Experimental report

09/10/2019

Proposal: **5-24-621** **Council:** 10/2018

Title: Development of single crystal gas pressure cells for in situ hydrogenation reactions

Research area: Chemistry

This proposal is a new proposal

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Samples: Si

Instrument	Requested days	Allocated days	From	To
D20	2	1	20/06/2019	21/06/2019

Abstract:

The proposal focusses on testing gas pressure cells for solid gas reactions followed by in situ neutron scattering. These reactions are getting high attention due to far-reaching applications. For example, the synthesis of functional materials, which can be used for hydrogen storage or proton conductivity, as used for renewables and fuels cells. Two recently developed gas pressure cells, based on previous cells are presented and should be tested. They are developed for the D20 diffractometer using deuterium pressure. A newly designed environment for the currently used sapphire single crystal sample holder should reduce mechanical tension leading to higher pressure stability. The second developed cell separates the inner sample holder from the outer pressure holding element. This allows a lower background contribution of the sample holder to the diffraction pattern. The pressure holding tube does not contribute to the diffraction pattern as all reflections are filtered out by the radial collimator at D20. The increased gas volume and thus high background contribution should be reduced by using Gd2O3 vanished shades inside the reaction chamber.

Development of single-crystal gas-pressure cells for *in situ* hydrogenation reactions

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The experiment was part of the development of sapphire single-crystal gas-pressure cells for *in situ* neutron powder diffraction on solid-gas reactions. Neutron absorbing Gd_2O_3 containing varnish was tested successfully to prevent the activation of stainless-steel parts of the gas pressure cells.

For both gas pressure cells (see proposal text), the width for the opening of the primary beam has been varied. The reflection-to-background ratio stays nearly constant for about 10° in case of the double-walled cell (Fig. 1, top left) and 20° for the adapted cell (not shown), respectively. This will enable a proper alignment procedure without data quality losses for both cells.

Additionally, an inlet for the double-walled cell to reduce the gas volume in the reaction chamber has been tested with a silicon powder sample in an aluminium crucible (Fig. 1, top right). The background, mainly caused by the incoherent scattering of hydrogen gas, is reduced by 50%. One parasitic reflection (2θ = 40°) has been observed due to a hole in the neutron absorbing varnish.

Finally, four new sapphire single crystals have been tested by ω scans. As expected, the background scales with the wall thickness. Fig. 1 (bottom left) shows an example of a sapphire suitable for *in situ* neutron diffraction, Fig. 1 (bottom right) shows an example for an unsuitable one.

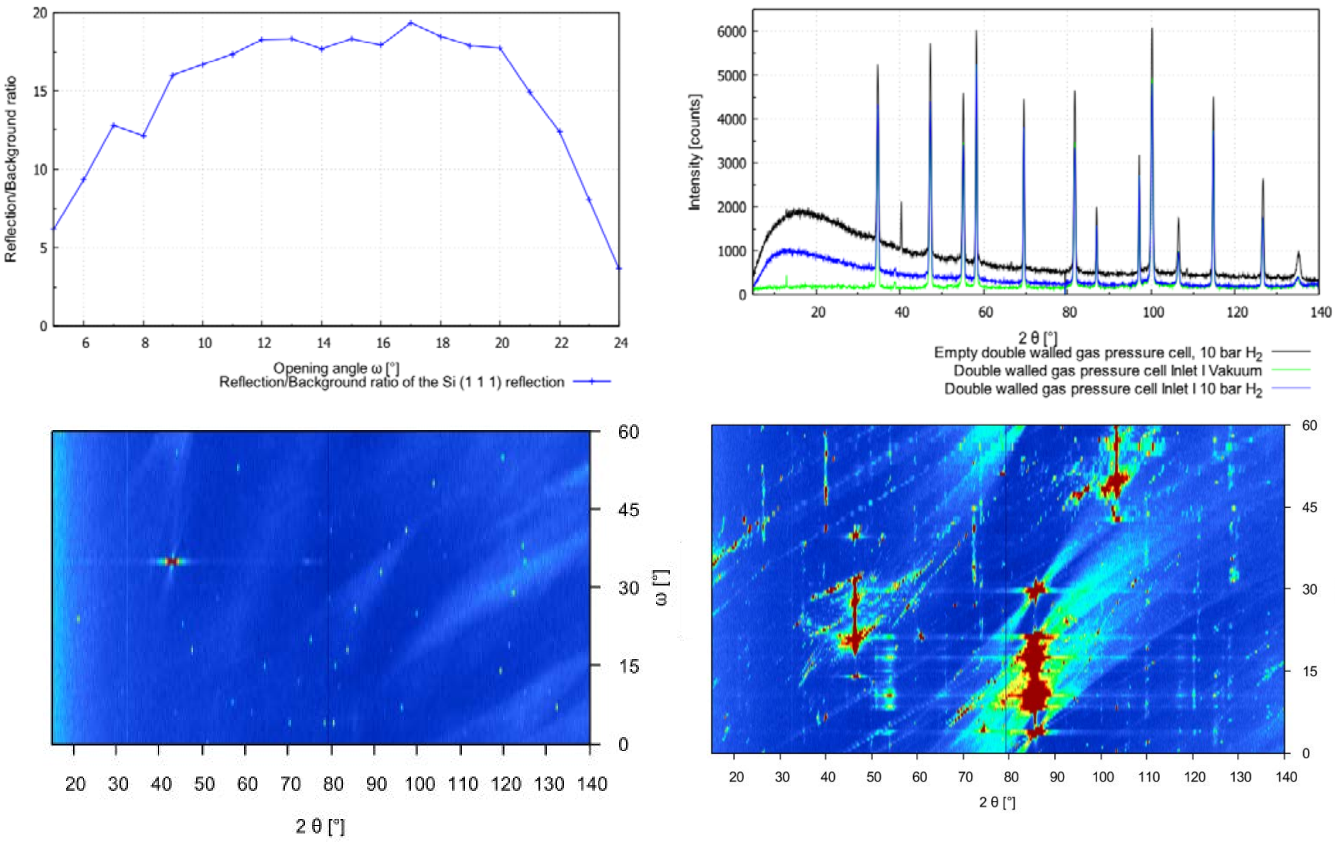

Figure 1 Effect of the opening angle of the double walled cell on the reflection-to-background ratio for the Si (111) reflection (top left), effect of an Inlet for the double walled cell on background at various hydrogen pressures (top right), ω-scan of a sapphire crystal suitable for in situ neutron diffraction (bottom left) and ω-scan of a sapphire crystal unsuitable for that purpose (bottom right), intensity in false colours