PHOSPHORUS DOPANT DIFFUSION, ACTIVATION, AND ANNEALING. USING INFRARED LASER FOR SYNTHESIS OF $n$-TYPE SILICON THIN FILM

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Abstract

Thin film of oriented crystalline intrinsic polysilicon films were grown on alkali-free borosilicate glass substrate using hot-wire chemical-vapor-deposition (HWCVD) technique. A layer as a source of phosphorus dopant on top of intrinsic polysilicon films were introduced in two different approaches: (i) spin-on one-micrometer-thick phosphorus dopant and (ii) phosphorus ion implantation. We investigate the possibility of dopant diffusion, activation, and annealing, using the irradiation of 1064 nm wavelength infrared laser. The annealing is performed under various conditions. The laser power and scan speed are varied to ensure the suitable laser annealing condition. We carry out resistivity measurements to validate the laser annealing process. For structural investigation, we use several characterization techniques, such as scanning electron microscopy, high-resolution X-ray diffraction, photoluminescence spectroscopy, and confocal Raman spectroscopy measurements. We use optical transmission spectra for determining optical characteristics of the film. The electrical measurement shows that the phosphorous-doped $n$-type polysilicon films are suitable as an emitter layer in photovoltaic device.

Keywords: Hot-wire chemical vapor deposition (HWCVD), infrared laser, $n$-type silicon, thin film, ion implantation, spin-on dopant.

1. Introduction

The polycrystalline silicon thin film on glass for photovoltaic application has become a low-cost solution in the recent years. This reduces the cost and dependence of crystalline-silicon-wafer-based technology in the field of optoelectronics [1]. The growth of polycrystalline silicon film on glass, using low-cost hot-wire chemical-vapor-deposition (HWCVD) technique shows promising alternate method...
over plasma-enhanced chemical-vapor-deposition (PECVD) technology [2, 3]. Though HWCVD technique is regarded as high filament temperature (1600°C–2000°C) process but the substrate can be kept at low temperature (200°C). Hence a low-temperature growth process is observed with a higher growth rate (16 Å/s) [4]. This feature makes HWCVD compatible with PECVD. The intrinsic silicon-film grown on glass substrate using HWCVD requires impurity doping, followed by dopant activation and annealing, for further application in photovoltaics. There are several established techniques for the doping process, such as HWCVD [4], atmospheric diffusion furnace [5], ion implantation [6], spin-on dopant [7], sputtering [8], etc. The dopant diffusion, activation, and annealing are explained in other published literatures that have used rapid thermal processing [9], laser processing [10, 11], atmospheric diffusion furnace [12], microwaves [13], etc.

In this article, we describe the fabrication of oriented crystalline intrinsic polysilicon (i-polySi) thin film on glass, using hot-wire chemical-vapor-deposition (HWCVD) technique [14]. The doping of phosphorus into these films were introduced with a pre-doped layers, using phosphorus-ion implantation [6] and spin-on phosphorus dopant (SOD) [7]. An infrared (IR) laser of 1064 nm was used for diffusion, activation, and annealing of the phosphorus dopant for samples prepared in (i) ion implantation, (ii) spin-on phosphorus dopant, and (iii) HWCVD techniques [14]. It is observed that all these three routes are useful for the fabrication of n-type silicon film on glass for photovoltaic application. However, the ion implantation process requires huge cost, where SOD process requires isolated environment, such as glove box as it is exhaustible; also it added few additional steps, for example, spin coating, heating, and solvent removal.

2. Experimental Details

2.1. Growth Process of Intrinsic Silicon Film

Thin 20 nm nucleation layers were grown on alkali-free borosilicate Corning 7059 glass substrate at 400°C with a gas ratio SiH₄ : H₂ = 1:20 for 100 s. For thickening stage, a mixture of SiH₄ and H₂ were used as process gas with a ratio of SiH₄ : H₂ = 5:15 for 18 min at 600°C substrate temperature [15]. The intrinsic silicon film was annealed at this stage under 20 SCCM (standard cubic centimeters) of H₂ flow for 30 min, followed by a H₂ soaking during cooling the sample from the growth temperature to a lower temperature of 200°C for another 45 min. The thickness of the film was 500 nm measured using DektakXT surface profiler. The detail of samples prepared in this study are given in Table 1.

2.2. Spin-on Phosphorus Dopant

Ethanol-based phosphorus (P) solution from Filmtronics, PA, USA was used as dopant for spin-on. An 1 μm thick layer of phosphorus dopant was coated by spinning the sample on a spinner at 3000 rpm for 45 s followed by 15 min post baking at 250°C on a hot plate. In Fig. 1, we show the schematic of different layers on glass substrate during spin-on dopant process. The spinning and baking were performed under controlled atmosphere.

![Fig. 1. Schematic of different layers of silicon film on glass.](image-url)
Table 1. List of Samples: Synthesis of Intrinsic Polysilicon Film Using HWCVD, n-Type Silicon Film Using Ion Implantation, Spin-on Dopant, and HWCVD Techniques on Glass Substrate.

| Sample name | Process                                      | Recipe                                      |
|-------------|----------------------------------------------|---------------------------------------------|
| 82_Glass    | Intrinsic silicon film using HWCVD           | 500 nm thick intrinsic polysilicon film on glass |
| 70_Glass    |                                              |                                             |
| 83_Glass    | Ion implantation                             | Phosphorus ion implanted into 500 nm thick intrinsic polysilicon film on glass |
| 84_Glass    |                                              |                                             |
| 61_Glass, 66_Glass | Spin-on dopant (SOD) | Spin 1 \( \mu m \) phosphorus solution on 500 nm thick intrinsic polysilicon film on glass |
| 74_Glass, 78_Glass |                                              |                                             |
| 87_Glass    | \( n \)-type silicon film using HWCVD       | 180 nm phosphorus-doped silicon film as grown on glass substrate without intrinsic silicon film on the glass |

inside glove box. Standard atmospheric variables as chamber pressure kept between 2–4 mbar, amount of oxygen was between 1 and 2 ppm, and moisture level maintained at 0.70 to 1 ppm. A sealed stainless steel chamber of two-inch diameter, with a quartz window, was used to take the sample outside the glove box. The samples were kept inside the same chamber during laser annealing.

2.3. Phosphorus Ion Implantation

In this study, EATON NV 3204 medium-current ion-implantation system was used for phosphorus-ion implantation. The phosphorus ions were implanted at 100 keV energy on intrinsic polysilicon film on glass substrate without any mask. The process used solid phosphorus source to have phosphorus vapor using free-man-type source. The implant doses were \( 10^{15} \) ion/cm\(^2\). For implantation with 100 keV phosphorus ions, the range is expected to be about 0.12 \( \mu m \) and straggle, about 0.05 \( \mu m \) [6]. During implantation, beam line vacuum was kept 1 \( \cdot 10^{-6} \) mbar, and sample chamber pressure was 2.66 \( \cdot 10^{-6} \) mbar. The focused ion beam scans the sample surface in a highly-controlled manner to achieve uniform implantation over the 1\( \times \)2 cm sample area. The sample was kept stationary during the scanning of ion beam.

2.4. Phosphorus-Doped Si Film Using HWCVD

Thin phosphorus-doped microcrystalline silicon layer was deposited in a HWCVD system different from the HWCVD system used for the growth of intrinsic polycrystalline silicon [4]. The growth was done at 350ºC substrate temperature with a tantalum filament kept at a temperature of 1650ºC. Base pressure in the system was 1 \( \cdot 10^{-6} \) mbar, while process pressure was maintained at 6.6 \( \cdot 10^{-2} \) mbar. The \( n \)-type doped microcrystalline silicon films were grown on glass substrate using gas flow ratio of \( \text{SiH}_4:\text{H}_2: \text{PH}_3 = 2:50:1 \) SCCM. The growth duration was 45 min for 180 nm thin film.
2.5. Laser Annealing of Phosphorus-Doped Silicon Film

An infrared laser from single-emitter diode source with wavelength 1064 nm by SPI Lasers Limited, Germany was used to activate, dope, and anneal the intrinsic polysilicon film from pre-doped phosphorus layer coated on top surface [16]. The laser operated in transverse electromagnetic mode (TEM-00) with a Gaussian beam profile. The laser frequency was kept 10 kHz with a power output of 0.8 W. Computer-controlled \( x \), \( y \), and \( z \) movements with a minimum line-to-line translation of 5 \( \mu \)m were designed by Scantech Laser, India. This arrangement of infrared laser tool, shown in Fig. 2, allowed to control the stage movement also referred to scan speed to a range of 10 to 100 mm/s. A beam delivery cable and beam focusing lens with 58 mm focal distance were used to focus the beam on to the sample with a 10 \( \mu \)m beam-spot diameter in focused condition. A red beam as reference was used to align the sample scan area before the laser exposur. The pulse duration and scan speed were two key parameters, which were varied to reach the required annealing condition. The details of laser parameter such as scan area, pulse duration, scan speed, etc. are given in Table 2. Samples were kept inside a nitrogen-filled sealed container with a quartz window to expose laser during annealing process. The packaging was performed in standard glove box, 1.2 bar of 99.999\% pure nitrogen (\( \text{N}_2 \)) gas jet was used during laser annealing to ensure the cooling of lens as well as the quartz window.

**Table 2.** List of Samples: Dopant Diffusion, Activation, and Annealing of \( n \)-Type Silicon Film on Glass Substrate Using Infrared Laser.

| Sample  | Scan area mm\(^2\) | Laser annealed region | Laser pulse duration \( \mu \)s | Laser fluence J/cm\(^2\) | Scan speed cm/s |
|---------|-------------------|----------------------|-----------------------------|----------------|----------------|
| 61_Glass_SOD | 7×12 | R1 R2 R3 | 9.5 10 10 | 9.7 10.2 10.2 | 2 4 2 |
| 66_Glass_SOD | 3×12 6.5×12 | R1 R2 | 9.5 9 | 9.7 9.2 | 4 4 |
| 74_Glass_SOD | 10×4 | R1 R2 | 9 9.5 | 9.2 9.7 | 2 4 |
| 78_Glass_SOD | 2.5×12 | R4 | 9 | 9.2 | 1 |
| 83_Glass_Ion | 7×12 | R1 R2 R3 | 12 11.5 11 | 12.3 11.7 11.2 | 4 4 4 |
| 84_Glass_Ion | 6×12 | R1 R2 R3 | 10.5 10.5 10.5 | 10.7 10.7 10.7 | 4 2 3 |
| 87_Glass_n-Si | 3×8 8×14 | R1 R9 | 10 10 | 10.2 10.2 | 3 3 |
3. Results and Discussion

3.1. Topographical Analysis

We employed a field-emission gun-scanning electron-microscope (FESEM) images at different magnification to evaluate the submelting condition of grains due to laser exposer. In Fig. 3a, we present FESEM images of intrinsic polysilicon film on glass while in Fig. 3b, c, d, we present the laser-irradiated n-type silicon film of SOD, ion, and HWCVD, respectively. The estimated temperature of melting region is expected to be in between 1000°C to 1400°C. It can be seen that grains have melted, regrown, and merged to form larger grains due to the laser irradiation. The grains as large as 10 μm size are achieved by these laser-irradiation processes. These grains are well connected because of recrystallization processes. From resistivity measurement, as described in Sec. 3.7, the improvement of conductivity due to laser

![FESEM images of silicon films](image1)

Fig. 3. FESEM image of 70_i-polySi as grown i-polySi film on glass before laser irradiation (a), FESEM image of 70_i-polySi laser-annealed spin-on phosphorus-dopant (SOD) silicon film on glass (b), FESEM image of 84_i-polySi laser-annealed phosphorus-ion-implanted silicon film on glass (c), and FESEM image of 87_R9_i-polySi, laser-annealed HWCVD-grown phosphorus-doped silicon film on glass (d).
irradiation is also observed. In this work, we established that, with the laser system, it is possible to increase the conductivity of silicon film on glass.

3.2. Ion-Induced Damage Analysis

Phosphorus-ion-implantation process allows ion to penetrate the target by colliding and displacing the silicon atoms in its path. Both the ion and displaced host atom continue further damage until they became halt as the energy is spread over larger number of atoms. As a result, after a complete process of ion implantation, the initial polycrystalline silicon film becomes highly disordered in crystalline state. Eventually the film became amorphous [17]. Raman spectroscopy spectrum in Fig. 4a shows a broad Raman peak at 480 cm$^{-1}$, and high-resolution X-ray diffraction shown in Fig. 4b demonstrates a background bump with a (220) plane orientation [18], which establishes the amorphous condition of the silicon film.

3.3. Crystallographic Orientation

For ion implanted sample, in Fig. 5a, we show that the laser-annealed n-type silicon films have different directions of growth. We observed that the (111) and (220) both peaks had intensity of the same order, hence this film became randomly oriented in various crystal planes. Figure 5b demonstrates the laser-annealed SOD film. The laser irradiation changed the crystalline orientation of spin-on phosphorus-dopant silicon film. However, a preferential growth along (220) orientation is observed in Fig. 5b. Intrinsic polysilicon thin film grown on glass substrate shows a particular (220) directional crystalline growth; see Fig. 5c.

3.4. Raman Spectroscopy

The Raman spectra for several samples were recorded to quantify the crystallinity of i-polySi film, as well as for laser-treated films using a 514 nm laser; see Fig. 6. The penetration depth of Raman laser beam is 1 $\mu$m into the film, which is comparable to the thickness of the sample, that means the findings

![Raman Spectroscopy](image)

**Fig. 4.** Sample 84, Raman spectrum of phosphorus ion implanted intrinsic polysilicon film (a) and high-resolution X-ray diffraction spectrum of phosphorus-ion-implanted polysilicon film (b).
provide an average value with respect to the depth of the sample. Raman peak position observed at 519 cm$^{-1}$ for ion-implanted laser-annealed sample is shown in Fig. 6a, while at 517 cm$^{-1}$ for spin-coated laser-annealed sample it is shown in Fig. 6b, and at 519 cm$^{-1}$ for i-polySi film on glass it is shown in Fig. 6c. The Raman peak shift towards the lower wavelength (below 521 cm$^{-1}$) can be explained as the tensile stress in i-polySi thin film due to laser annealing. This deviation of the peak position also attributed to the lack of complete crystallinity.

3.5. Photoluminescence Spectrum

Photoluminescence (PL) spectrum of doped and intrinsic polysilicon film on glass has advantage of being noncontact and nondestructive, allowing characterization of sample before and after each step of synthesis sequence. In Fig. 7a, b, c, we show room-temperature PL spectra of laser-annealed P ion implanted, laser-annealed SOD, and as grown intrinsic polysilicon film, respectively. Using of shorter wavelength laser allows shorter absorption depth and results in more carriers generated in phosphorus-doped films. Also carriers generated by short wavelengths are generated further from glass-side surface [19].
The PL peak shift is observed towards 1.1 eV for laser-annealed P-ion-implanted and SOD silicon films shown in Fig. 7a, b compared to as grown film shown in Fig. 7c, which shows less defects and radiative recombination due to laser annealing.

![Fig. 7. Photoluminescence (PL) spectrum of 83_R3_laser-annealed P-ion-implanted i-polySi film on glass (a), 61_R3_laser-annealed SOD i-polySi film on glass (b), and 82_i-polySi film on glass (c).](image1)

![Fig. 8. Transmission spectra on glass substrates of 83_R3_laser-annealed phosphorus-ion-implanted silicon film (a), 74_R2_laser-annealed spin-on-phosphorus-dopant silicon film (b), and 82_i-polySi film (c).](image2)

3.6. Transmission Spectrum

The transmission spectra were taken in a range of wavelength from 300 to 1100 nm, which covers ultraviolet–visible–near-infrared (UV-VIS-NIR) using integrating sphere [20]. The interferences fringes in intrinsic polysilicon films are due to the multiple interference of light reflected back and forth from the substrate and the film; see Fig. 8c. These spectra also show that, for high energies, there is no transmission, because all the light is absorbed on the other hand for low energies, and there are no appropriate electronic transitions possible, so transmission is very high in this range. Over the visible range, transmission was steady around 50% on both laser-irradiated ion-implanted and spin-on dopant silicon films shown in Fig. 8a and b, respectively. In the regions, where there is no film on glass due to laser irradiation, continue to transmit light even at wavelengths, where silicon has become opaque.
Table 3. Resistivity Data of Laser Annealed n-Type Silicon Thin Film on Glass for Spin-on-Phosphorus Dopant (SOD), Phosphorus-Ion-Implanted (Ion), and n-Type Silicon Film (n-Si) HWCVD.

| Laser annealed sample | Laser parameter | Voltage V | Current I | Sheet resistance $V \cdot 4.532/I$ Ω/□ | Thickness cm | Resistivity Ω·cm |
|-----------------------|----------------|----------|-----------|-------------------------------------|--------------|-----------------|
| 78_Glass_SOD_R4       | 1 9            | 8 $\cdot$ 10^{-2} | 4.53 $\cdot$ 10^{-8} | 8 $\cdot$ 10^{6} | 5 $\cdot$ 10^{-5} | 4 $\cdot$ 10^{2} |
| 61_Glass_SOD_R1       | 2 9.5          | 2.2 $\cdot$ 10^{-1} | 1 $\cdot$ 10^{-6} | 9.9 $\cdot$ 10^{5} | 5 $\cdot$ 10^{-5} | 49               |
| 74_Glass_SOD_R1       | 2 9            | 3.25 $\cdot$ 10^{-1} | 4.53 $\cdot$ 10^{-5} | 3.25 $\cdot$ 10^{4} | 5 $\cdot$ 10^{-5} | 1.6              |
| 66_Glass_SOD_R2       | 4 9            | 7.1 $\cdot$ 10^{-3} | 4.53 $\cdot$ 10^{-6} | 7.1 $\cdot$ 10^{3} | 5 $\cdot$ 10^{-5} | 3.6 $\cdot$ 10^{-1} |
| 74_Glass_SOD_R2       | 4 9.5          | 4 $\cdot$ 10^{-1} | 4.53 $\cdot$ 10^{-4} | 4 $\cdot$ 10^{3} | 5 $\cdot$ 10^{-5} | 2 $\cdot$ 10^{-1} |
| 84_Glass_Ion_R2       | 2 10.5         | 7 $\cdot$ 10^{-1} | 1 $\cdot$ 10^{-6} | 3.17 $\cdot$ 10^{6} | 5 $\cdot$ 10^{-5} | 1.6 $\cdot$ 10^{2} |
| 83_Glass_Ion_R3       | 4 11           | 2.1 $\cdot$ 10^{-2} | 1 $\cdot$ 10^{-6} | 9.5 $\cdot$ 10^{4} | 5 $\cdot$ 10^{-5} | 4.75             |
| 84_Glass_Ion_R3       | 3 10.5         | 4 $\cdot$ 10^{-3} | 1 $\cdot$ 10^{-6} | 1.81 $\cdot$ 10^{4} | 5 $\cdot$ 10^{-5} | 9 $\cdot$ 10^{-1} |
| 84_Glass_Ion_R1       | 4 10.5         | 1.6 $\cdot$ 10^{-3} | 1 $\cdot$ 10^{-6} | 7.25 $\cdot$ 10^{3} | 5 $\cdot$ 10^{-5} | 3.6 $\cdot$ 10^{-1} |
| 87_Glass_nSi_R9       | 3 10           | 2 $\cdot$ 10^{-3} | 1 $\cdot$ 10^{-6} | 9.06 $\cdot$ 10^{3} | 1.8 $\cdot$ 10^{-5} | 1.6 $\cdot$ 10^{-1} |
| 87_Glass_nSi_R1       | 3 10           | 7 $\cdot$ 10^{-4} | 1 $\cdot$ 10^{-6} | 3.7 $\cdot$ 10^{3} | 1.8 $\cdot$ 10^{-5} | 5.7 $\cdot$ 10^{-2} |

3.7. Resistivity Data

Laser parameters were varied to obtain melting condition of i-polySi thin film to get the phosphorus atom be diffused (in the case of spin-on dopant), phosphorus ion be activated (in the case of ion implantation), and thin film be annealed (in the case of HWCVD). Resistivity was calculated from I-V measurements using four probe instruments given in Table 3. Scan speed and pulsed durations were varied to achieve lowest resistivity for n-type silicon film, which is in the range similar to other published literatures that have used IR laser [21]. Slow scan speed allows longer laser exposere, where pulse duration ensures specific energy for annealing condition. For spin-on phosphorus-dopant silicon film, the lowest resistivity was 0.2 Ω·cm, with the optimized laser pulse duration 9.5 μs and scan speed 4 cm/s. For phosphorus-ion-implanted silicon film, the lowest resistivity was 0.36 Ω·cm with the optimized laser pulse duration 10.5 μs and scan speed 4 cm/s. For HWCVD grown n-type silicon film, the lowest resistivity was 0.06 Ω·cm with the optimized laser pulse duration 10 μs and scan speed 3 cm/s. The resistivity of the HWCVD grown n-type silicon film was 0.5 Ω·cm before laser irradiation. We scanned 1 cm² area using 10 μm diameter spot size laser beam. Over this area, resistivity was not uniform. One possible reason might be that i-polysilicon film was not uniform over the 5×5 cm area, which could lead to nonuniform energy absorption from laser beam. Another reason is that the laser annealed films are not uniform, as seen in the SEM imaging. The films become patchy and poorly connected. As a result, the measurements vary depending on the connectivity and contacts with the probes.
4. Conclusions

The feasibility of fabrication of \( n \)-type polycrystalline silicon film on glass using laser annealing and dopant activation process is performed. In this study, we established a technique for dopant diffusion, activation, and annealing of phosphorus in oriented crystalline intrinsic polysilicon film, using 1064 nm infrared laser. The doping and annealing occurred by melting of the surface region followed by liquid-phase epitaxial regrowth. The resistivity analysis along with other characterization shows that the doped layers are suitable to use for device fabrication as an emitter layer in photovoltaic application.

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