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In-situ hydrogen sorption 2D-ACAR facility for the study of metal hydrides for hydrogen storage

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Abstract

We developed a dedicated hydrogen sorption setup coupled to a positron 2D-ACAR (two-dimensional Angular Correlation of Annihilation Radiation) setup employing a ^{22}Na-source, which will enable to collect 2D-ACAR momentum distributions *in-situ* as a function of temperature, hydrogen pressure and hydrogen content. In parallel, a dedicated glovebox was constructed for handling air-sensitive metal and metal hydride samples, with a special entrance for the 2D-ACAR sample insert. The 2D-ACAR setup was tested in first measurements on a $Pd_{0.75}Ag_{0.25}$ foil and on a ball-milled MgH_2 powder in both the hydrogen loaded and desorbed states. The hydrogen loaded $Pd_{0.75}Ag_{0.25}H_x$ sample was kept under a 1 bar hydrogen pressure to prevent partial desorption during measurements at room temperature. The collected 2D-ACAR distributions of $Pd_{0.75}Ag_{0.25}$ and $Pd_{0.75}Ag_{0.25}H_x$ showed similar features as observed in previous studies. The broadening of the ACAR distributions observed for the Mg to MgH_2 metal-insulator transition was compared in a quantitative manner to ab-initio calculations reported in the literature.

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Keywords: 2D-ACAR, air-sensitive samples, in-situ hydrogen sorption, vacancies, metal hydrides, metal-insulator transition, positron methods

1. Introduction

Metal hydrides are promising materials for the storage of hydrogen as a sustainable energy source for the development of fuel cell cars [1]. Magnesium hydride (MgH_2) is a promising cheap and abundant hydrogen storage material, with a high gravimetric storage capacity of up to 7.6 wt%. However, the uptake and discharge of hydrogen proceeds slowly and at low hydrogen equilibrium pressures for practical operation temperatures below ~100 °C. These properties can be improved by turning to nanoscale MgH_2 grain sizes [2]. Vacancy-related defects play an important role in the mobility of hydrogen in metal hydrides, which is a crucial factor to achieve sufficiently fast hydrogen sorption kinetics. Recent positron Doppler broadening and positron lifetime studies monitored the

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evolution of vacancies and small vacancy clusters in MgH_2 [3-6], $Pd_{1-y}Ag_yH_x$ [7] and $NaAlH_4$ [8] metal hydrides in measurements as a function of hydrogen cycling. The metal-to-metal-hydride transition is often accompanied by large changes in the electronic structure, in particular when the metal hydride is an insulator, such as MgH_2 [9]. These large changes in the electronic structure have a pronounced effect on their optical properties, leading to promising applications of metal hydrides in fast optical-switching hydrogen sensors [10]. As discussed in [9], the electron momentum distribution of MgH_2 is correspondingly expected to broaden significantly relative to that of the Mg metal phase, as should be observable in 2D-ACAR distributions.

In order to enable *in-situ* collection of 2D-ACAR distributions as a function of temperature, hydrogen content and pressure, we developed a dedicated hydrogen sorption setup coupled to the Delft positron 2D-ACAR setup employing a ^{22}Na-source. The airtight sample holder insert further enables 2D-ACAR studies on air-sensitive samples in general. To this aim, we further employ a new dedicated glovebox with a special entrance for the 2D-ACAR sample insert. Here, we describe the instrumental details of the *in-situ* hydrogen sorption 2D-ACAR facility and first results obtained on a $Pd_{0.75}Ag_{0.25}$ foil and a ball-milled MgH_2 powder in the metal and metal hydride phases, demonstrating its potential for the study of metal hydrides for hydrogen storage and hydrogen sensors.

2. In-situ 2D-ACAR setup and dedicated glovebox

Figure 1a shows the central part of the 2D-ACAR setup at the Reactor Institute Delft (RID) destined for measurements of bulk samples with thicknesses in the (sub-)mm range [11]. It consists of an electromagnet, a lead container with the positron source and a vacuum chamber, all mounted on a supporting table. The 2.7×10^8 Bq (strength in June 2011) ^{22}Na positron source is mounted on a rod which can move into the bore of one of the poles of a water-cooled electromagnet. The uniform magnetic field generated by the electromagnet is used to guide the positrons with a maximum kinetic energy of 540 keV towards the sample. The spot size of the positron beam on the sample depends on the strength of the magnetic field, which can be changed with the current through the electromagnet and the gap between the poles. In the present setup the distance between the poles of the iron yoke is 60 mm (Figure 1b). In this configuration a current of 16.5 A through the electromagnet generates a magnetic field of 0.71 T. This yields a spot size on the sample of ~8 mm.

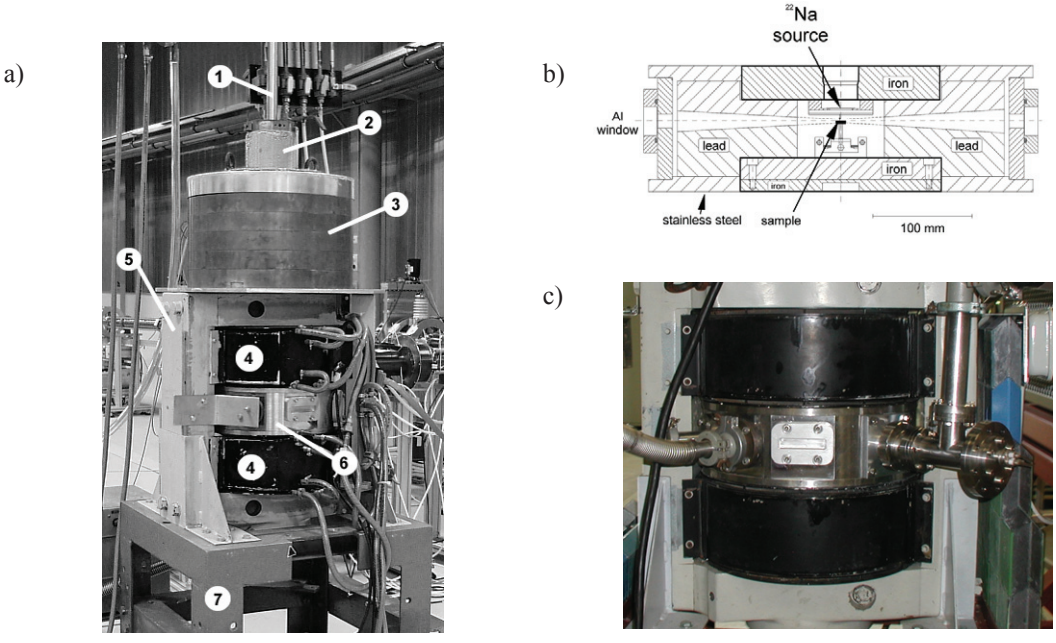

Figure 1 a) Overview of the ^{22}Na-source based 2D-ACAR vacuum chamber. 1 – rod holding the ^{22}Na positron source, 2 – source lead container, 3 – additional lead shielding, 4 – electromagnet coils, 5 – iron yoke, 6 – vacuum chamber, 7 – supporting table; b) Cross-sectional view of the ^{22}Na-source based 2D-ACAR vacuum chamber (without inserted load-lock sample holder); c) ^{22}Na-source based 2D-ACAR vacuum chamber with inserted load-lock sample holder (right) for *in-situ* hydrogen sorption studies.

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