



Short communication

Hybrid system for potential room temperature hydrogen storage

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ABSTRACT

In this study, the hydrogen storage of porous silicon-based systems is considered. In this regard, the electrochemical anodization process was employed to introduce porous structure over silicon wafer. Moreover, the Pd nanoparticles as great reservoirs of hydrogen species were deposited over porous silicon; using electroless technique. X-ray diffraction, Fourier transfer infrared, and Field emission scanning electron microscopic measurements were employed to study the structure and morphology of fabricated samples. Crystal faces and various Si–H as well as Si–O bonds were defined through X-ray diffraction and Fourier transfer infrared characterizations. Also, palladium clusters were recognized through Field emission scanning electron microscopic measurements. Hydrogen storage measurements revealed that porous silicon might contribute in hydrogen storage, slightly; while Pd/porous silicon demonstrated a great potential, in this regard. Moreover, variation of electroless quality proved that as deposition time increases, Pd clustering over porous substrate enhances; consequently, improve the storage capacity.

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Today's, wide environmental impacts such as global warming, climate change, etc. have simulated the policies to limit the supply of fossil fuels. In this regard, use of renewable energy resources becomes more important as it appears. Amongst, hydrogen as a clean, renewable, and high dense energy carrier plays a great role in sustainable development. To apply this potential, scientists have reported various hydrogen production systems [1–4]. With this approach the hydrogen production is a mature technology; however, the main issue has been defined in the sector of storage. Several structures, include porous media [5,6], have been reported as storage systems. According to the literature, silicon as an abundant element in the earth's crust has played a great role in this regard. Scientists proved the low affinity of a pristine silicon surface to hydrogen through examining the adsorption energy between those arrays [7]. The roughness and porosity on the surface of silicon substrate improve the absorption of hydrogen, inherently. Amongst, porous silicon has received attentions and has been examined as hydrogen storage media in theory [8]. Moreover, Lysenko et al. [9] experimented monocrystalline silicon, solely, as hydrogen storage system. They used attenuated total reflection

infrared method for storage measurements and achieved the maximum value of 66 mmol/g. Also, practically, scientists have studied several systems with incorporation of porous silicon as a substrate; e.g., He et al. [10] fabricated a hybrid system based on carbon nanotube (SWCNT) and porous silicon. They reported the maximum value of 7.93×10^{-2} mmol/g. Clearly, porous media can offer efficient storage system through void volume [11] provided by various interconnected macro, micro, and nanopores.

Also, it has been proved that metallic nanoparticles contribute in the hydrogen storage [12]. Transitional metals, especially V catalysts, improve the hydrogen storage capacity (HSC) of structures through atomization of hydrogen molecules via spillover mechanism [13]. Also, metal nanoparticles such as Pd could be a potential reservoir for hydrogen, through intrinsic void volume [12].

As far as we know, the hybrid system of Porous polysilicon/Pd has not been considered for hydrogen storage. In this study, we examined the HSC for hybrid composition of Porous Si/Pd. The HSC were measured with Sievert setup. The structural and morphological investigations were characterized by X-ray diffraction (XRD), Fourier transfer infrared (FTIR), and Field emission scanning electron microscopy (FESEM).

In the experiments, P-type polysilicon wafers with thickness of about 320 μm and with resistivity in the range of 0.5–1.5 Ωcm, were used as substrate. The electrical contact for electrochemical etching

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was achieved by evaporation of 250 nm Al film.

Electrochemical anodization (EA) as its name suggests, propose an electrochemical cell that prepares chemical media to pursue the etching process. The electrolyte was used to electrochemical anodization consisted of ethanolic based solution of hydrofluoric acid (40%) and deionized water (DI). We found the optimized conditions for anodization process assigned in previous experience. In this regard, we prepared electrolyte in the given volume ratio of (40% HF: EtOH; 1: 1) and pursue EA process under condition of 10 mAcm^{-2} for 10 min. The electrochemical process established continuously; employing direct current.

Immediately after EA, palladium nanoparticles were deposited as catalyst over the porous polysilicon surface; by electroless technique. The process was progressed using diluted aqueous PdCl_2 in the presence of HCl (trace) at Room temperature (RT). The concentrations of PdCl_2 and HCl in the solution were $1.13 \times 10^{-3} \text{ M}$ and $1.21 \times 10^{-3} \text{ M}$, respectively.

Two samples were fabricated using above procedure with the different in metal deposition condition; the electroless duration of 3 and 5 min were examined (which are defined as E3 and E5, respectively). Lastly, the samples were rinsed with DI.

The XRD (Philips X'pert operating with $\text{CuK}\alpha$ radiation ($\lambda = 1.54178 \text{ \AA}$) at 40 kV/40 mA), FTIR (PerkinElmer Spectrum BXII), and FESEM (TESCAN-MIRA3) measurements were used to study the samples characterizations.

Hydrogen storage measurements of the hybrid samples were carried out at ambient condition, temperature of 298 K and atmospheric pressure, using the Sievert volumetric setup. The setup consisted of hydrogen buffer-tank, jointed with the reaction chamber and a sensitive-calibrated pressure transducer (0.05% error at full scale). The leakage measurement (using helium gas) was found to be negligible for the examined system. Prior to the storage evaluation, the samples were thermally activated at $300 \text{ }^\circ\text{C}$ for 2 h under evacuation up to 10^{-4} Torr to ensure that the composites were degassed and desorbed the moisture upon exposure to ambient conditions. When the samples were cooled down to RT, high-purity hydrogen gas (99.999%) was inlet to the measuring chamber up to a pressure of 6.89 MPa and maintained until a thermal equilibrium was established. As equilibrium was achieved, hydrogen was exposure to the hybrid systems, isothermally.

Fig. 1 shows the typically XRD pattern of the hybrid structures. The peaks are defined as strong Si (1 0 0), face centered cubic (fcc) Pd (2 0 0), Pd (1 1 1) and Pd_3Si (1 2 1) reflection which is in good agreement with reference data (JCPDS numbers 271402, 050681, 011310 and 360932, respectively). The presence of both crystal

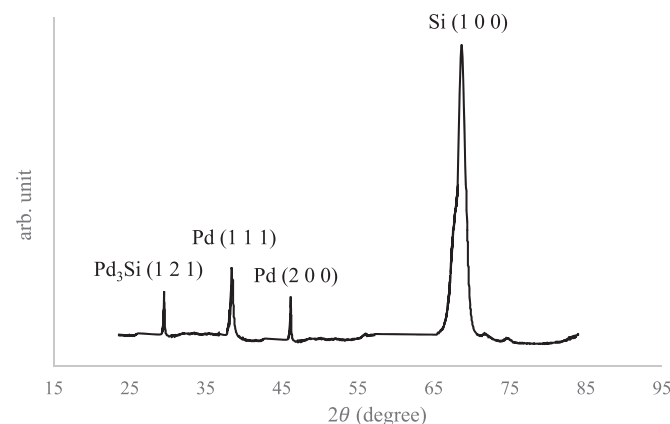


Fig. 1. Typically XRD pattern for Pd deposited on porous silicon by electroless technique.

faces of (2 0 0) and (1 1 1) translate the polycrystalline nature of Pd lattice; in turn, this demonstrates the existence of grain boundaries and vacancies in these structures. Naturally, a barrier layer of silicon oxide prevents the merge of silicon and palladium. According to simultaneous palladium deposition over porous silicon during electroless process and silicon oxidation [14], the appearance of palladium silicide peak may come from the insufficient thickness of oxide layer [15] and/or the existence of discontinuous oxide layer inserted in the interface of silicon and palladium.

Fig. 2 demonstrates the FTIR spectrum in the wavenumber range of $350\text{--}1350 \text{ cm}^{-1}$. For Porous silicon/Pd samples, the absorption peaks correspond to the $1070\text{--}1086 \text{ cm}^{-1}$, $798\text{--}800 \text{ cm}^{-1}$, and $455\text{--}470 \text{ cm}^{-1}$ are stretching, bending, and rocking vibration of SiO_2 . Also, the bending vibration band at the wavenumber of $879\text{--}882 \text{ cm}^{-1}$ is defined for Porous silicon/Pd samples which belongs to the O_3SiH surface groups. For Porous silicon, the intense bands of 906 , 660 , and 625 cm^{-1} correspond to SiH_x groups. As it was known, pure porous silicon surface is covered with the hydrogen atoms; thus, the SiH_x species are distributed on the surface. Stretching intense bands of SiH_3 , SiH_2 , and SiH at 2137 , 2114 , and 2088 cm^{-1} have been demonstrated at the Fig. 2-inset. However, this approach is different when transitional metals such as Pd, are present at the surface. Therefore, the FTIR measurement detects the peaks belong to SiH_x groups less intense while the silicon oxide are recognized strongly in the case of Porous silicon/Pd; and vice versa for pure porous silicon. Indeed, the decreasing peaks intensities of SiH_x for Porous silicon/Pd could be agree with the consumption of SiH_x species during the reduction of Pd^{2+} : $2\text{Si} - \text{H} + \text{Pd}^{2+} \rightarrow \text{Si} - \text{Si} + \text{Pd} + \text{H}_2$. Also, increase the intensities peaks of SiO refer to reduction of Pd in the presence of H_2O : $\text{Si} - \text{Si} + \text{Pd}^{2+} + \text{H}_2\text{O} \rightarrow \text{Si} - \text{O} - \text{Si} + \text{Pd} + 2\text{H}^+$.

The surface morphology of samples was characterized with the help of FESEM, as shown in the Fig. 3.

As it is illustrated in the Fig. 3, the increase in electroless duration may enhance the growth of Pd nanoparticles. Clearly, the growth of Pd nanoparticles in the E5 is several times higher than that of E3. Also, the Energy disperse spectroscopy (EDS) characterizations confirm this claim (Fig. 4). Clearly, the presence of oxygen in the EDS characterizations demonstrates that formation of oxygen film on the surface of proposed systems should be considered. It has been proved that the oxide barrier effects and occlusion of hydrogen species within the surface oxide film [16]. This phenomenon may takes place through hydrogen bonding established as O–H functions.

However, the deposition and growth process, for both samples, was in presence of ultra violet. Generally, it seems that increase in

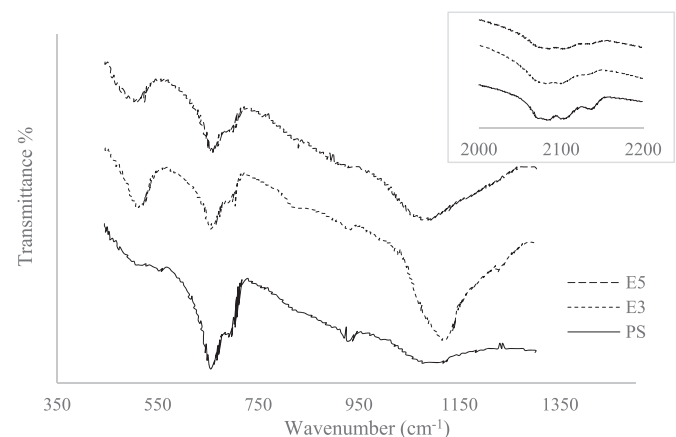


Fig. 2. FTIR spectrum for E5, E3, and porous silicon (PS).

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