A Single Chip Standalone Water Splitting Photoelectrochemical Cell

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Abstract: Water splitting photoelectrochemical cell (PEC) was fabricated in which the electrolyzer parts were made on a single chip. This was achieved by depositing an optically transparent Mn-oxide-TiO_2 thin layer on the front of a triple junction amorphous Si photovoltaic cell which acted as the anode and the back stainless steel layer acted as the cathode under illumination of light. This single chip water electrolysis cell operates like an artificial leaf. Water splitting was observed by simply submersing the device in a basic electrolyte solution under solar simulated light of 1 sun (0.1 W cm^{-2}). This self-driven PEC was found to produce hydrogen gas at the rate of 12.42 L m^{-2} h^{-1} and a solar to hydrogen efficiency (STHE) of 3.25 % from the collected H_2 gas in 2.5 M KOH solution. No signs of degradation of this single chip PEC were observed during water splitting when the device was run continuously for 6 hours.

Keywords: Water-splitting, Triple-Junctions amorphous-Silicon, photovoltaic.

1. INTRODUCTION

Photoelectrochemical splitting of water converting solar energy to hydrogen fuel offers an alternative energy solution, which is clean and renewable [1-12]. The cost of converting solar energy to hydrogen can be minimized by the use a self-driven monolithic photoelectrochemical cell (PEC) that would eliminate the costs associated with separate construction and interconnection of solar cells and the water electrolyzer [13]. The concept is to develop a leaf-like water splitting PEC as shown in Figure **1** that would split water into hydrogen and oxygen under sunlight illumination. Such process is promising with the use of semiconductors that generate enough power under sunlight illumination to drive the thermodynamically uphill water splitting reaction. Among the existing solar photovoltaic, silicon based solar cells can satisfy the criteria of such water splitting PECs.

Since the first publication on amorphous silicon (a-Si) in the late 1960s, a-Si has been considered as a promising material compared to the crystalline silicon based solar cells [14, 15]. Furthermore, a-Si has a better photoresponse in the more energetic visible region of the solar spectrum compared to crystalline silicon which is mostly sensitive in the less energetic longer wavelengths (IR) region [14, 15]. Amorphous silicon (a-Si) has been investigated as an alternative to polycrystalline silicon for the use in solar cells. However, stability issues of a-Si in aqueous electrolytes and its high valence band position with respect to the oxygen oxidation potential [12] make it unfavorable for direct water splitting under sunlight illumination. These limitations can be surmounted by covering a-Si based photovoltaic with visible light transparent stable thin films. While a-Si based solar cell provides the necessary photovoltage to drive the water splitting reaction under sunlight illumination, the appropriate deposited thin film layer can protect the underlying a-Si and as well as provides the favorable valence band position for water splitting. High band gap oxide based semiconductors are visible light transparent and stable in basic electrolytes. Such oxides can be used as a transparent conductive corrosion resistant (TCCR) layer to protect a-Si based photoelectrode in electrolyte solutions.

Recently, coating with thin films has become accessible with the use of dip coating, spin coating, chemical vapor deposition and electro-deposition techniques [16-19]. The cost effective thin films to be used as TCCR protective layer on a-Si are the transition metal oxides. For example, titanium oxide (TiO_2) is stable in alkaline medium and absorbs only in the UV region of the solar spectrum. Also, a thin film coating of Mn_2O_3 was found to stabilize crystalline n-Si, n-TiO_2 and n-GaAs thin films in earlier studies [18, 19]. Some important studies pioneered in utilizing PECs such as crystalline Si (pin-pin) [20], p/n-GaAs [19], p/n-Si [21-23], n/p-GaAs/p-$GaInP_2$ [24], p/n-Fe_2O_3 [25], and triple junction amorphous silicon [26, 27] for monolithic photoelectrochemical water splitting without the need of additional power from an external source.

In this work we report a single chip water splitting photoelectrochemical cell (PEC) in which a triple junction amorphous silicon (Tj-a-Si) cell deposited on stainless steel substrate was covered with a thin layer

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of Mn-oxide-TiO_2 and submersed in 2.5 M KOH electrolyte solution. Under solar simulated light of 1 sun the device splits water without the use of any external power source where oxygen evolution occurred on the front Mn-oxide–TiO_2 coated Tj-a-Si surface (photoanode) and hydrogen evolved at the stainless steel (cathode) as seen in Figure **1**.

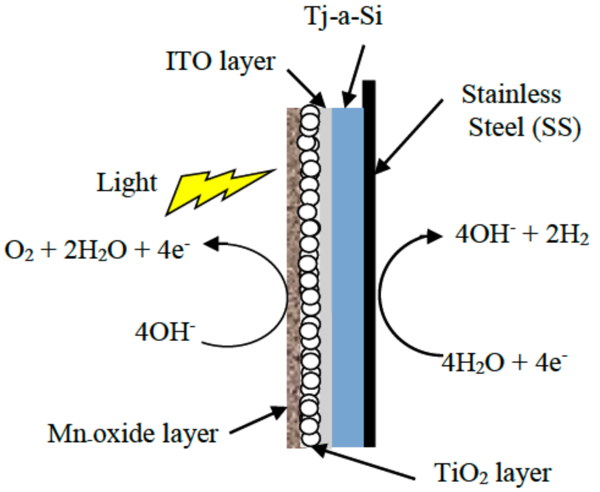

Figure 1: A single chip standalone water splitting photoelectrochemical cell (PEC).

2. EXPERIMENTAL PROCEDURE

2.1. Deposition of a Transparent TiO_2 Thin Layer on Tj-a Si Surface

An advanced method was used to deposit a uniform and durable thin film of TiO_2 on Tj-a-Si surface. In this method an ultrafine colloidal solution of TiO_2 (Degussa p 25) instead of the suspension was utilized. The colloidal solution consisted of 80 mg of TiO_2 powder in 10 mL ethanol, in presence of 10 μL of a structure directing agent such as acetylacetone [28]. A binder Triton X100 (5 micro liters in 10 mL ethanol) was added to the suspension to generate uniform films. The mixture was first homogenized in a high-speed centrifuge (Avanti J-20XP) at 3,000 rpm for 15 min producing a clear colloidal solution at the top and a thick white precipitate at the bottom. A single drop from the top of the ultrafine colloidal solution of TiO_2 was spread on the surface of Tj-a-Si with an area of 0.80 cm^2; spin coated for 1 min, the procedure was repeated 10 times and then dried at room temperature. The resulting films were sintered at 200°C for 1 hour to obtain a uniform solvent free TiO_2 thin film on Tj-a-Si surface.

2.2. Deposition of Manganese Oxide Thin Layer on TiO_2 Covered Tj-a Si Cell

The bath for the deposition of manganese oxide thin layer consisted of aqueous solutions of 0.03 M $MnCl_2$ and 1.4 M NH_4OH, having a total volume of 50 mL. A 0.25 M NH_4Cl solution was added to this mixture to lower the pH so that slow precipitation of $Mn(OH)_2$ occurs. This solution was stirred continuously at room temperature. The TiO_2 thin film covered Tj-a-Si sample was immersed in the deposition bath for 15 minutes to allow deposition of $Mn(OH)_2$ on its surface. The sample was then rinsed with double deionized water and dried at room temperature. The dried $Mn(OH)_2$ covered TiO_2-Tj-a-Si sample was transferred to a glass funnel followed by evacuation to $2x10^{-5}$ torr. The sample in the evacuated glass funnel was heated at 220 °C for 15 min. Curing $Mn(OH)_2$ under vacuum results in the loss

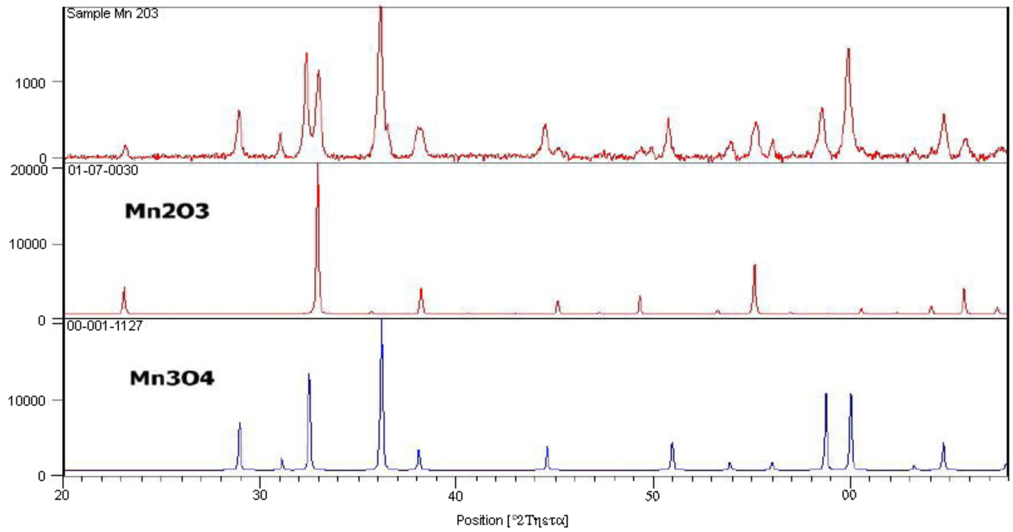

Figure 2: XRD powder patterns for Mn-oxide deposited by drying the $Mn(OH)_2$ in vacuum at 220 °C (top plot). Patterns for Mn_2O_3 and Mn_3O_4 are observed, see jcpds card 01-071-0636 and jcpds card 00-001-1127, respectively.

of water and hydrogen, thereby leaving a thin film of Mn_2O_3. The deposition process of Mn_2O_3 can be described by chemical reactions [18, 19],

$$MnCl_2\,(aq) + 2\,NH_4OH\,(aq) \rightarrow Mn(OH)_2\,(s) + 2NH_4Cl\,(aq) \quad (1)$$

During heating at 220°C under vacuum of $2\mathrm{x}10^{-5}$ torr:

$$2\,Mn(OH)_2\,(s) \rightarrow Mn_2O_3\,(s) + H_2\,(g) + H_2O\,(g) \quad (2)$$

Formation of Mn_2O_3 was confirmed earlier by X-ray photoelectron spectroscopy (XPS) [19]. However, the X-ray diffraction (XRD) patterns of the deposited and vacuum cured Mn-oxide thin film in this study shows the presence of Mn_2O_3 and Mn_3O_4 crystalline phases as seen in Figure **2**. This is because drying $Mn(OH)_2$ at 220 °C in vacuum can also form Mn_3O_4 according the reaction,

$$3Mn(OH)_2\,(s) \rightarrow Mn_3O_4\,(s) + H_2\,(g) + 2H_2O\,(g) \quad (3)$$

2.3. Hydrogen Gas Collection at Pt Cathode in Mn-oxide–TiO_2 Coated Tj-a-Si /Pt PEC

Schematic diagram of this single chip Mn-oxide–TiO_2 coated Tj-a-Si/SS PEC as shown in Figure **1** was modified by connecting a platinum wire (cathode) to the back stainless steel (SS) surface to facilitate H_2 gas collection. Oxygen evolution occurred on the front Mn-oxide–TiO_2 coated Tj-a-Si surface (photoanode). Hydrogen gas was collected by displacing the electrolyte solution inside a graduated test tube inverted over the platinum wire (cathode). The collected H_2 gas was identified by performing gas chromatographic (GC) analysis using an SRI Model 30 TCD gas chromatograph. The measured volume ratio of H_2 and O_2 collected during water splitting was found to be 2:1 as expected. The Mn-oxide–TiO_2 coated Tj-a-Si- solar cell was placed at a distance of 7 inches from the solar simulator (Oriel, Model 51192) with global AM 1.5 filters having light intensity of 0.1 W cm^{-2} (1 sun). The intensity of light was measured by a silicon detector, model 10DP/SB (UDT Sensors Inc.). The bias needed for water splitting reaction was provided by the underlying Tj-a-Si solar cell under the same illumination conditions.

3. RESULTS AND DISCUSSION

3.1. Volumes of H_2 Gas Collected During Water Splitting

The volume of hydrogen collected using Mn-oxide–TiO_2 covered Tj-a-Si electrode (surface area 0.80 cm^2) as a function of time of exposure under solar simulated light is given in Figure **3**. From the slope (0.0207 mL H_2 cm^{-2} min^{-1}) of the fitted straight line of the plot (Figure **3** inset), the rate of the volume of H_2 gas evolution per unit area of cathode surface was obtained. The steady state H_2 generation rate was found to be 12.4 L m^{-2} h^{-1}. The moles of H_2 produced per unit area of electrode surface per second, n_{H2}= 1.37 x 10^{-8} mol cm^{-2} sec^{-1}, were calculated using the ideal gas law as,

$$n_{H2} = [(P_{atm} - P_{water\ vap}) \times v_{H2} / RT] \quad (4)$$

where v_{H2} is the volume of H_2 gas collected by displacement of electrolyte solution per unit area of electrode surface per unit time, assumed hydrogen to behave as an ideal gas at atmospheric pressure, and the vapor pressure of water in the electrolyte solution to be equal to that of pure water, $P_{water\ vap}$ = 0.03126 atm (23.76 mm Hg), at room temperature. Also, no signs of degradation of Mn-oxide–TiO_2 coated Tj-a-Si were observed when this water splitting PEC was run continuously for 6 hours in 2.5 M KOH (See Figure **3**).

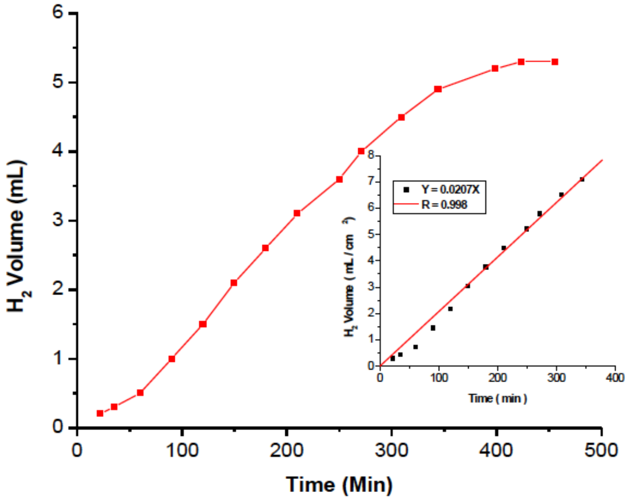

Figure 3: Volume of Hydrogen gas (mL) collected versus time (min). In the inset a fitting line was drawn for the linear part of the plot of volume of H_2 gas per photo-electrode area (mL cm^{-2}) versus time.

3.2. Solar to Hydrogen Efficiency, STHE (%)

The percentage of solar to hydrogen efficiency, STHE (%), was calculated from the photocurrent density, j_p obtained from the number of moles of hydrogen, n_{H2}, collected using Faraday's equation and was found to be 2.64 mA cm^{-2}. The percentage of solar to hydrogen efficiency STHE (%) for H_2 generation was obtained from the following expression [24-27]:

$$\mathrm{STHE}\ (\%) = [(j_p \times E^{o}_{rev}) / P_{light}] \times 100 \quad (5)$$

Using the standard reversible potential, E°_{rev} = 1.23 V for the overall water splitting reaction and the light

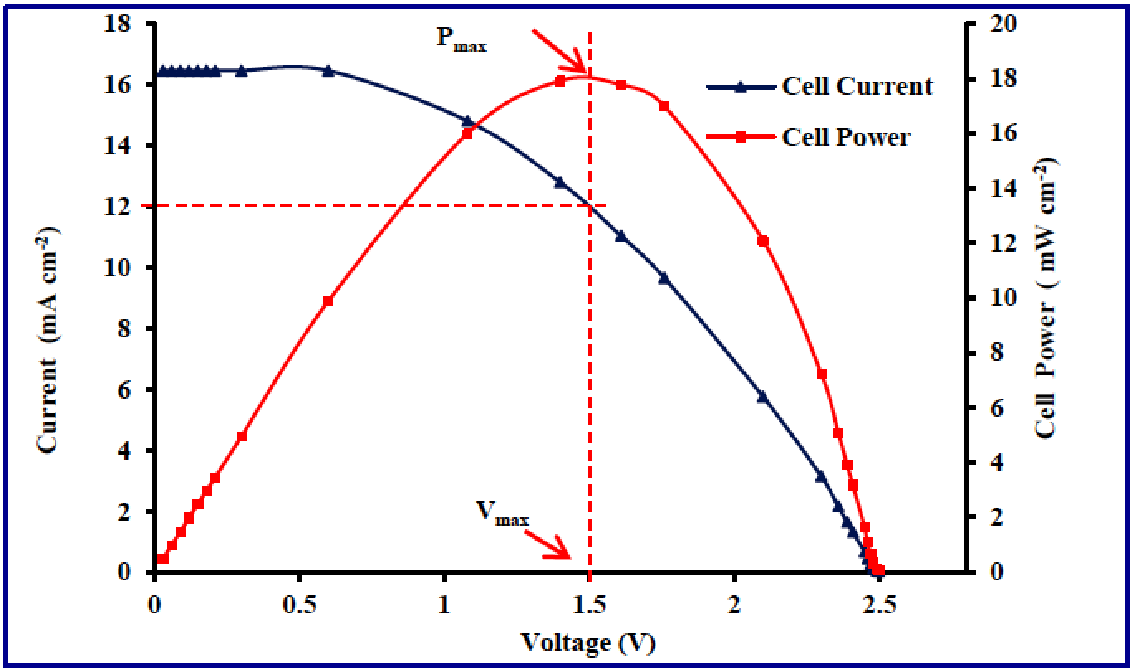

Figure 4: Plots of photocurrent density, J_p, and cell power density versus photovoltage for the unmodified Tj-a-Si solar cell with a surface area of 0.80 cm^2 to determine the maximum photovoltage (V_{max}) at a maximum power under solar simulated light of 1 sun (0.1 W cm^{-2}).

intensity, P_{light} = 0.1W cm^{-2} in equation (5) the STHE (%) of 3.25 % was obtained. This is in agreement with STHE (%) calculated directly from the number of moles of H_2 collected, n_{H2} using the equation [29],

$$\text{STHE (\%)} = [(-\Delta G^\circ \times n_{H2}) / P_{light}] \times 100 \qquad (6)$$

with ΔG° = - 237.2 kJ $mole^{-1}$ for the free energy of formation of H_2O (*l*) from H_2(*g*) and O_2 (g) under standard state condition, n_{H2} = 1.37 x 10^{-8} mol_{H2} cm^{-2} sec^{-1} and P_{light} = 0.1W cm^{-2}.

This observed low solar to hydrogen efficiency of 3.25 % was attributed to low maximum photovoltage of 1.5 volt supplied by the Tj-a-Si solar cell for water splitting as shown in its I-V plot in Figure **4**. The Mn-oxide–TiO_2 coating also decreased the photovoltage and photocurrent by preventing some of the incident light from reaching the underlying Tj-a-Si solar cell.

It should be noted that the Tj-a-Si solar cell that we used in this study could generate maximum photovoltage of 1.5 V only. Though this photovoltage is just enough to split water, it is not sufficient to overcome the overpotential developed due to H_2 and O_2 gas evolution on electrode surfaces during water splitting reaction. High maximum photovoltage (> 2.0 V) and high photocurrent generating solar cells are needed to be used in a monolithic PEC for the self-driven photosplitting of water with high solar to hydrogen efficiency (≥ 10.0 % [13]). Furthermore, the thin layer of Mn-oxide on TiO_2 covered Tj-a-Si acted as a protective layer; however, it was not an efficient electrocatalyst for the oxygen evolution reaction (OER). The protective layer should possess high electrocatalytic activity towards OER. Though rare metal oxide such as RuO_2 acts as one of the best electrocatalyst for OER, some other earth abundant oxides such as Co-oxide [30, 31], Ni-Co-mixed oxide [32, 33] and other transition metal oxides and mixed oxides [34, 35] can be used as efficient catalysts for OER and as well as optically transparent protective layer to enhance the efficiency of water splitting reaction.

3.3. Scanning Electron Microscopic (SEM) Pictures of TiO_2 Covered and Mn-oxide–TiO_2 Covered Tj-a-Si

The Scanning electron microscopic (SEM) pictures of TiO_2 covered Tj-a-Si and Mn-oxide–TiO_2 covered Tj-a-Si are given in Figures **5**. It is observed that the TiO_2 layer was not thick enough to protect the underlying Tj-a-Si. Noticeably, the Mn-oxide-TiO_2 appears robust and thick enough to protect Tj-a-Si cell. However, at some darker spots the layer is porous and is not uniform where pinholes were generated after continuous water splitting for six hours in 2.5 M KOH solution. Hence, it is essential to deposit robust non-porous uniform thin films to avoid generation of pinholes during water splitting reaction.

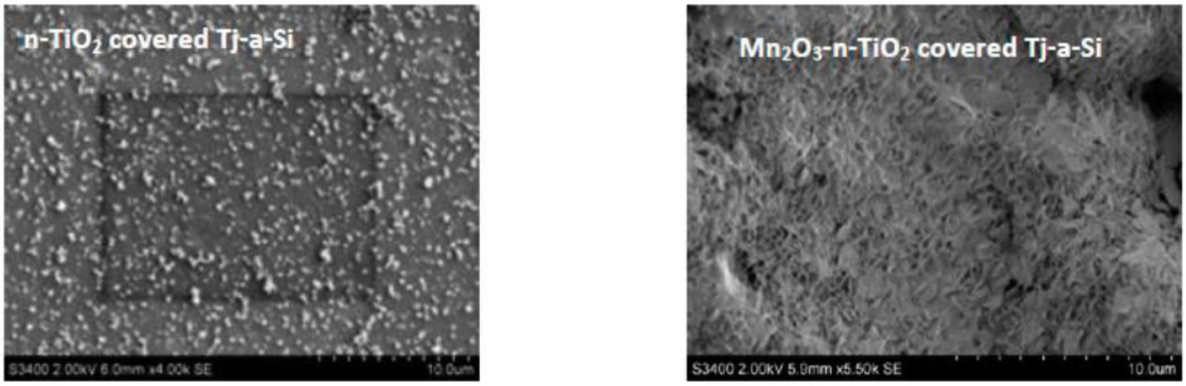

Figure 5: Scanning Electron Microscopic (SEM) pictures of TiO_2 covered Tj-a-Si and Mn-oxide-TiO_2 covered Tj-a-Si.

4. CONCLUSION

In this study Mn-oxide–TiO_2 coated Tj-a-Si was successfully used for monolithic self-driven water splitting to H_2 and O_2 having solar to hydrogen efficiency of 3.25 % under solar simulated light illumination of 1 sun. Higher solar to hydrogen efficiency could be achieved if higher maximum photovoltage generating Tj-a-Si cell were used and protected with a better corrosion resistant electrocatalyst layer. The coating on Tj-a-Si by a corrosion resistant transparent layer of Mn-oxide-TiO_2 was essential to enhance its stability during water splitting reaction which is more than ten-fold increase in stability compared to an earlier study [27] in which Tj-a-Si started to degrade sharply after half an hour in 0.1 M KOH solution. Long-term stability can be achieved if a pinhole free transparent corrosion resistant non-porous layer could be deposited on Tj-a-Si cells. Hence, future studies should focus on depositing a better adherent visible light transparent thin layer on the surface of Tj-a-Si for its stability for long operating time.

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