Experimental report

13/02/2019

Proposal: TEST-2977 **Council:** 4/2018

Title: Diffusion of Hydrogen in Molybdenum Carbide Powder

Research area:

This proposal is a new proposal

Main proposer: Peter FOUQUET

Experimental team: Peter FOUQUET

Local contacts: Peter FOUQUET

Samples: 10 mg Hydrogen / 5 g Mo2C

Instrument	Requested days	Allocated days	From	To
IN11	8	8	16/10/2018	24/10/2018

Abstract:

Experimental Report Test-2977 „Dynamics of hydrogen Mo_2C“

1. Introduction

Recently, there has been immense progress in technologies related to the production of renewable energy, however storage concepts have not yet reached the same maturity. ‘Power-to-gas’ is one of the most promising concepts: Hydrogen gas is produced from water when energy is available and reconverted when energy is in demand. Each conversion process needs to be carried out as efficiently as possible using optimized materials.

In the frame of this technology the molybdenum compounds MoS_2 and Mo_2C are intensely studied catalyst candidates for the hydrogen evolution reaction (HER) in water electrolyzers [1,2]. Recent electrochemical studies on MoS_2 allow detailed conclusions about its catalytic activity [3-9], which is much more governed by dynamics than the original explanations of reactivity solely in terms of defect sites suggested. A drawback of MoS_2 for electrochemical application, however, is its poor electrical conductivity of only 2,17▪10-2 Ω-1 cm-1 [10], which is five orders of magnitude lower than the conductivity of graphite. From this point of view, Mo_2C and MoC are very promising due to their good electrochemical performance combined with good conductivity [11-14]. Similar to some oxidic catalysts they reveal a large flexibility in structures and stoichiometry and have favourably low work functions for their active surface (e.g., 3.4 eV for the (111) surface of Mo_2C) [15]. For both substances, the role of hydrogen intercalated below the surface and hydrogen moving along the surface is not fully understood.

2. Samples and Experiment

It was the aim of this test to study the dynamics of hydrogen in Mo_2C powder in a neutron spin-echo experiment on IN11 in its high signal set-up IN11C. The sample was loaded ex-situ with hydrogen gas by electrolyses in contrast to the main programme of proposal 7-05-501, which aims for in-situ sorption of hydrogen.

For reasons of maximising the signal for the short test time we used a wavelength of 5.5 Å with a *Q* range of 0.1 .. 0.7 $Å^{-1}$ and measured six representative temperatures: 2 K (resolution), 50 K, 100 K, 200 K, 300 K, and 500 K.

3. Results

As a first important result, we can confirm that we have found strong dynamic signal that shows a Q-dependence and temperature dependence. Fig. 1 shows spectra recorded in the Q range 0.1 .. 0.7 $Å^{-1}$ for the hydrogen loaded Mo_2C sample. At <100 K there is negligible diffusion, but at 200 K and above signal from diffusion is found. At 500 K we find that the hydrogen desorbs.

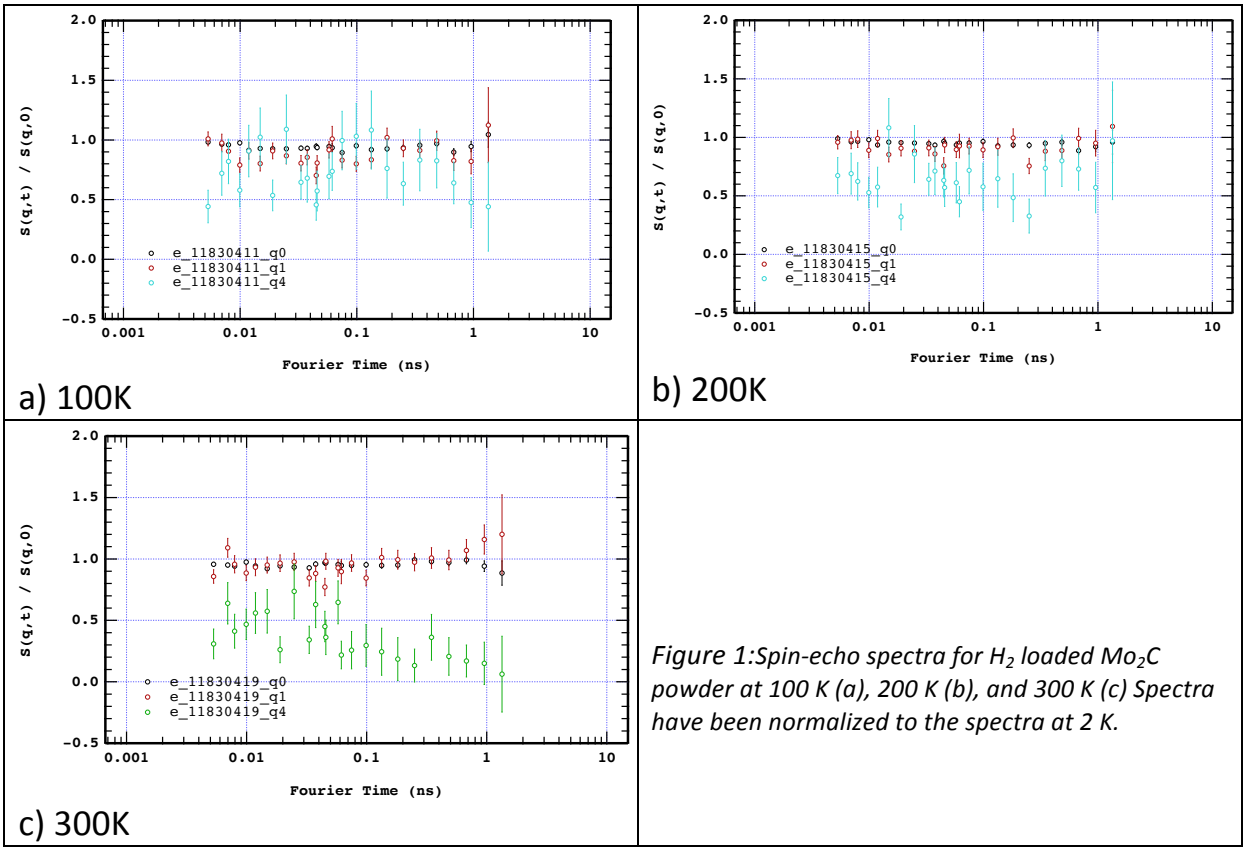

Figure 1:Spin-echo spectra for H_2 loaded Mo_2C powder at 100 K (a), 200 K (b), and 300 K (c) Spectra have been normalized to the spectra at 2 K.

References:

[1] B. Hinnemann et al. JACS 127(15),5308-5309, (2005).
[2] D. Merki, St. Fierro, H. Vrubel and X. Hu, Chemical Science 2, 1262-1267, (2011).
[3] Xiaohong Xia et al., International Journal of Hydrogen Energy 39, 9638–9650 (2014).
[4] Xiaoping Dai et al., International Journal of Hydrogen Energy 40, 8877–8888, (2015).
[5] Ya Yan et al., ACS Catal 4, 1693-1705, (2014).
[6] Jesse D. Benck et al., ACS Catal. 4, 3957-3971, (2014).
[7] Xiaoxin Zou and Yu Zhang, Chem. Soc. Rev.,44, 5148-5180, (2015).
[8] T. F. Jaramillo et al., Science 317, 100-102, (2007).
[9] J. D. Benck et al., ACS Catal. 2, 1916-1923, (2012).
[10] Ch. G. Wiegenstein and K. H. Schulz, Rev. Sci. Instr. 68, 1812 - 1813, (1997).
[11] Yagya N. Regmi et al., ChemCatChem 7, 3911-3915, (2015).
[12] Yagna N. Regmi et al., J. Mater. Chem. A, 3, 10085-10091, (2015).
[13] Chaoyun Tang et al., Journal of Power Sources, 296, 18-22, (2015).
[14] G.García et al., Journal of Electroanalytical Chemistry, 793, 235-241, (2017).
[15] José Roberto dos Santos Politi et al., PhysChemChemPhys 15, 12617-12625, (2013).
[16] H.-W. Becker et al., presented at Bunsen conference, Bochum Germany, 2015.
[17] E. Bahn, O. Czakkel, B. Nagy, K. Laszlo, S. Villar-Rodil, J.M.D. Tascon, F. Demmel, M.T.F. Telling, P. Fouquet, Carbon 98, 572-581 (2016).