

# Optical characterization of Intrinsic Poly Silicon Film for Photovoltaic Application on Sapphire and TiO<sub>2</sub> Substrate by HWCVD

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**Abstract:** We have investigated the possibility of growing silicon films, 2 to 3 micron thick, from silane-hydrogen mixture, by hot wire chemical vapour deposition (HWCVD). Several substrates i) oriented TiO<sub>2</sub> layer on glass, ii) crystalline sapphire have been used in this work. The growth is performed in two stages; a) nucleation step using dilute mixture SiH<sub>4</sub>:H<sub>2</sub> in the ratio 1:20 at 400 degC, followed by b) thickening step at 540 degC, in which SiH<sub>4</sub>:H<sub>2</sub> ratio is gradually enhanced to 8:20. Transmission and reflection spectroscopy have been used for determining optical characteristics of the film.

**Keywords :** HWCVD; crystalline silicon, reflectance

## I. INTRODUCTION

Large grain polycrystalline silicon film can be grown using several techniques such as sputtering, low pressure chemical vapour deposition (LPCVD), plasma enhanced chemical vapour deposition (PECVD), plasma immersion ion implantation (PIII), and HWCVD. Among these processes for sputtering, optimization needs to be done for gate dielectric deposition, doping, activation and hydrogenation to control the growth condition. However, RF sputtering tends to yield low deposition rates [1]. LPCVD techniques involved residual stresses and residual stress gradients of the film which must be controlled. Deposited films with high residual stresses and residual stress gradients through the film thickness can cause to increase the stiffness of the film and can also cause deformation of asymmetric structures. [2]. Growth of poly silicon films deposited at PIII techniques depends on pulse repetition rate, microwave power, deposition time, substrate temperature and bias voltage. At low surface temperature the grains density is low [3]. PECVD technique for poly silicon film deposition is a low temperature process, below 300 degC [4]. Hot-wire chemical vapour deposition has been shown to be a promising techniques for growing high quality hydrogenated amorphous silicon films at low (below 250 degC) substrate temperature [5,6]. Low-temperature (<600 degC) epitaxial thick film were reported using HWCVD [7,8,9,10]. The motivation of this work is to evaluate the feasibility of the HWCVD techniques to deposit large grained poly silicon thick film at high deposition rate of 4.5 Å/sec (0.15 Å/sec [11]). Evaluation of various topographical and optical properties of the films is also a concern of this study. Another strategy of our study consists of the growth of poly silicon thick films on different substrates. This will help to

find suitable and low cost substrate. Here we have used two different substrates sapphire and TiO<sub>2</sub>. We aim to use them as substrates for crystalline silicon solar cell.

## II. EXPERIMENTAL

### A. Substrate preparation

For TiO<sub>2</sub>, we used (112) oriented anatase TiO<sub>2</sub> synthesis by atmospheric chemical vapour deposition (ACVD) techniques on ITO coated glass substrate by Washington University in St Louis, Missouri, USA. Sapphire (Al<sub>2</sub>O<sub>3</sub>) substrates were both side polished hexagonal c-plane oriented. For cleaning of all the substrates before putting into the reaction chamber we blow 99.999% pure N<sub>2</sub> gas in the laminar flow bench.

### B. HWCVD system

Silicon films were synthesized using the HWCVD apparatus. All thin film growth experiments were performed in the HWCVD system having base pressure of no higher than  $1 \times 10^{-6}$  mbar. Process pressure were maintained between  $1 \times 10^{-1}$  mbar to  $1.3 \times 10^{-1}$  mbar. A throttle valve at the exit controls the reactor pressure by changing the volumetric flow rate of the gases leaving the reactor. A mixture of pure SiH<sub>4</sub> and pure H<sub>2</sub> were used as process gas. A single filament of tungsten wire, 65 cm long and 0.5 mm diameter wound in a zigzag shape was resistively heated to 1900 degC using a dc power supply about 5 cm height from the substrate. The area covered by the filament is about 9 cm × 9 cm. The wire radiatively heated the substrate to 250 degC. The temperature of the substrate was determined in situ using a thermocouple in contact with the top of substrate. Higher substrate temperature was achieved by a resistive heater placed in contact with the substrate from below. A translatable shutter between the wire and the substrate allows several growth processes to be performed under identical gas ambient, wire temperature and also provide a definite starting and ending point for film growth.

Deposition rate and film thickness were determined after the deposition process from dummy wafer that placed inside the reaction chamber with a step using Profilometer. Using HWCVD, Silicon nucleation was grown on Sapphire and TiO<sub>2</sub>.

### C. Growth Process of undoped silicon film

#### 1) Nucleation Stage

The nucleation state was grown at 400 degC temperature for 120 sec with H<sub>2</sub> : SiH<sub>4</sub> ratio of 20 : 1 gas flow.

#### 2) Thickening stage

After thin layer has been deposited, poly silicon thick films were grown on Sapphire and TiO<sub>2</sub> substrates. Keeping the filament on, we increased the substrate temperature to 540 degC using the substrate heater from below. After getting the required temperature for the substrate we turn on gas flow and increased the H<sub>2</sub> : SiH<sub>4</sub> ratio to 20 : 8. Process pressure of the chamber increased at the end because of the increased gas flow. Detailed about effect of process condition for depositing silicon film using HWCVD were discussed by Atul Pant *et al* [12].

### D. Growth Process of p-doped silicon film

#### 1) Nucleation Stage

The nucleation state was grown at 400 degC temperature for 120 sec with H<sub>2</sub> : SiH<sub>4</sub> : B<sub>2</sub>H<sub>6</sub> ratio of 15 : 1 : 5 gas flow.

#### 2) Thickening stage

We increased the substrate temperature to 540 degC using the substrate heater from below. After getting the required temperature for the substrate we turn on gas flow and increased the SiH<sub>4</sub> concentration keeping diborane (B<sub>2</sub>H<sub>6</sub>) flow fixed at 5 sccm. To keep the chamber pressure at 0.1 mbar we decreased H<sub>2</sub> flow in some stages.

### E. Film's surface topography

For characterization of thin film AFM data was taken into consideration. We have taken 1  $\mu\text{m} \times 1 \mu\text{m}$  area for imaging using tapping mode. Related parameters such as nucleation density, grain size (diameter) etc. were also determined from AFM images. SEM images of thick films were taken for viewing the feature size and shape.

### F. Experimental setup for optical measurement

For transmission and reflection spectroscopy we used a spectrometer that was capable of recording spectra in the visible range as well as in the near infrared and UV (300 nm to 1100 nm). Absorption coefficient of films on sapphire and TiO<sub>2</sub> substrates were calculated. The setup was having an integrating sphere thus the spectrometer did not measure the absolute values but compared the signal from the sample to a reference beam. Transmission and reflection data were not taken on the same spot of the sample as substrate has to be replaced on the other side of the integrating sphere.

### G. Determination of alpha ( $\alpha$ ) and optical band gap ( $E_g$ )

In this section we derived the absorption coefficients  $\alpha$  from the transmission and reflection measurements. We used the following approximation formula [13].

$$\alpha = -\frac{1}{d} \ln \left( \frac{T}{1-R} \right) \quad (1)$$

Here d is thickness of film, T is amount of transmitted light and R is amount of reflected light. The resulting curves are plotted with linear scale, see Figure 3.2 (b), 3.4 (a).

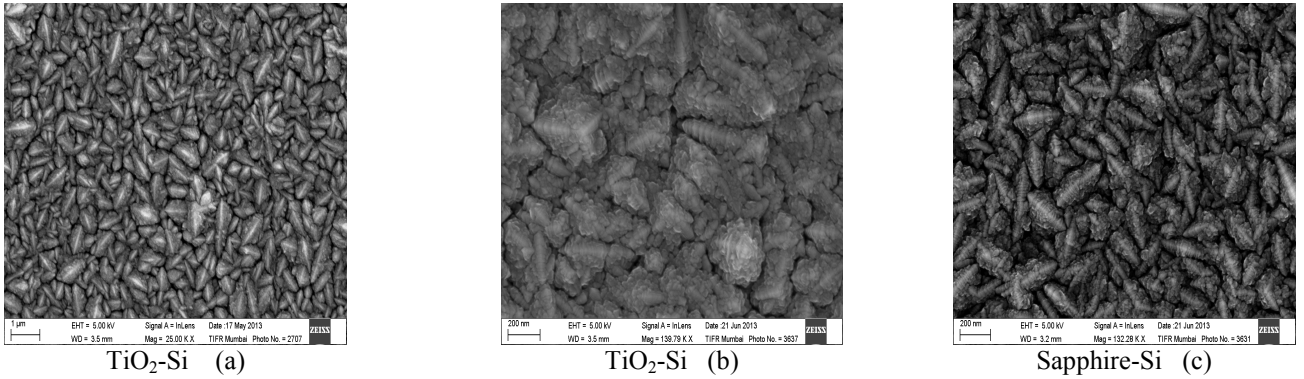


Fig 3.1: SEM image (a) TiO<sub>2</sub> 43% SiH<sub>4</sub>, 480 degC, (b) TiO<sub>2</sub> 29% SiH<sub>4</sub>, 540 degC, (c) Sapphire 29% SiH<sub>4</sub>, 540 degC

## III. RESULTS & DISCUSSION

### A. Surface topography using AFM for thin film

We observe an increasing in H<sub>2</sub> dilution leads to an increase in grain size. As we further approach to decrease the H<sub>2</sub> dilution, the grain size decreased. A decrease in H<sub>2</sub> dilution leads to decrease the process pressure from 3 mbar to 2 mbar which was not promising so we repeated the experiment and the process pressure were maintained at  $1.3 \times 10^{-1}$  mbar. An

increase in grains size observed for both substrates. Comparative to other substrate, grains on sapphire substrates are smaller, see Fig: 3.4.

### B. Surface topography using SEM for thick films

SEM (Raith 150) was used to characterize the surface topography such as grain size, grain shape of a series of films grown at 480 degC and 540 degC temperatures at different SiH<sub>4</sub> concentration from 2 micron to 5 micron in thickness. So

far we have three different growth conditions. With the increase in temperature, grain size increased for sapphire but not for  $\text{TiO}_2$  substrate. With the decrease in silane concentration grain size decreased for both substrates. From the SEM images, see Figure 3.1 (c) of Sapphire shows fish like features with a directional axis which are mostly from (220) crystal orientation. Crystal of orientation (111) has a shape of Bucky ball [14]. SEM image prevails that substrate effect is absent on Sapphire and  $\text{TiO}_2$  for thick films of more than 3 microns.

### C. Optical Characterization

In this section we will present the results of our transmission and reflection measurements and derive several properties of our semiconductor samples from them.

### D. Reflectance measurement using integrating sphere.

The interpretation of reflection spectrum for intrinsic poly silicon thick film on Sapphire and  $\text{TiO}_2$  has been done from Figure 3.2 & 3.4. For high energies, at UV range where absorption is high, reflection increases as well for thick film. The peak corresponding to the energy at 3.3 eV (375 nm) shows crystallinity of the film, see Figure 3.2 (a). An increase in temperature as well as decreasing the silane concentration increases the reflection in visible range for both samples, where for  $\text{TiO}_2$  its 2 % and for Sapphire its 5 %, see Figure 3.2 (a). For low energies we observe thin film interference effects that result from the overlaying of light that is reflected on both sides of the thin film. This interference fringes gives us an idea of the increase in film thickness due to change in the silane concentration, see Fig. 3.2 (a).

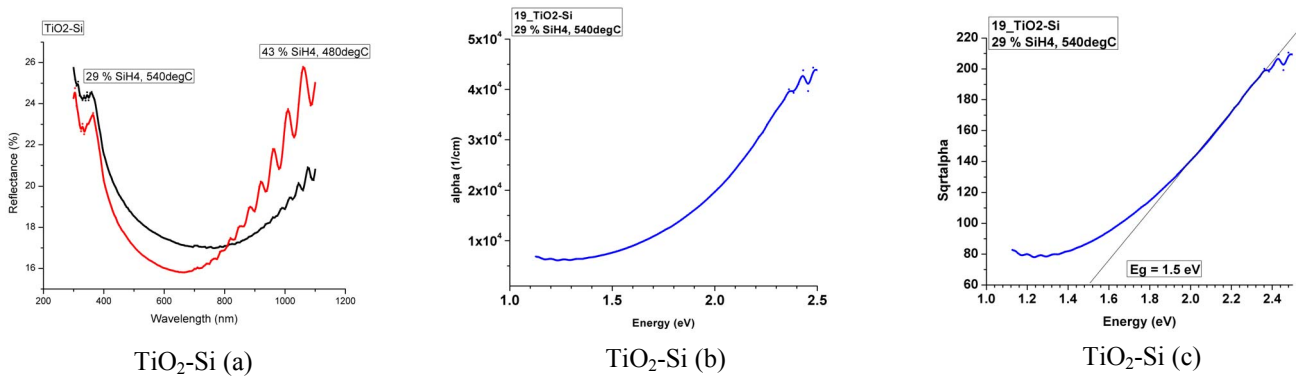


Fig 3.2: (a) Reflectance spectrum at two different growth conditions on  $\text{TiO}_2$  (b) Absorption coefficient of thick film on  $\text{TiO}_2$  (c) optical band gap of thick film on  $\text{TiO}_2$

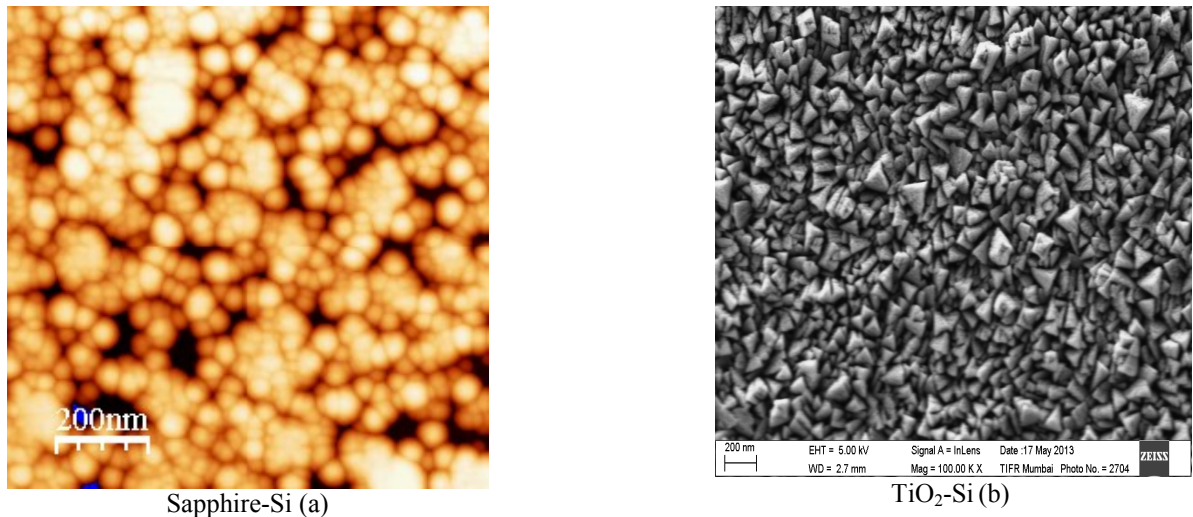


Fig 3.3: (a) Tapping mode AFM images of intrinsic poly silicon thin film (nucleation state) on sapphire (b) SEM image of thin film on  $\text{TiO}_2$  substrate.

### E. Determination of alpha ( $\alpha$ ) and optical band gap ( $E_g$ )

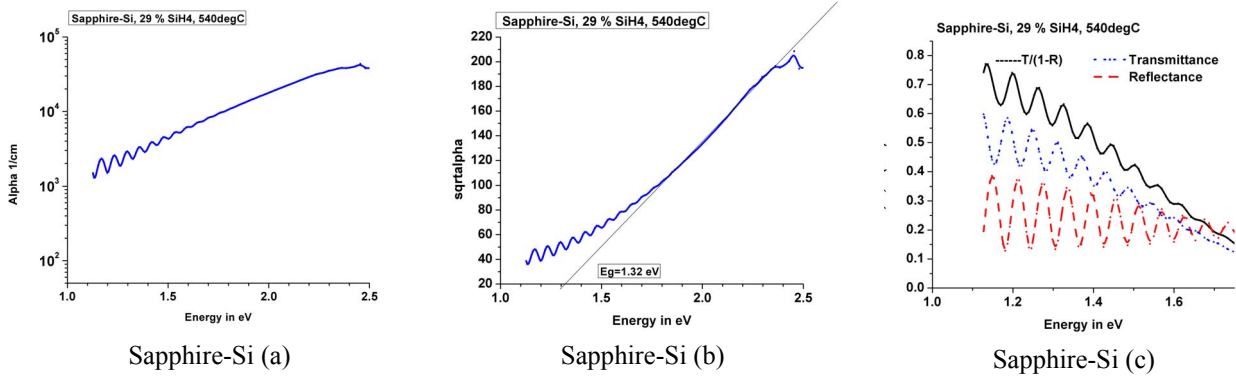


Fig 3.4 (a) Absorption coefficient of thick film on sapphire (b) optical band gap of thick film on sapphire (c) combined reflectance and transmission data on Sapphire.

### F. Transmission measurement using integrating sphere.

The spectrum (see Fig 3.4) shows that for high energies there are no transmission because all the light is absorbed. For low energies there are no appropriate electronic transitions possible so transmission is very high in this range. The interpretation of transmission spectrum for intrinsic poly silicon thick film on Sapphire and  $\text{TiO}_2$  has been done from Figure 3.4 (b) & 3.2 (c). These transmission data shows the energy at which absorption starts for thick film on Sapphire at 1.32 eV see Fig 3.4 (b) and for thick film on  $\text{TiO}_2$  at 1.5 eV see Fig 3.2 (c). Absorption coefficient for film on sapphire and  $\text{TiO}_2$  at 1.32eV and 1.5eV was  $1.6 \times 10^4 \text{ cm}^{-1}$  and  $7.6 \times 10^3 \text{ cm}^{-1}$  respectively see Fig 3.4 (b), 3.2 (b). Optical band gap of intrinsic poly silicon thick films on Sapphire and  $\text{TiO}_2$  substrates were calculated.  $\alpha$  start to oscillate and even become zero as the Equation 1 is valid only for high  $\alpha$ . In this high absorption, the transmission become close to zero. Optical band gap determined as  $E_g$  of Thick film on Sapphire is 1.32 eV and for  $\text{TiO}_2$   $E_g$  is 1.5 eV. Optical band gap for amorphous hydrogenated silicon is 1.69 eV using PECVD [15] and for crystalline poly silicon is 1.2 eV [13].

### IV. CONCLUSION

We have explored the viability for the growth of large grain (220) oriented intrinsic poly crystalline silicon films on Sapphire and  $\text{TiO}_2$  substrates for photovoltaic application. Grain size was as large as 700 nm reported for 43 %  $\text{SiH}_4$  concentration. Films on these substrate contains both amorphous and crystalline phase. For film on  $\text{TiO}_2$  substrate an increase in silane concentration enhance the crystallinity of the film. As films on sapphire and  $\text{TiO}_2$  substrates got lifted due to high stress, we reduced Silane concentration to 29 % and grain size of 400 nm obtained. In this stage the film got highly sticky to both substrates. The stable thicknesses of the poly silicon thick film were 2.2 micron with 29 %  $\text{SiH}_4$  at 540 degC temperature. For photovoltaic application sapphire can be used as it is but for  $\text{TiO}_2$  silane concentration has to be increased as well as film stress has to be maintained so film does not get lifted.

### V. ACKNOWLEDGEMENT

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