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Growth and characterization of large grained Poly-Si films grown on biaxially textured Ni-W substrate by hot-wire CVD

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ABSTRACT

Polycrystalline Si (Poly-Si) film with highly crystalline nature, and having most of the grains in the range of 50-100 µm has been grown over biaxially textured Ni-W substrate by Hot-wire chemical vapor deposition technique, using a single buffer layer of CeO_2 thin film. This result has been achieved for the SiH_4 source gas diluted to 95% with added H_2 gas, and for the substrate temperature of 840±10°C and a deposition pressure of 40 mTorr. XRD analysis shows that the Poly-Si films have grown with (111) and (200) orientations. Raman studies reveal that a crystalline volume fraction of 95.3% has been achieved. The imaginary part of pseudo dielectric function, $\langle\varepsilon_2\rangle$, as extracted from ellipsometric data, shows two prominent shoulders at energy positions 3.4 eV and 4.2 eV corresponding to the optical absorption of crystalline Si, indicating a high crystallinity of the Poly-Si film. SEM micrograph shows that the grown Poly-Si film is following the morphology and grain size as that of biaxially textured Ni-W substrate. SIMS analysis of the total multilayer structure shows a formation of very sharp interfaces, with no diffusion between Si and Ni, indicating that a single buffer layer of CeO_2 is sufficient to avoid the formation of nickel silicide while growing Si over Ni substrate. Thus, these results are very encouraging for the fabrication of Poly-Si film based solar cells with increased efficiency by minimizing the undesired recombination of charge carriers at grain boundaries. Copyright © 2015 VBRI Press.

Keywords: Poly-Si film; CeO_2 buffer layer; hot-wire CVD; Raman spectra; ellipsometer.

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Introduction

The fastest growing photovoltaic energy conversion technology is expected to become a major source of power generation in the future. As of today, 90% of all commercial solar cells are made of Si **[1, 2]**. Si materials presently used for photovoltaic cells include monocrystalline Si (c-Si), amorphous Si (a-Si) and, polycrystalline Si (Poly-Si). The c-Si solar cell has

maximum efficiency but is quite expensive, and the a-Si solar cells are cost effective solution but has low efficiency and suffers from photo degradation. Poly-Si is a compromise in between c-Si and a-Si in terms of cost and degradation with reasonably good efficiency of about 15% **[3, 4]**. A primary concern in the photovoltaic industry is the cost/watt which can be reduced by increasing the cell efficiency, low material consumption and adopting a process that includes minimum processing steps and a large growth rate of the material. A Si layer of 2-20 µm thick is sufficient for solar cell application and also the thin film solar cell can be formed in-situ during thin film deposition. Thus, both the material cost and the process cost can be reduced by using a thin film instead of a bulk material of about 250 µm thicknesses, as normally used, and avoiding slicing and polishing of Si ingots and related equipments **[5, 6]**. However, growing a Si film with adequate material quality has been a challenging task.

In Poly-Si material grain boundaries (GBs) are recombination-active and significantly reduce the diffusion length of charge carriers, and are the main reasons for the decreased efficiency of Poly-Si based solar cells **[7-9]**. This adverse GB effect can be reduced considerably in two ways: (i) with low density of GBs by increasing the grain size and (ii) by forming very low angled GBs (biaxially textured). One very simple technique to grow such Poly-Si films is to use a biaxially textured substrate having large sized grains as template **[8, 9]**. Following this template concept, Poly-Si films can be grown on variety of substrate materials. However, to grow high quality films with large sized grains the base materials should allow high temperature process. Also, for the given base material, it should be possible to prepare the biaxially textured surface very easily. The metal base is a good choice, because it is easy to prepare large area biaxially textured surface containing large sized grains simply by thermo-mechanical rolling process **[10]**. In addition, metal base allows high temperature processes. The fabrication of solar cell on metal substrates adds additional benefits to the cell in terms of increased conversion efficiency due to back reflection of light. Moreover, the metal allows making it flexible as thin foil, and fabrication of solar cells on such flexible metal substrates makes it feasible to some extent to place over lightly curved surfaces. Among various metal substrates Ni is preferred due to its high corrosive resistance. Cubic-textured Ni foils having large grains can be fabricated easily by thermo-mechanical process. In practice, a little amount (~ 5%) of tungsten (W) is added to Ni to provide good mechanical strength to it **[11]**. However, Si films cannot be deposited directly on to Ni substrate due to large lattice mismatch and also unavoidable formation of nickel silicide through diffusion between the Si layer and the metallic Ni substrate, and thus requires a buffer layer. CeO_2 has good structural compatibility with Si (< 0.1% lattice mismatch) and also has an excellent thermal stability, thus provides adhesion adequate to prevent delamination after Si film growth despite the large difference in the coefficient of thermal expansion (α) between Si (~ 2.5 ppm/K) and Ni substrate (~ 13 ppm/K). Therefore, a thin layer of CeO_2 can serve as a good buffer between Si and Ni **[12]**.

A cost effective deposition technique which allows depositing high quality Si films of about 20 µm thick at high deposition rates is desired. Hot-wire chemical vapor deposition (HW-CVD) technique is a simple and very attractive technique to deposit high quality Si films at high deposition rates, and with many other added advantages like high gas utilization, broad area deposition, low equipment cost etc., **[13, 14]**. In HW-CVD process, the source gases get dissociated mainly due to catalytic cracking reactions on the surface of a hot filament (normally at temperature in the range of 1500 to 2000^{o}C), and the solid byproduct species get deposited over the surface of a substrate kept nearby and contribute to form a continuous film. Silane (SiH_4) is normally used as a source gas with hydrogen dilution to grow different Si and hydrogenated-Si films. In HW-CVD process, atomic Si is the main content and the amount of SiH_n radicals is very small. Moreover, atomic H is produced very efficiently due to high filament temperature, therefore, hydrogenated-Si films can be deposited easily even at low hydrogen dilutions and low substrate temperatures. Due to the low hydrogen dilution requirements in HW-CVD technique, the possibility of occurrence of Poly-hydrogenation and formation of low density Si network (full of voids) is very less, thus leading to high growth rates and easy control of the required crystallinity of Si films. All these obvious features of HW-CVD process make this technique different from widely used PECVD technique and technologically acceptable **[15-21]**. Several workers have reported deposition of amorphous Si, microcrystalline (µc-Si), nanocrystalline Si (nc-Si) and crystalline Si films by HW-CVD technique **[22-26]**. Only recently, Teplin et al. **[8]** and Wee et al. [9] have grown c-Si film on Ni-W foil by HW-CVD technique with a buffer stake consisted of different oxide layers in order to get good quality epitaxial film for solar cell application. Findikoglu et al. **[7]** have also reported the growth of c-Si on polycrystalline Ni substrate using MgO/Al_2O_3 oxide buffer layers. However, a single buffer layer of CeO_2 may be sufficient for the growth of Poly-Si films on Ni-W substrate, and no details are available in this direction.

In this communication, we report the growth of highly crystalline Poly-Si film having large sized grains over biaxially textured Ni-W substrate using a single CeO_2 buffer layer by HW-CVD technique, and the results of its structural and optical characterizations.

Experimental

Materials

For this investigation, we have procured biaxially textured Ni-5at% W (Ni-W) substrate from the manufacturer Evico GmbH, Germany. The substrate parameters are: thickness 80 µm, surface roughness < 5 nm, texture > 98% cubic fraction, and majority of grains with lateral dimension in the range of 50-100 µm. CeO_2 (99.99%, China Rare Metal Material, China) target and Ar (99.999%, BOC, India) gas were been used for the deposition of CeO_2. SiH_4 (99.9999%, Praxair, India) and H_2 (99.9999%, Praxair, India) were used as source gases for the growth of Poly-Si films. Tungsten (99.999%, Murex Ltd, England) was used as a filament wire.

Method

In the first step, about 170 nm thick CeO_2 buffer layer has been sputtered on Ni-W substrate using CeO_2 target and Ar gas in an in-house designed and fabricated downstream RF magnetron sputtering system. The RF sputtering power, deposition pressure and substrate temperature were set at 200 W, 30 mTorr and 700°C, respectively for the deposition of CeO_2. In the second step, Poly-Si films were grown on sputtered CeO_2 buffer layer using an in-house designed and fabricated HW-CVD system. A Turbo-based pumping system (Edwards, model: STP-451C) backed by roots and rotary pumps was used to achieve the base pressure below 5×10^{-7} Torr. A filament assembly of 10 cm × 10 cm dimensions was used, in which, straight tungsten wires are mounted in parallel to each other with a separation of 1 cm. A well regulated DC power supply (Aplab, model: L3299) has been used to maintain the filament temperature of 1800±20°C. The filament temperature was measured by IR optical pyrometer (Raytek, model: 3i). A shutter arrangement, close to the substrate, was used to avoid any undesired deposition before starting the actual growth process. The substrate temperature was varied in the range 640-840(±10) °C through a thermocouple and temperature controller arrangement. SiH_4 was used as a source gas and was diluted to 95% with added H_2 gas. These process gases were introduced into the chamber through a gas shower-head arrangement for uniform distribution of gases over the filaments. The gas flow rates were accurately controlled by mass flow controllers (Aalborg, model: GFC-17). The deposition pressure was kept at 40 mTorr and controlled through a throttle valve (MKS, model: 600 series). It is to be noted that we have observed highly crystalline volume fraction for the Poly-Si film grown at the substrate temperature of 840°C, so the results are shown only for this temperature growth.

The structural properties of CeO_2 and Poly-Si films were characterized by an X-ray Diffractometer (Bruker, Germany, model: D8-Avalance), using Cu Kα radiation at 1.54 Å in θ-2θ geometry. The structural properties were also evaluated at room temperature by Raman Spectrometer (InVia Renishaw) using 514 nm Ar ion laser as the excitation source with 5 mW power. Determining the crystallinity of Si in the solar cell manufacturing is important because the presence of non-crystalline Si leads to reduced conversion efficiency. Distinguishing and quantifying crystallinity in Si can be done using Raman spectroscopy. Due to very uniform and ordered bond angles, bond strength and bond energy in crystalline Si, highly crystalline Si has very sharp peak centered at ~ 520 cm^{-1}. In non-crystalline (i.e., amorphous) Si, the bond angles, bond strength and bond energy vary leading to broad diffused spectral features around 480 cm^{-1} **[18, 27]**.

The optical properties of CeO_2 buffer layer and Poly-Si films were studied by Spectroscopic ellipsometer (J.A. Woollam, model: VASE32) and the optical parameters were calculated including the information on dielectric function in the optical energy range 1.0-5.0 eV, for the incident angles 65° and 75°. Ellipsometer is a model fitting based technique, which minimizes the difference between measured experimental and calculated fitting values as a function of photon energy or wavelength **[28]**. The thickness of CeO_2 and Poly-Si samples were measured using a Stylus Profiler (Ambios, model: XP-200). The surface morphology was examined by a Scanning Electron Microscope (SEM, model: LEO 400). The secondary ion mass spectrometry (SIMS, model: TOF-SIMS 5) measurements were carried out to see the interfaces in the multilayer structure for diffusion, if any, between Poly-Si and Ni-W substrate. SIMS measurements were done with 25 keV Bi1 beam for the Si, H, Ce, O, and Ni analyses.

Results and discussion

Crystalline nature of the grown Poly-Si films was investigated by recording the Raman spectra. We have used the XPS peak fitting software for de-convoluting the recorded Raman spectra, which normally de-convolute the Raman spectrum into three peaks. **Fig. 1** shows the de-convoluted spectra in the wavelength range 400-600 cm^{-1} of the Poly-Si film grown by HW-CVD for the temperature range 640-840°C.

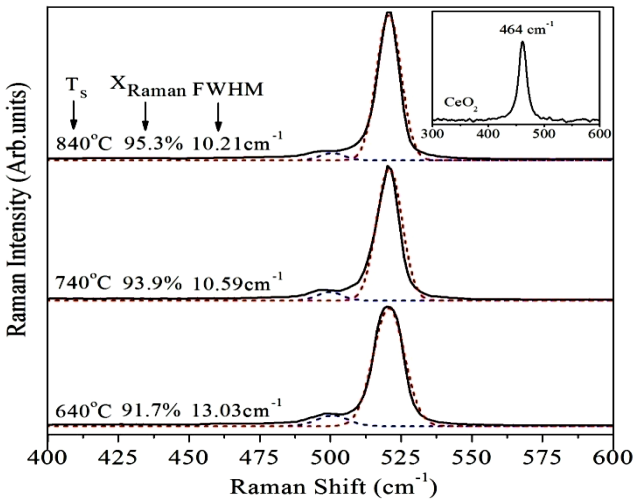

Fig. 1. De-convoluted Raman spectra of Poly-Si film grown at different substrate temperatures (Dotted lines present Gaussian peak fit spectra). Inset shows the Raman spectrum of CeO_2 buffer layer.

The recorded spectra are represented by solid lines and the peak fit spectra by dotted lines. The measured Raman spectra were found to be best fitted only for two Gaussian peaks. Thus, in our results, the de-convoluted Raman spectrum shows only two peaks, a sharp peak at ~ 520 cm^{-1}, which corresponds to the highly crystalline phase of Si and a secondary peak at ~ 500 cm^{-1} corresponding to an intermediate phase present in the film. The nature of the peak at 500 cm^{-1}, relatively low intensity and little broadened compared to the sharp intense peak at 520 cm^{-1}, indicates the intermediate phase as microcrystalline. These Raman results can be used to calculate the crystalline volume fraction (X_{Raman}) with the following relation:

$$X_{Raman} = \frac{I_{520}}{I_{520} + I_{500}}$$

Where I_{520} and I_{500} are the integrated intensities correspond to the crystalline phase and an intermediate phase at 520 and 500 cm^{-1}, respectively.

The crystalline volume fraction, $X_{Raman,}$ is found to be increasing from 91.7 to 95.3%, while the FWHM of Raman peak decreased from 13.03 to 10.21 cm^{-1} when the growth temperature was increased from 640 to 840°C. The rest of the crystalline contribution appears to be from the intermediate phase of microcrystalline structure. Raman spectrum of CeO_2 has also been recorded (see the inset of **Fig. 1**) and it shows a sharp intense peak at 464 cm^{-1}, corresponding to F_{2g} Raman mode of CeO_2, which indicates the formation of highly crystalline CeO_2 film. We have not observed any other peak arising due to defects or oxygen vacancies and hence confirm the high quality CeO_2 film **[29, 30]**.

Fig. 2 shows the XRD patterns of Ni-W substrate, as deposited CeO_2 and as grown Poly-Si films at the substrate temperature of 840°C. The XRD patterns of CeO_2/Ni-W (**Fig. 2b**) and Poly-Si/CeO_2/Ni-W (**Fig. 2c**) show two intense peaks corresponding to their (111) and (200) crystallographic orientations. It has been observed that the peak intensity of (200) orientation of CeO_2 film was increased compared to its (111) after the growth of Poly-Si film. This indicates alignment of more crystallites of CeO_2 with the (200) orientation of Ni-W substrate at the deposition temperature (840 °C) of Poly-Si films. The absence of any other peak in the XRD spectra indicates high purity of the films obtained.

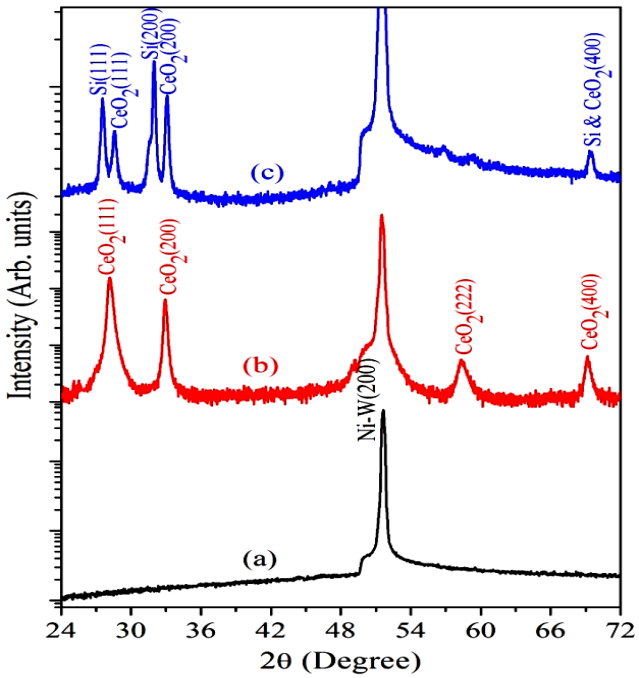

Fig. 2. XRD results for the (a) Ni-W substrate, (b) CeO_2/Ni-W buffer layer and (c) Poly-Si/CeO_2/Ni-W structure grown at 840°C substrate temperature.

Fig. 3 and **Fig. 4** represent the Spectroscopic ellipsometer (SE) data for the ellipsometric parameter (Ψ) of the Poly-Si film grown at the substrate temperature of 840°C, and CeO_2 buffer layer for the incident angles 65° and 75°, respectively. The solid line in the figures represents the model-fit data and it can be seen that all the features present in the experimental spectra are reproduced by the model fit. The fitting parameters within the parametric dispersion model yield thickness of Poly-Si film and CeO_2 buffer layer about 598±0.5 nm and 170±0.5 nm, respectively. These thicknesses are in good agreement with thicknesses (602±5 nm, and 172±5 nm) measured by the stylus profiler.

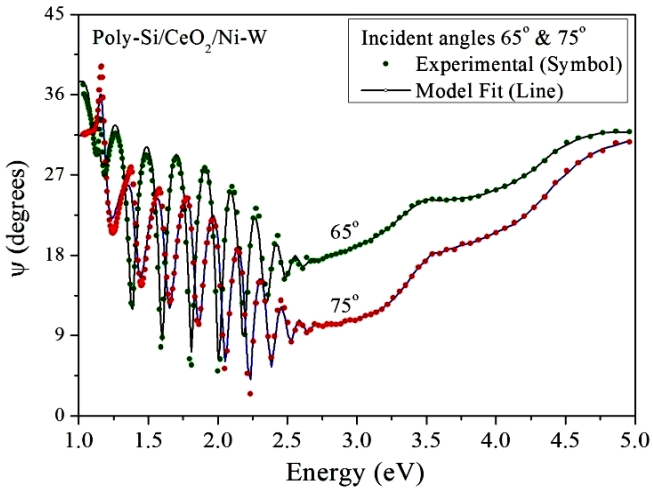

Fig. 3. Experimental and fitted ellipsometric parameter (Ψ) of Poly-Si/CeO_2/Ni-W structure grown at 840°C substrate temperature. Solid line indicates model fit.

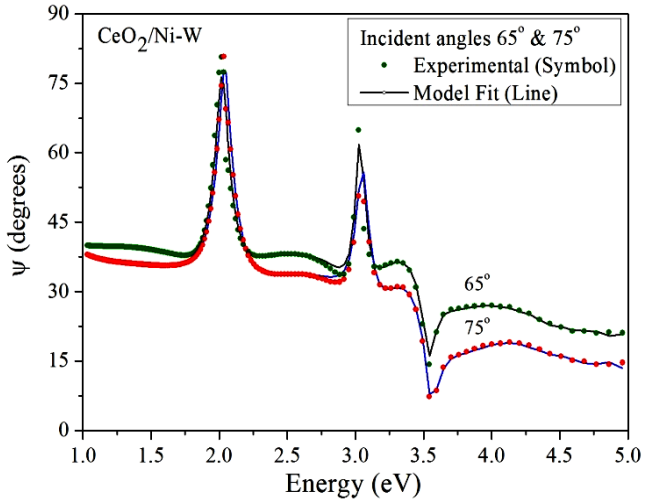

Fig. 4. Experimental and fitted ellipsometric parameter (Ψ) of CeO_2/Ni-W buffer layer. Solid line indicates model fit.

Fig. 5 shows the imaginary part of the pseudo-dielectric function, $<\varepsilon_2>$, as extracted from the SE data, and it contains two prominent broad shoulders at the energy positions 3.4 eV and 4.2 eV representing the optical absorption of crystalline Si, as predicted by the theoretical electronic band structure **[31, 32]**. For the clarity of presentation, the spectrum recorded for the incident angle of 65° is shown here.

The prominence of these two shoulders indicates excellent crystallinity of the grown Poly-Si film. The low energy photons penetrate the grown film causing the interference fringes to appear due to the reflected beam from the surface of the film and form the interface between the substrate and the film. Further, **Fig. 6** represents the refractive index (n) and extinction coefficient (k) values as a function of wavelength of Poly-Si film grown at the substrate temperature of 840 °C, as obtained from the corresponding ellipsometric data and are well matched to

the values as reported by ellipsometric determination of optical constants for Si in the literature **[28, 33]**. In the inset of **Fig. 6** the refractive index (n) and extinction coefficient (k) values of CeO_2 buffer layer are shown, and these are in good agreement with the values as reported in the literature **[29, 34, 35]**.

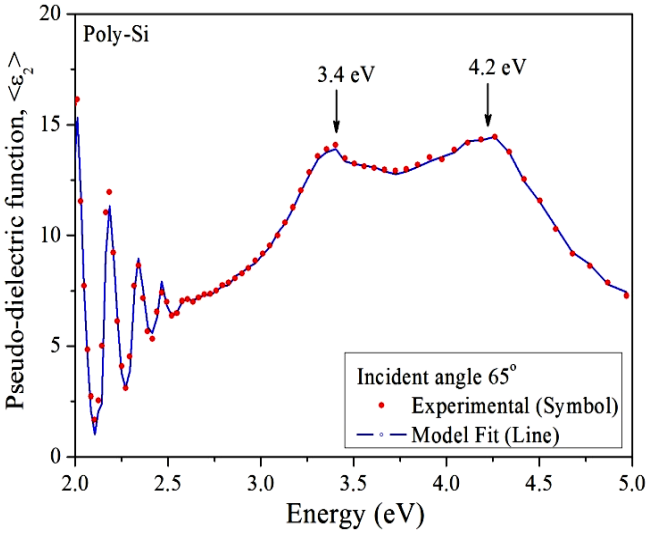

Fig. 5. Imaginary part of the pseudo-dielectric function, $<\varepsilon_2>$, spectrum of Poly-Si film on CeO_2 buffer layer sputtered Ni-W grown at 840°C substrate temperature. Solid line indicates model fit.

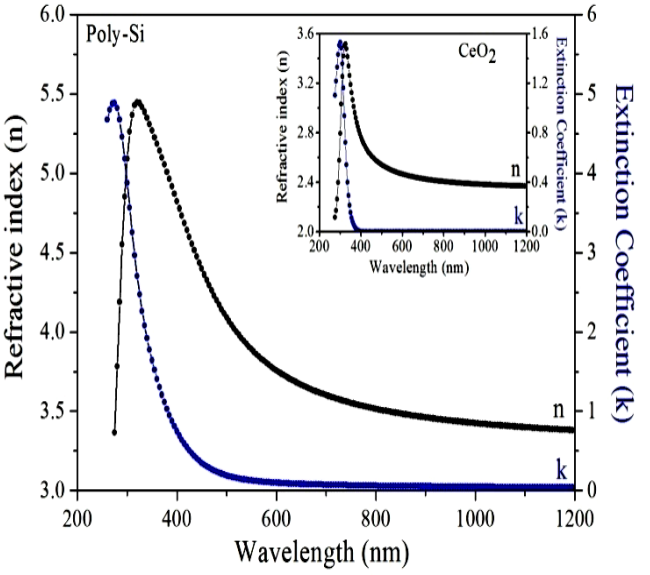

Fig. 6. Dispersion behavior of refractive index (n) and extinction coefficient (k) of Poly-Si film and CeO_2 buffer layer film (in the inset).

The SEM micrograph in **Fig. 7** shows the surface morphology of Poly-Si film grown at the substrate temperature of 840°C. Large sized grains of Poly-Si separated by GBs can be seen clearly in the micrograph. From the SEM micrograph, it is observed that most of the grains are having the size in the range 50-100 µm, following the texture of Ni-W substrate. The SIMS depth profile for the total multilayer structure Poly-Si/CeO_2/Ni-W (as shown in **Fig. 8**) reveals the formation of very sharp interfaces, with no diffusion of Si into CeO_2. This indicates that a single buffer layer of CeO_2 is sufficient to growing Poly-Si film over Ni-W substrate.

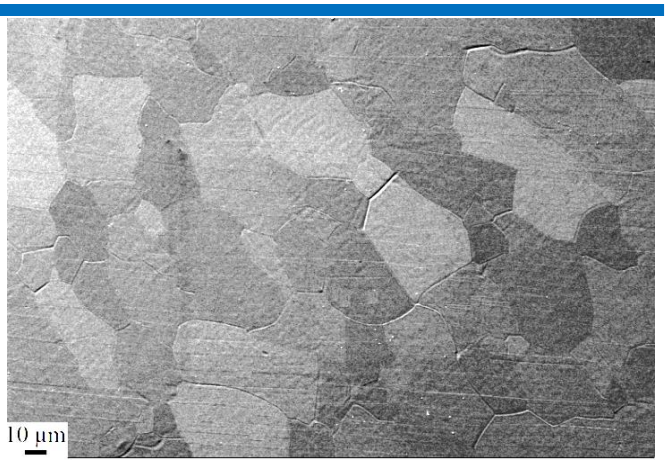

Fig. 7. SEM image of Poly-Si film grown at 840°C substrate temperature on CeO_2 buffer layer sputtered Ni-W substrate.

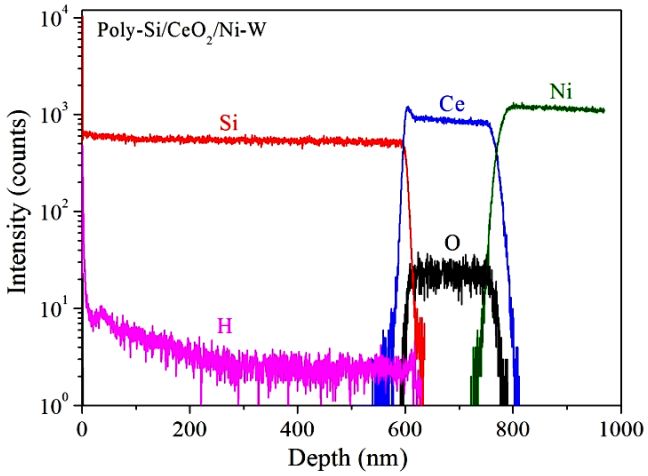

Fig. 8. SIMS depth profile of Si, H, Ce, O and Ni ions in Poly-Si/CeO_2/Ni-W structure grown at 840 °C substrate temperature.

Conclusion

Highly crystalline Poly-Si film with large sized grains can be grown on biaxially textured Ni-W substrate using a single buffer layer of CeO_2 for solar cell applications. XRD and ellipsometer results reveal high crystallinity of the grown Poly-Si film. Raman spectrum shows high crystalline volume fraction of 95.3% in the grown Poly-Si film at the substrate temperature of 840°C. SIMS investigation clearly shows sharp interfaces in the Poly-Si/CeO_2/Ni-W structure indicating that a single buffer layer of CeO_2 can prevent the diffusion between Si and Ni substrate to avoid the formation of nickel silicide. These results indicate that Poly-Si films can be deposited so as to minimize the grain boundary recombination which is a possible effective way to increase the efficiency of the solar cells.

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