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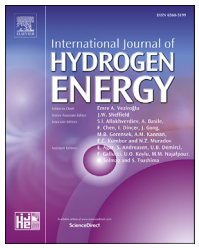

Review Article

Pitfalls in the characterisation of the hydrogen sorption properties of materials

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ABSTRACT

Characterising the hydrogen sorption properties of materials is important for a range of applications, including solid state hydrogen storage, electrochemical and thermal energy storage using metal hydrides, and H_2 gas compression and purification. However, it can be technically demanding and subject to significant error if not performed with care. In this article, potential pitfalls in the performance of hydrogen sorption measurements are discussed. The topics covered include instrument design and calibration, sample size choice, sample and gas purity, isotherm measurement procedure and issues associated with data reduction. Approaches to validating equipment and isotherm measurements are also discussed. Different sample types are considered, including metal and complex hydrides and nanoporous adsorbents, such as porous carbons, zeolites and metal-organic frameworks (MOFs).

Contents

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Introduction

Accurately determining the hydrogen sorption properties of materials is critical to assessing their use in a range of applications, but it is also required for fundamental studies. Hydrogen absorption by elemental metals, alloys and intermetallics [1–7] has been of interest ever since the reversible absorption of hydrogen by Pd was first discovered, over 150 years ago [8]. However, more recently, the uptake and release of hydrogen by a range of other materials has been studied, including complex hydrides [9,10], such as sodium alanate [11], borohydrides [12–14] and the Li-N-H system [15,16], nonporous polymers [17–19], and nanoporous materials that include porous carbons [20–22], zeolites [23], metal-organic frameworks [24–26] and microporous organic polymers [27–29].

Much of the recent work has been driven by the intense interest in mobile hydrogen storage [30–35]. As a result, most of the above materials have been considered for this purpose [9,10,33,36–39]. However, metal hydrides have many other applications, including electrochemical [40,41] and thermal energy storage [42,43], in actuators [44] and heat switches [45,46], compressors [47,48], heat pumps and cryocoolers [49], as getters [50–53], and for isotope (H_2/D_2) separation and H_2 purification [36,54–56]. To date, the most commercially successful of these is the use of $LaNi_5$-based materials in Ni-MH (Nickel-Metal Hydride) battery electrodes [57–59], but research into new materials for this application is being actively pursued [41,60]. Meanwhile, traditional nanoporous materials, such as activated carbons and zeolites, are commonly used to separate and purify H_2 using pressure swing adsorption (PSA) [61]. Polymeric membranes are also widely used in separations involving H_2, including ammonia purge gas recovery, refinery gas purification and syngas ratio adjustment [17,62]. These processes rely on H_2 separation from N_2, CH_4 and CO, respectively [62]. Such membranes are also among the many types being investigated for H_2 purification for fuel cell applications [56,63], while isotope (H_2/D_2) separation is an emerging H_2 sorption-based application for nanoporous materials [64–68].

Different applications require different hydrogen sorption properties [3,36,54,55]. For example, for H_2 storage, a significant amount of reversible uptake must occur at above ambient pressures due to delivery pressure demands. In contrast, battery electrode materials must reversibly absorb hydrogen at sub-ambient pressures because of the operating conditions of electrochemical cells [3,57]. When metal hydrides are used to compress H_2 gas to pressures of tens of MPa, materials with reversible capacities at these higher pressures are required [47]. At the other end of the scale, for getters, absorption must occur at very low, sub-mbar, pressures

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