LABNOTE
1ST MEASUREMENT WITH THE NEW STOE INSITU HT2
THE OXIDATION OF Ru IN A CARBON SUPPORTED Pt-Ru CATALYST

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Figure 1: STOE INSITU HT2

Preliminary tests with the STOE INSITU HT2 (Figure 1) to observe the temperature calibration by investigating the phase transitions from α to β-quartz (Figure 2) at 573°C and from orthorhombic to cubic celestine at 1150°C (Figure 3) had been carried out with a steady flow of 0.06 l/min N_2 in a quartz capillary with 1.5mm inner diameter for the first, and sapphire capillaries (1 mm diameter) for the latter experiment.

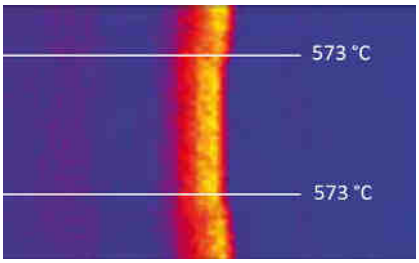

Figure 2: Phase transition from α to β-Quartz at T=573°C

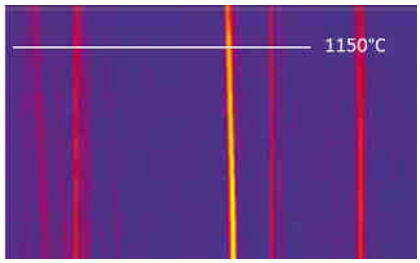

Figure 3: Phase transition from Celestine at T=1150°C

Though these results had already been very encouraging, the combination of the new STOE INSITU HT2 and reactive gases had still to prove functionality. Therefore a series of test measurements have taken place in the laboratories of the Applied Physical Chemistry group at the FU Berlin.

30 to 40 mm^3 of a carefully ground carbon supported Pt-Ru catalyst [1)] has been fixed between two pads of rock wool in the middle of a quartz capillary with 1.0 mm inner diameter. Then the capillary has been mounted in the STOE INSITU HT2, a flow of 0.5 l/min N_2 has been led through the chamber to protect the graphite heating element from ignition. O_2 as reactive gas streamed through the capillary with a flow of 0.03 ml/min. The heating rate has been chosen to 5°C/min.

X-ray patterns from the catalyst exposed to O_2 at room temperature, 200°C, 250°C and 300°C have been taken on STOE STADI P powder diffractometer with a sealed molybdenum tube, Ge(111) monochromator

yielding pure Mo $K\alpha_1$ radiation and a Dectris MYTHEN 1K detector (1mm chip), the X-ray patterns are shown in Figure 4.

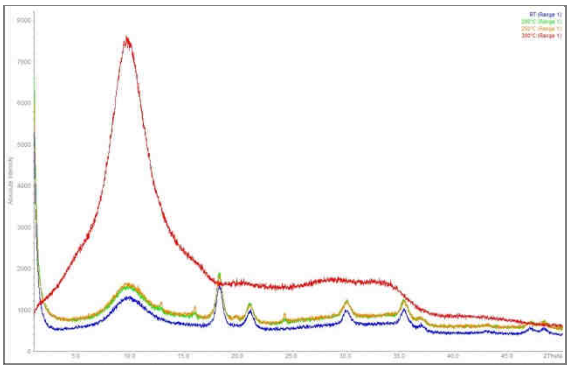

Figure 4: X-ray pattern at RT (blue), 200°C (green), 250°C (orange) and 300°C (red)

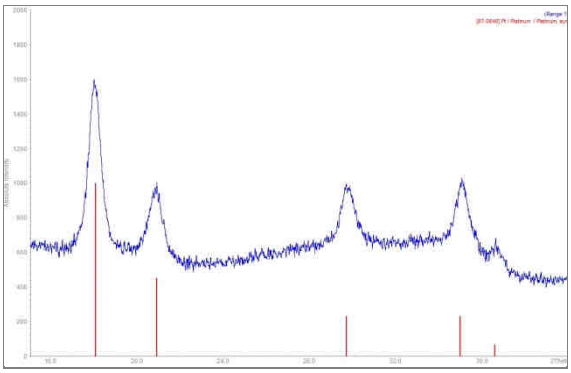

Figure 5: Pattern at RT with the red markers for Pt from the ICDD data base

Due to the catalyzing effect of Pt, an exposure to pure O_2 at 300°C led to a complete combustion of the sample. The big hump at 10° 2θ is a signal from the capillary material.

The measurement at room temperature (Figure 5) shows only the reflections of the rather crystalline Pt - Ru nanoparticles which are too small to yield sharp reflections.

At 200°C and 250°C the oxidation (and tempering) of the Ru particles starts, additional reflections appear for RuO_2 and Ru (Figures 6 and 7).

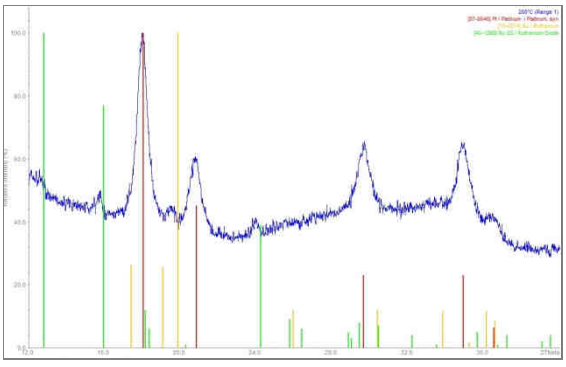

Figure 6: X-ray pattern at 200° (blue) with the markers for Pt (red), Ru (yellow), and RuO_2 (green)

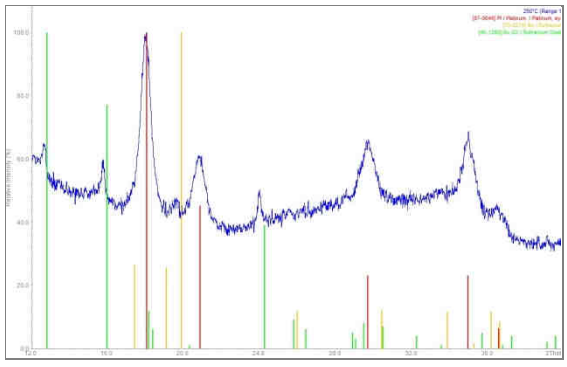

Figure 7: X-ray pattern at 250° (blue) with the markers for Pt (red), Ru (yellow), and RuO_2 (green)

As a summary, it could be demonstrated that the STOE INSITU HT2 fulfills all requirements to be the first commercially available in-situ heating chamber in Debye-Scherrer mode for small sample amounts in a temperature range from RT to 1600°C and gas flows (reactive or protective) between 0.01 to 0.1 ml/min.

The STOE IN SITU HT2 fits on all vertical mounted STOE STADI P and STADI MP diffractometers and is fully computer-controlled in the newest WinX^{Pow} software version.

1) C. Roth, N. Martz, H. Fuess, 'Characterization of different Pt-Ru catalysts by X-ray diffraction and transmission electron microscopy', *Phys. Chem. Chem. Phys.* **3** (2001) 315-319.