Experimental report

17/03/2016

Proposal: **INTER-310**

Council: 4/2015

Title: Sample holder capable of delivering electric stimuli to the sample

Research area:

This proposal is a new proposal

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Samples: clays

Instrument	Requested days	Allocated days	From	To
IN13	2	2	20/10/2015	22/10/2015

Abstract:

EXPERIMENTAL REPORT

EXPERIMENT NR. **INTER-310** INSTRUMENT **IN13**

DATES OF EXPERIMENT **20-21 October 2015**

TITLE **Sample holder capable of delivering electric stimuli to the sample.**

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ABSTRACT: Recently a new flat sample holder for high voltage experiments on backscattering instruments has been designed by Diallo [1], and reproduced with adaptations by R. Ignazzi at the Institute Laue-Langevin (ILL). Here we report the experiment conducted on IN13, where a DC electric field was applied on a dry clay sample, in order to test if the experimental setup worked properly.

This experiment was performed during R. Ignazzi's internship at the ILL.
The aim of the experiments were to test (i) the use of a Cadmium (Cd) and a Boron (B) masks, (ii) the experimental setup in vacuum and with the neutron beam and (iii) if there is any difference in the elastic fixed window signal due to the electric field (EF) stimuli.
The first test performed was the one comparing the B and the Cd masks. A mask is needed in our case to shield the neutrons from the teflon parts of the sample holder, used to electrically isolate the different parts of the EF-cell, in order to not contaminate the neutrons scattered from the sample with those scattered by the teflon. From Figure 1, which shows the raw elastic signal (normalized to the monitor) of the empty EF-cell with the two masks, it can be seen that the B mask is almost as effective as the Cd mask. The only difference is that the background is higher at low Q's for the B mask. Therefore in order to determine diffusion coefficients the Cd mask is more suitable. Here we note thar the B-mask tested contained hydrogen.

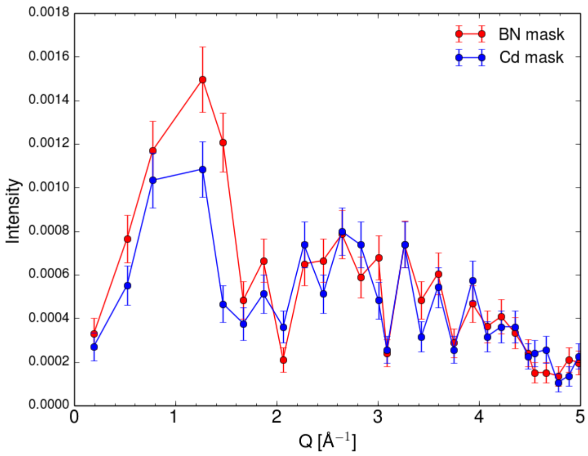
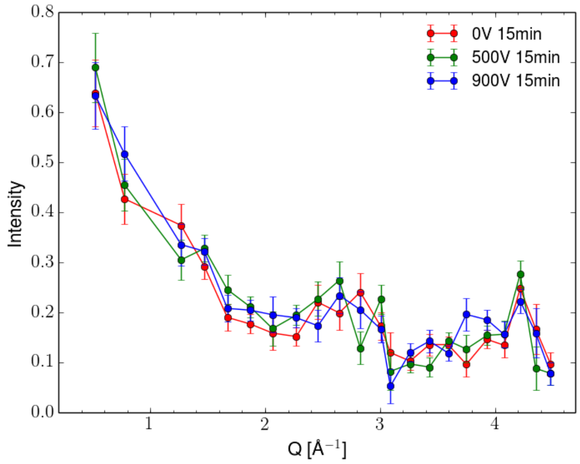


Figure 1: Comparison of the background signal of the empty cell with the Cd mask (blue) and the B mask (red).

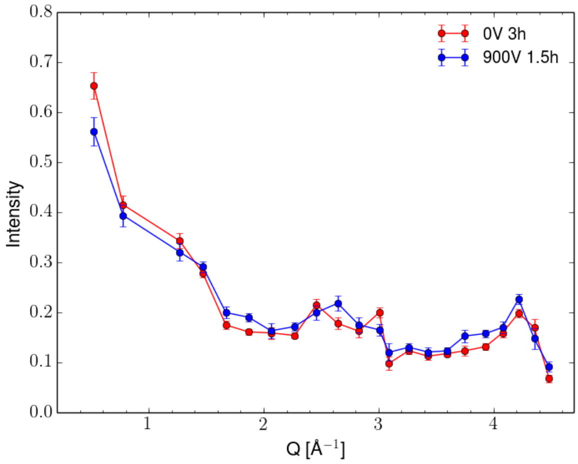
The measurements were performed using a NaFh clay sample (Na-fluorohectorite clay, a synthetic smectite clay [2]), which was equilibrated at an undefined RH level (the sample was exposed to air to transfer it to the EF-cell). To start, we connected the high voltage power supply (HIV), in order to see how much voltage the clays could resist to, then a pushed vacuum ($\sim 10^{-5}$mbar) was applied to the cryostat. The HIV was connected to the instrument control software NOMAD, which permitted us to monitor voltage and current, as well as remotly control the HIV power supply.
After solving different experimental problems, we performed a 15min measurement with 1.0kV/mm, a 15min measurement with 1.8kV/mm and a longer measurement with 1.8kV/mm (all elastic fixed window measurements, in a window of 8μeV). Due to an increase of the current to 4.0mA, probably due to a small change in the vacuum, the last run was interrupted after about 1.5 h. This caused the heating of the system, due to Jules effect:

$$P = IV,$$

where P is the power converted into heat, I is the current and V is the applied voltage.
We took measurements with 0kV/mm for several hours as well, both before and after applying the electric stimuli. Figure 2 shows the elastic intensity as a function of Q, where all raw data were normalized to the monitor, the background subtracted, the bad detectors removed and the data normalized using the vanadium scan. These data were collected for about 15 minutes (Figure 2a) or 3 hours at 0V and 1.5 hours at 900 V (Figure 2b).
In Figure 2a, due to the short counting time, no difference between the data at different voltages could be observed. In Figure 2b though it is seen, that at low Q there seems to be a difference. In order to confirm such observation another measurement ought to be performed focusing at the low Q range.



(a) Elastic intensity of the clay sample for 15 minutes for three voltages, 0V (red), 500V (green) and 900V (blue).



(b) Elastic intensity of the clay sample for two voltages, 0V (red) for ~3 hours and 900V (blue) for ~1.5 hours.

Figure 2: Data taken with the electric field applied on the NaFh clay sample.

From these experiments we can conclude that, if we do not have a B mask without hydrogen in it, the Cd mask is more appropriate if we want to investigate the scattering signal at low Q. Also, the setup is very sensitive, any slight change on vacuum can destabilize the electric field and proper isolation and control of all the elements is essential to have a fully working setup. At last, a small difference between the NaFh clay sample with and without the field is observed. Further experiments at low Q are underway to confirm this effect.

[1] S.O. Diallo, E. Mamontov, N. Wada, S. Inagaki and Y. Fukushima, Physical Review E **86**, 021506 (2012)

[2] L. Michels, J.O. Fossum, Z. Rozynek, H. Hemmen, K. Rustenberg, P.A. Sobas, G.N. Kalantzopoulos, K.D. Knudsen, M. Janek, T.S. Plivelic and G.J. da Silva,Scientific Reports, **5**, 8775 (2015)