

Experimental Report

05/09/2014

Proposal: INTER-280 **Council:** 10/2012
Title: Internal time on IN11
This proposal is a new proposal
Research Area:

Main proposer: FOUQUET Peter

Experimental Team: BAHN Emanuel

Local Contact: FOUQUET Peter

Samples: Graphite
C and hydrogen
H2

Instrument	Req. Days	All. Days	From	To
IN11	5	5	19/07/2013	24/07/2013

Abstract:

Inter 280 – Experimental Report

Experimental Team: Peter FOUQUET, Emanuel BAHN

Local Contact: P. FOUQUET

Hydrogen (molecular) diffusion on carbons

This experiment was a proof-of-principle experiment for the feasibility of NSE studies of the diffusion of molecular hydrogen on carbon aerogel and on Papyex exfoliated graphite (as reference). We used the 30 degree detector bank set-up IN11C at the highest flux wavelength 5.5 Å. We used the detector settings with the middle of the detector bank at 20 and 55 degrees, respectively, covering a Q-range of 0.2 to 1.4 Å⁻¹.

As a reference we measured a Papyex exfoliated graphite sample with a mass of 11.1 g without hydrogen loading at the cryostat base temperature of T = 2 K for 6 h for the 20 degree detector setting and 21 h for the 55 degree detector setting. We then adsorbed H₂ to a loading of about 0.2 monolayers (ML) relative coverage at 40 K. A polarized diffraction measurement delivered the data shown in Fig. 1. The diffraction measurement gave a small but sufficient signal of 15 cps from hydrogen across the detector bank.

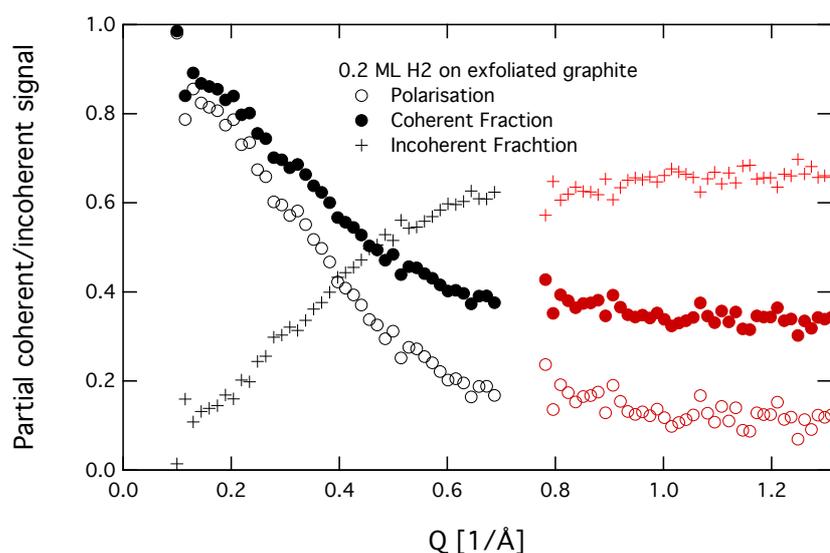


Figure 1: Polarisation measurement of 0.2 ML hydrogen adsorbed on exfoliated graphite sample. The data was obtained at 40 K.

In the following we measured NSE spectra on the 55 degree detector setting, i.e., $Q = 0.7\text{-}1.4 \text{ \AA}^{-1}$, at temperatures of 2 K, 10 K and 40 K for 24 h at each temperature. The spectra are shown in Fig. 2. From the data it is clear that dynamics can be traced at 40 K and there is a hint of dynamics at 10 K. The dynamics, however appear to be too fast for the IN11 spectral window.

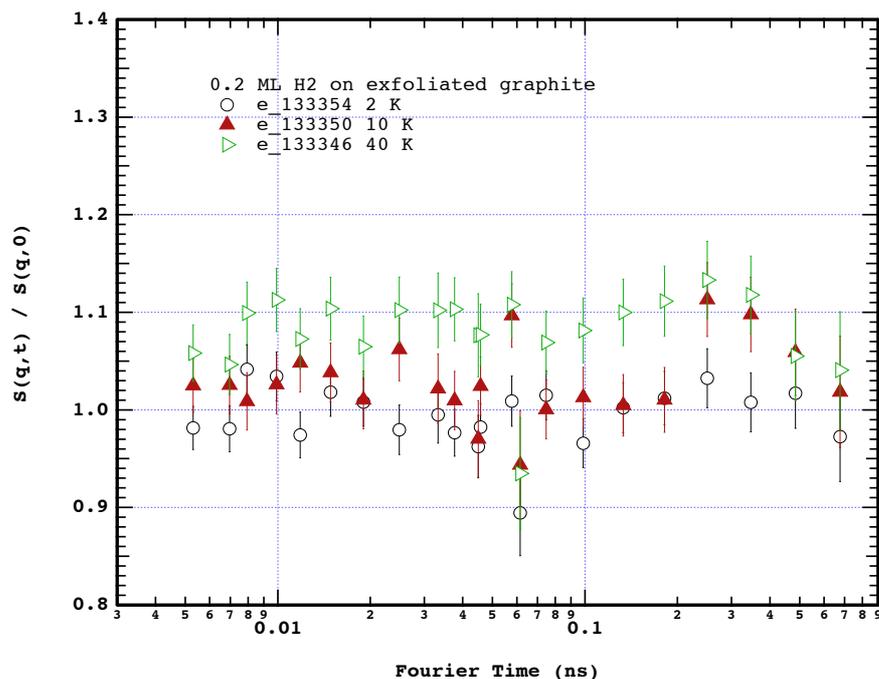


Figure 2: NSE spectra for 0.2 ML of hydrogen, H₂, adsorbed on exfoliated graphite.

After the exfoliated graphite measurements we tested a carbon aerogel sample with a loading of 0.5 ML hydrogen, H₂. The polarized diffraction measurement of this sample is displayed in Fig. 3, which makes it clear that the relative hydrogen signal is weaker than in the exfoliated graphite sample. This is very likely caused by the stronger spread of the coherent signal of carbon aerogel. However, we also experience a clearly visible change of the signal with time. The hydrogen was adsorbed at 40 K in an open system and we get a strong incoherent signal initially, which drops substantially with time (blue points from the first data taken immediately after adsorption).

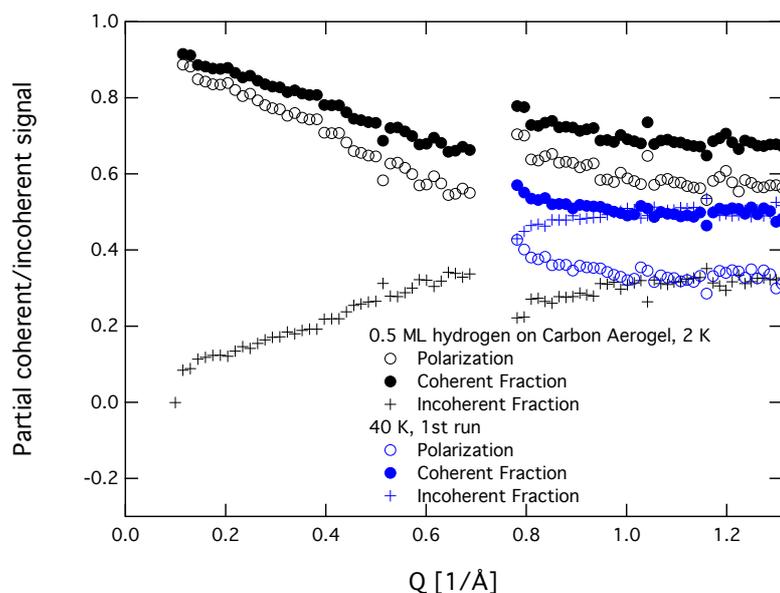


Figure 3: Polarized diffraction measurement of 0.5 ML hydrogen on Carbon Aerogel. The black symbols were measured at 2 K, whereas the blue symbols were obtained at the first spectrum of the freshly adsorbed hydrogen at 40 K.

NSE spectra of 0.5 ML hydrogen/carbon aerogel were measured for the 20 and 55 degree detector bank settings at 2 K and at 40 K. All spectra were measured for 4 h. The results are shown in Fig. 4. Clearly, the first 40 K spectrum measured at 55 degrees shows a beautiful trace of dynamics, whereas the second spectrum shows dynamics similar to the signal from exfoliated graphite (fast dynamics and weak signal). No dynamics are identified at the 20 degrees detector setting.

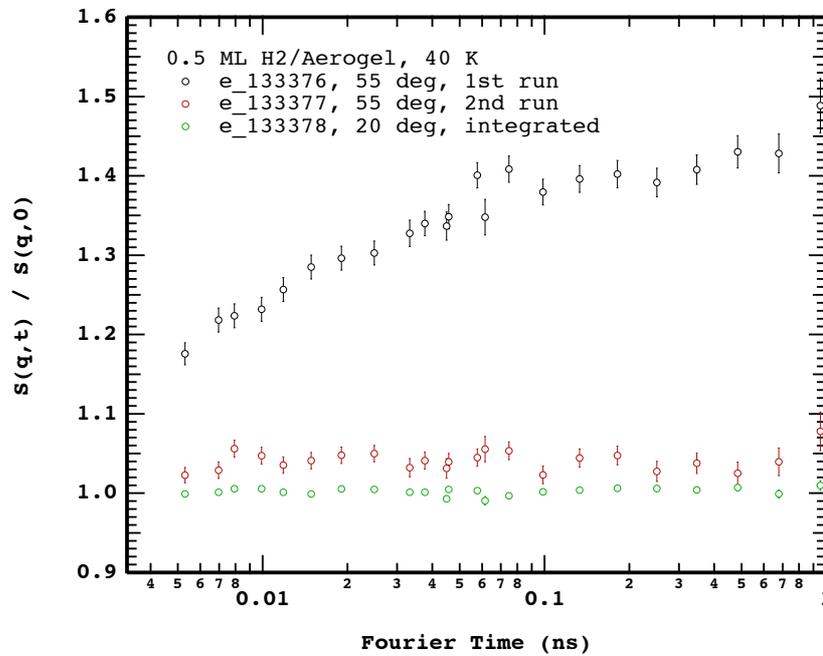


Figure 4: NSE spectra measured on 0.5 ML of hydrogen adsorbed on a carbon aerogel matrix.

The data from this experiment are insufficient for a separate publication, but they will be used for the planning of future experiments. Experiments using TOF spectrometers or WASP for higher signal can be envisaged.