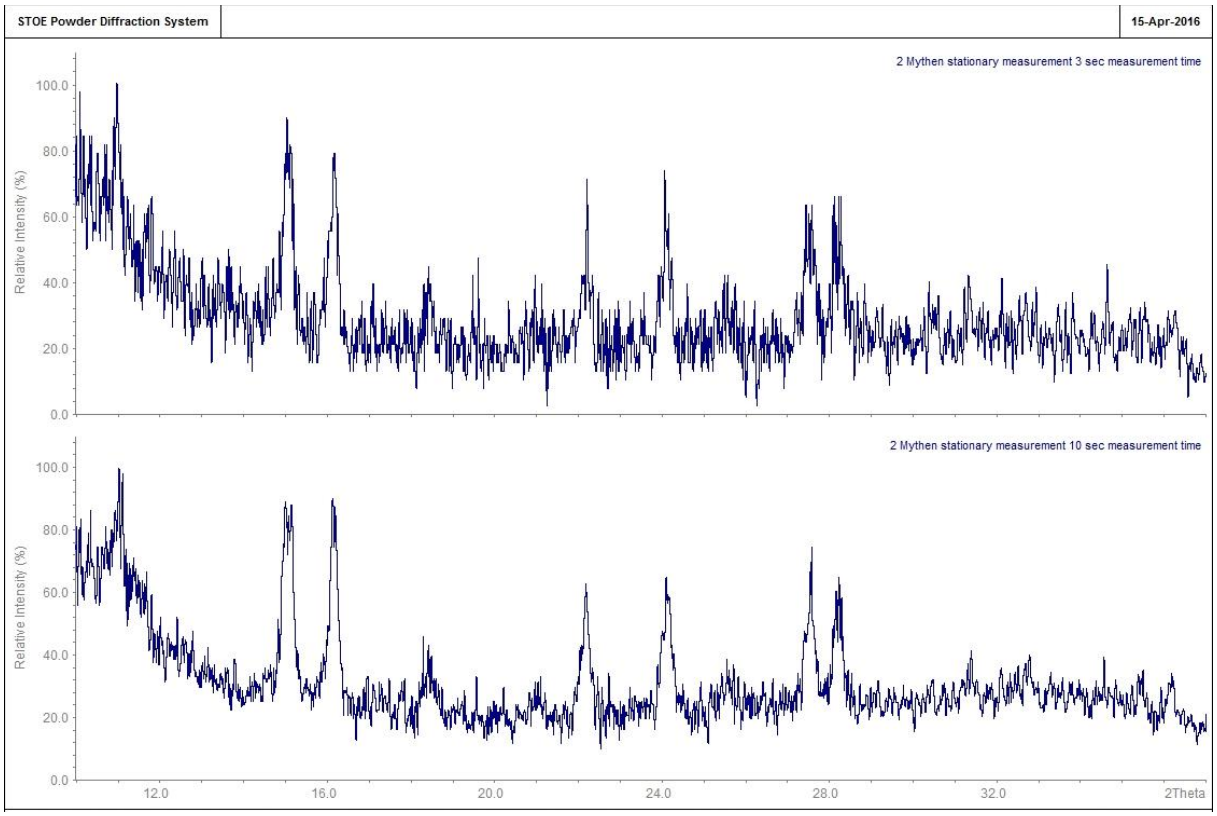

Figure 2: Measurements simulating a MYTHEN1K dual detector array in stationary measurement mode with 3 sec measurement time (top) and 10 sec measurement time (bottom)

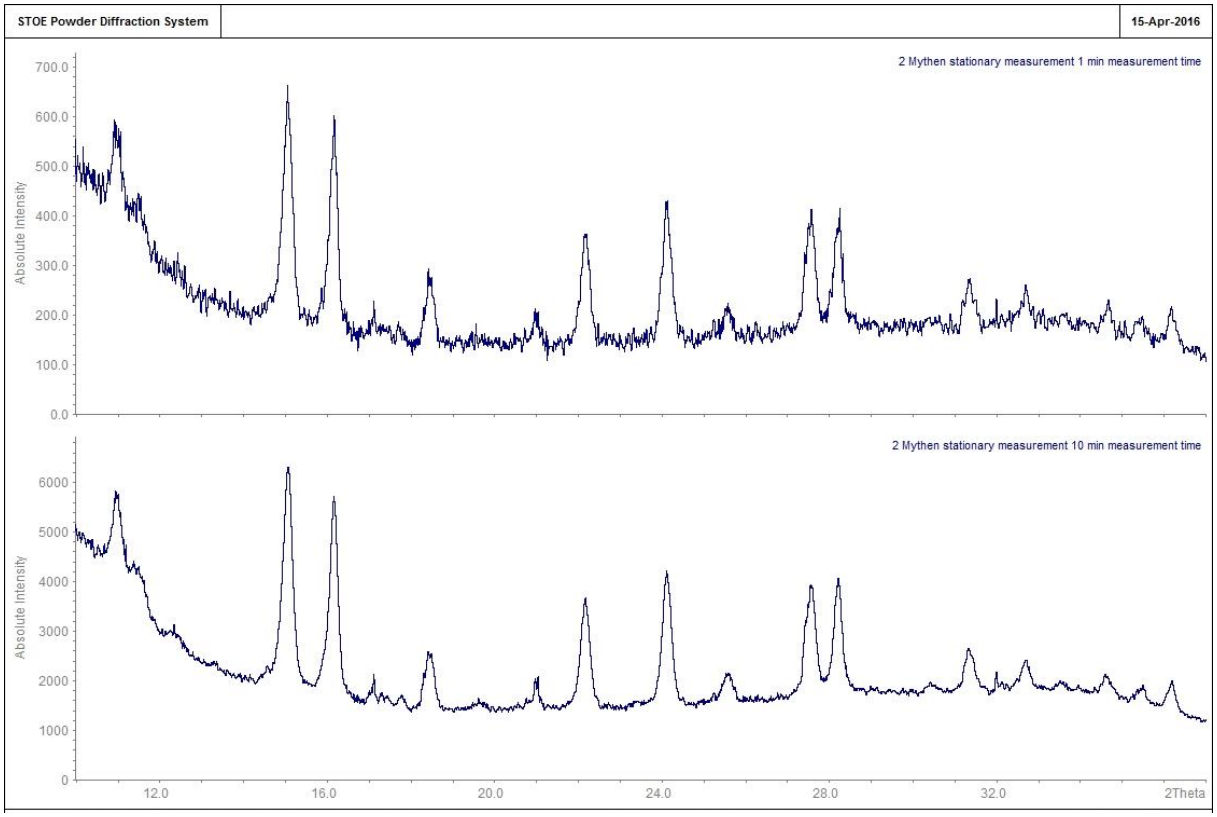

Figure 3: Measurements simulating a MYTHEN1K dual detector array in stationary measurement mode with ~1 min measurement time (top) and ~10 min measurement time (bottom)

The HT2 in-situ measurement cell uses capillaries with 2.0 mm outer and 1.0 mm inner diameter made from silica or sapphire depending on the temperature of the heating experiment. The HT2 has to be mounted on a vertically installed STOE diffractometer and can provide temperatures up to 1600 °C with a measurement range of 90° 2θ. The system works best with fast MYTHEN1K detectors and, in case of Fe containing samples, Mo-$K_{\alpha 1}$ radiation. Figure 2 shows diffraction pattern measured with 3 sec and 10 sec measurement time. As there is no read-out time for MYTHEN1K detectors, the measurement time is equal to the exposure time in this case. It is possible to collect evaluable data in a 3 sec diffraction experiment and after 10 sec the results are satisfactory for phase analysis. If more precise data is needed, perfect measurements are done in ~1 to 10 min (c.f. Figure 3). If data up to higher 2θ angles is needed measurements in a two-step measurement mode covering a 2θ range between ~2° and ~77° are possible (c.f. Figure 4). The data shown in both figures simulates a Multi-MYTHEN1K detector using 2 MYTHEN1K

modules, which is under development by STOE right now. STOE will provide also the possibility to upgrade Single-MYTHEN1K detectors to Multi-MYTHEN1K detectors.

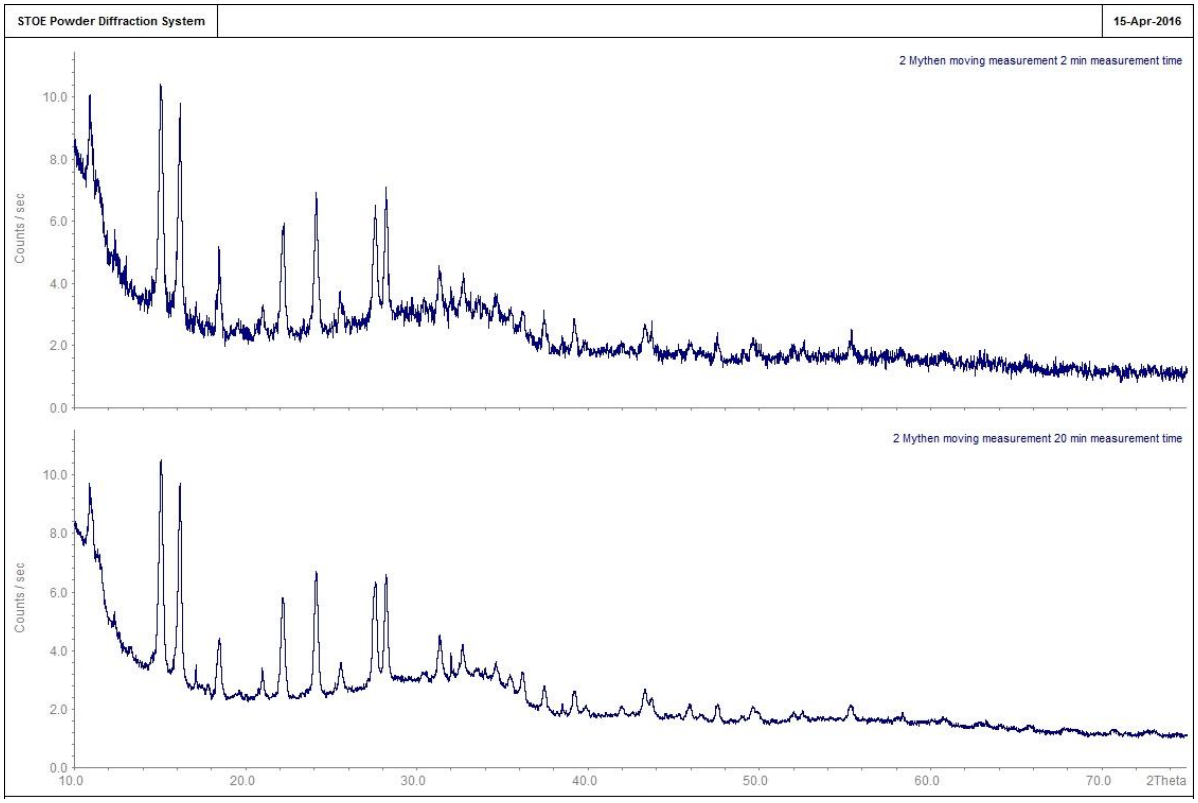

Figure 4: Measurements simulating 2 MYTHEN1K detectors in a two-step measurement mode; 2 min measurement time (1 min exposure time each step) top and 20 min (10 min exposure time each step) bottom

3. Conclusion and Outlook

Using Mo-$K_{\alpha 1}$ radiation and a (Multi-)MYTHEN1K detector, fast measurements of a nano-crystalline Fe_2O_3 sample are possible using the STOE HT2 in-situ measurement cell already under ambient conditions, and it is most likely to observe also fast reactions or phase transitions in this sample when performing non-ambient measurements with that set-up.