Experimental report

18/05/2020

Proposal: 7-05-509 **Council:** 4/2019

Title: Diffusion of water and hydrogen inMoS2 (single crystal and powder)

Research area: Chemistry

This proposal is a continuation of 7-05-460

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Samples: MoS2 powder
MoS2 single crystal with H2O
MoS2 powder with D2O
MoS2 single crystal with atomic hydrogen

Instrument	Requested days	Allocated days	From	To
IN11	10	0		
IN6-SHARP	3	3	20/01/2020	23/01/2020

Abstract:

MoS2 is an intensely studied catalyst candidate for the hydrogen evolution reaction (HER) in water electrolyses. We have recently initiated a project to elucidate the structural and dynamic foundations for the reactivity of MoS2 by use of neutron scattering and complementary techniques. A first NSE experiment (7-05-460) revealed considerable reactivity and very fast diffusion within the layers of this 2D material, which could be underpinned by a TOF study on TOFTOF at MLZ, Garching.
A recent NSE study on electrolyses materials (MoS2 on carbon substrate), showed that both hydrogen and water dynamics can be detected in a similar dynamic window.
Therefore, we propose to perform a dedicated study to identify and separate the respective components of water and hydrogen dynamics in MoS2 single crystals and powders.

Experimental report exp.7-05-509 IN6

Molybdenum disulphide (MoS_2) is an intensely studied catalyst candidate for the hydrogen evolution reaction (HER) in water electrolyses. The deep understanding of hydrogen dynamics inside MoS_2 is crucial for improving its catalytic properties. Our last experiments have shown that in Time-of-Flight (TOF) experiments it is almost impossible to distinguish different hydrogen species, as they are usually detected in a similar dynamical window. Thus the main purpose of the current experiment on IN6/SHARP was to study separately the diffusion of H^+ ions and H_2O inside the MoS_2 single crystals.

Two sets of MoS_2 single crystals were prepared: one set was bombarded with a beam of atomic hydrogen and vacuum heated to 120 ºC before the experiment, while the other was bathed in purified H_2O prior to the experiment. The results show different dynamical behaviour for the two hydrogen species: at high momentum transfer, q, H_2O-MoS_2 exhibits an ordinary behaviour (Fig. 1): the higher the temperature the bigger the QENS broadening, with the latter being similar for 400 K and 500 K; while for H^+-MoS_2 the situation is different (Fig. 3): the QENS broadening at 500 K is much smaller than at 400 K, almost as small as at 200 K. We attribute this preliminarily to desorption of a hydrogen containing species.

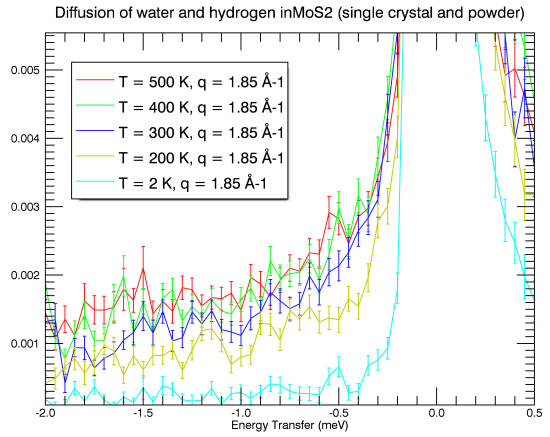

Fig. 1. Diffusion of water in MoS_2, TOF data, q = 1.85 Å^{-1}, colours according to the legend.

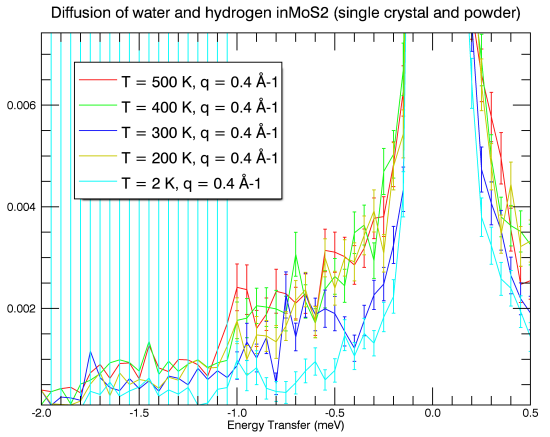

Fig. 2. Diffusion of water in MoS_2, TOF data, q = 0.4 Å^{-1}, colours according to the legend.

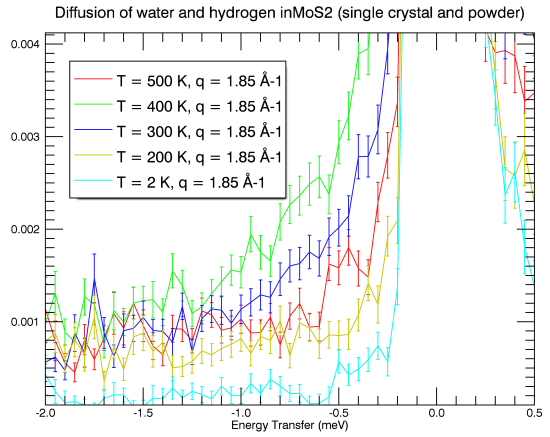

Fig. 3. Diffusion of atomic hydrogen in MoS_2, TOF data, q = 1.85 Å^{-1}, colours according to the legend.

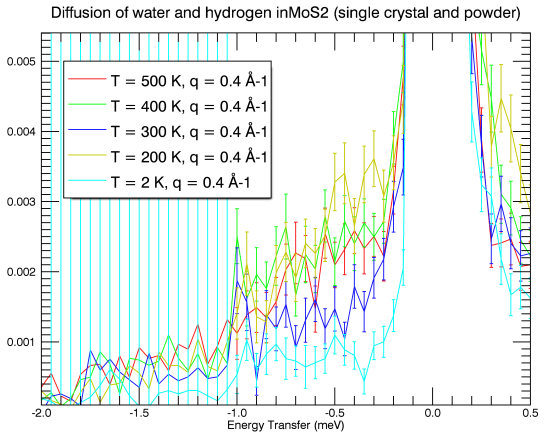

Fig. 4. Diffusion of atomic hydrogen in MoS_2, TOF data, q = 0.4 Å^{-1}, colours according to the legend.

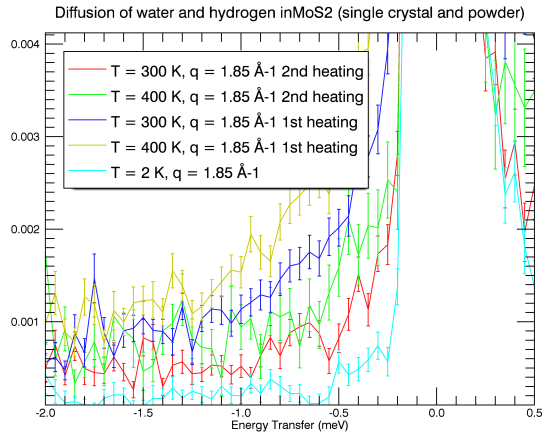

Fig. 5. Diffusion of atomic hydrogen in MoS_2, TOF data for the 1st and the 2nd heating cycle, q = 1.85 $Å^{-1}$, colours according to the legend.

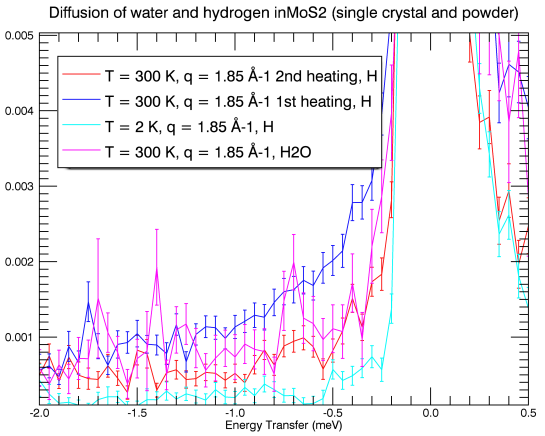

Fig. 6. Diffusion of atomic hydrogen and water in MoS_2, TOF data, T = 300 K, q = 1.85 $Å^{-1}$, blue line - H^+ 1st heating cycle, red line - H^+ 2nd heating cycle, pink line - H_2O, turquoise line - resolution function.

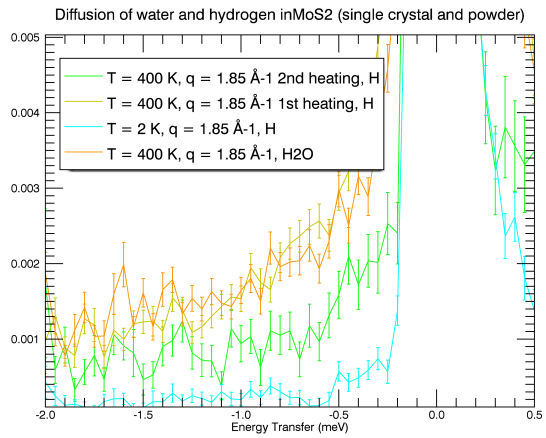

Fig. 7. Diffusion of atomic hydrogen and water in MoS_2, TOF data, T = 400 K, q = 1.85 $Å^{-1}$, yellow line - H^+ 1st heating cycle, green line - H^+ 2nd heating cycle, orange line - H_2O, turquoise line - resolution function.

At low q both H_2O-MoS_2 and H^+-MoS_2 exhibit an unexpectedly small broadening at 300 K, being lower than the broadening at 200 K (Figs. 2 and 4).

The experiment with atomic hydrogen included cooling down to 2 K, heating to 500 K and then again cooling down to 2 K and heating to 400 K. In Fig. 5. one can clearly see the effect of hydrogen desorption: both the 400 K and 300 K lines corresponding to the 1st heating cycle are above the corresponding data of the 2nd heating cycle, such behaviour still holds after the normalisation of the data.

Our preliminary data analysis shows that a) the difference of the diffusion of water and hydrogen could be observed and b) that desorption can be observed at temperatures at or beyond 400K. Further detailed analysis of the data will be required to obtain a complete picture.