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Effects of solid phase crystallization by rapid thermal annealing on the optical constants of sputtered amorphous silicon films

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Rapid thermal annealing is applied to induce crystallization of sputtered amorphous silicon deposited on thermally grown oxide layers. The influence of annealing temperatures in the range of 600 °C–980 °C is systematically investigated. Using scanning-electron microscopy, ellipsometry and X-ray diffraction techniques, the structural and optical properties of the films are determined. An order-of-magnitude reduction of the extinction coefficient is achieved. We show that the optical constants can be tuned for different design requirements by controlling the process parameters. For example, we obtain a refractive index of ~3.66 and an extinction coefficient of ~0.0012 at the 1550-nm wavelength as suitable for a particular optical filter application where a high refractive index and low extinction coefficient is desired.

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1. Introduction

Polycrystalline silicon (poly-Si) has been widely investigated for use in thin-film transistors, solar cells, and various optical and thermal devices [1–4]. The performance of these devices relies on material qualities such as grain size, in-grain defect densities, level of surface roughness, and porosity of the film. Solid-phase crystallization (SPC) of amorphous Si (a-Si) upon low-pressure chemical vapor deposition (LPCVD) is a common method for growing poly-Si due to its simplicity and low cost as well as its capability to produce uniform and smooth surfaces with high reproducibility [5–7]. The SPC process, however, has limitations as it requires a long annealing time of ~20–60 h to transform to the polycrystalline phase with a large grain size and the attendant formation of in-grain defects [8–10]. As an alternative method, rapid thermal annealing (RTA) can be used for the crystallization of a-Si; the results are similar to those obtained by furnace annealing [11–14]. The particular heat treatment used affects the optical constants of the deposited material. Even though the crystallographic changes effected upon annealing of the Si films using chemical vapor deposition (CVD) techniques such as LPCVD and plasma-enhanced CVD have been widely studied, the influence of RTA with sputtered a-Si samples on the optical constants, i.e., the refractive index and especially the extinction coefficient of the film, has received less attention. Accordingly, in this paper we present an alternative technique applying direct sputtering and RTA to obtain thin a-Si films with favorable optical properties.

To provide context, we note that Modreanu et al. reported microstructures and refractive indices of as-deposited LPCVD Si films applying temperatures up to 650 °C [15]. Lioudakis et al. presented parametric analysis of ellipsometric angles (Ψ and Δ) of ion-implanted polycrystalline Si films annealed at various temperatures [16]. In the present work, we study the deposition and crystallization of a-Si thin films grown using sputtering and subsequent RTA. We explore the changes in the optical constants, i.e., the refractive index, n, and the extinction coefficient, k, as functions of annealing temperature and time. We report a significant order-of-magnitude improvement of the extinction coefficient relative to the as-deposited film. Our aim is to develop a method to produce high-quality a-Si film with a high refractive index and low loss for applications in photonics and optoelectronic devices. Therefore, we provide herein a systematic way to tune the optical constants according to different criteria.

2. Experimental details

All experiments were completed with 4-inch n-type Si (100) substrates. Upon annealing, the crystalline grain growth of a-Si depends on the as-deposited films. Hence, we performed numerous depositions under different sputter conditions set by the deposition parameters. The optimized process parameters are presented in Table 1.

Prior to a-Si deposition, the wafer went through a wet cleaning process; then a spin-rinse dryer removed the water from its surface. Wet thermal oxidation was conducted in a Tystar oxidation furnace for 600 s at 1100 °C yielding a layer of SiO_2 with a thickness of ~360 nm. Then we used an AJA ATC Orion Series UHV Sputtering System to deposit a ~1034-nm thick a-Si film on that 4-inch oxidized Si

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Table 1
Process parameters for a-Si deposition by sputter.

Parameters	Strike	Pre-sputter	Sputter
Pressure (Pa)	4.67	0.67	0.67
Gas Flow (SCCM)	30	30	30
Power (W)	60	150	150
Time (s)	60	120	18,000

wafer, with a deposition rate of 2.65 nm/min. We performed a-Si depositions by sputtering at chamber pressures of 1.33 Pa, 0.93 Pa, and 0.67 Pa and used a Woollam VAS ellipsometer with a 75 W light source including a high speed monochromator system to measure the refractive index of each film obtaining n ~3.09, ~3.481, and ~3.71, respectively, at the 1550-nm wavelength. The main chamber was pumped down to the base vacuum of ~1.067 × 10^{-5} Pa, applying no substrate heating. The correlation between the lower pressure as well as the lower deposition rate of the a-Si film and the increase in refractive indices indicates denser a-Si films [15]. For pressures lower than 0.67 Pa, the refractive index is found to decrease again. This data suggests that the near-optimum process condition is at 0.67 Pa with an Argon gas flow of 30 SCCM and a power of 150 W. We conducted nine-point ellipsometry measurements of the films to establish consistency in thickness d, n, and k, where n + ik denotes the complex index of refraction. A standard ellipsometric measurement technique is used to extract n, k, and d for our films. We use a function-based model layer (Cauchy layer) expressing n by a slowly varying polynomial function of wavelength as $n(\lambda) = A + B/\lambda^2 + C/\lambda^4$ and k as an exponential absorption tail as $k(\lambda) = \alpha e^{\beta(12{,}400(1/\lambda - 1/\gamma))}$ [17–19]. In the dispersion model, the six fitting parameters are weights A, B, and C; the extinction coefficient amplitude α; the exponent factor β; and the band edge γ. Our ellipsometer applies the Levenberg-Marquardt multivariate regression algorithm for data fitting whereby the optical constants and thickness are determined. Fig. 1 illustrates the results on a wafer map. The data set was taken from the center of the wafer in 10 mm increments.

After completing the a-Si film deposition and ellipsometry measurements, we diced the 4-inch wafer into 1 × 1-inch sample pieces using a Disco saw and cleaned them for further characterization. The samples were examined with X-ray diffraction (XRD) and scanning electron microscope (SEM) for base comparison prior to RTA heat treatment. The samples were subjected to RTA treatment with a matrix of different temperatures ranging from 600 °C to 980 °C for 10 min in an Argon ambient with a flow rate of 1000 SCCM. The RTA equipment (JetFirst-150 RTA by Jipelec) was used for this annealing process at 23% power. After RTA, each sample was characterized by a Siemens D-500 XRD system and a Woollam VAS ellipsometer to determine changes in structural properties and optical constants. The ellipsometry measurements were taken in reflection at angles of 65°, 70°, and 75° in the spectral range of 900 nm to 1700 nm. We measured the preferred crystallographic orientation of the films by XRD in reflection from 10° to 90° with a step size of 0.02° and a duration of 2 s at each step. The system uses Cu K-alpha radiation with a wavelength of 0.15418 nm. The XRD system is controlled by MDI datascan software and analyzed by JADE XRD pattern processing software. We operated the JEOL JSM 7600 SEM at 1 keV–2 keV with images at 80,000× zoom in LEI mode.

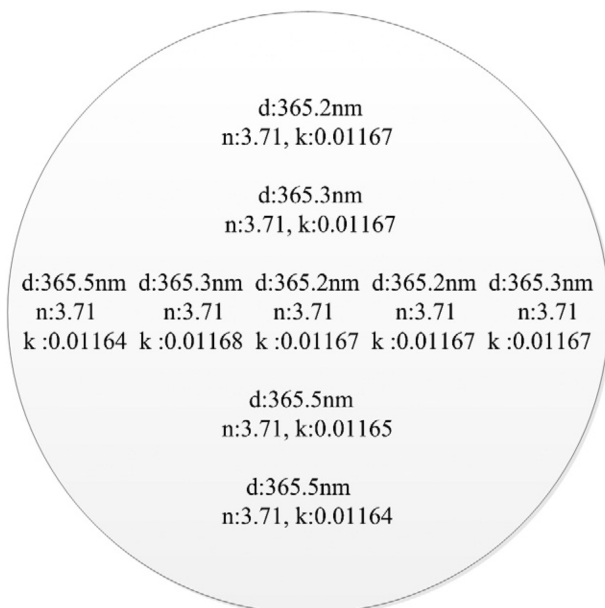

Fig. 1. Graphical presentation of uniformity of thickness d and values of n and k for an example a-Si film deposited on a 4-inch (100) Si wafer.

3. Results and discussion

To understand the effect of the annealing process on the a-Si in more detail, we examined the samples with SEM and ellipsometry for changes in surface roughness and for possible noticeable grain formation. The results for surface roughness are shown in Fig. 2. The surface roughness increases linearly with the annealing temperature of 600 °C to ~700 °C and saturates at higher temperatures. For the ellipsometry measurement, our model for the desired structure contains an effective medium approximation layer that simulates the surface roughness layer [17–19]; it has 50% air and 50% top-layer Si. SEM results for selected annealing temperatures are presented in Fig. 3.

The XRD results for the annealed samples are shown in Fig. 4. The data clearly show that the a-Si evolves to poly-Si as a function of temperature. Crystallization starts with a (211) peak at annealing temperature of 650 °C increasing with temperature up to 800 °C at which point the (211) orientation signature begins to decrease. This coincides with the emergence of (111), (220), and (311) orientations with (111) domination. This trend continues for higher annealing temperatures, up to the limit of our RTA equipment of 950 °C. The (211) peak completely disappears at 950 °C. On the other hand, the (111) peak intensity increases sharply while the intensities of the other two peaks, (220), and (311), increase slowly [14–16,20]. The preferred crystal growth in the (111) orientation at higher temperatures is due to the strong anisotropic growth rate of the grains, which acts as an orientation filter that in turn is due to growth competition of various possible orientations as explained in [10]. For the (111) orientation, we observe that the speed of growth is the highest after 800 °C. These results indicate that the faster growth of the (111) orientation at higher temperatures results in domination of the (111) orientation, which is due to the preferred free-energy minima of the film during heat treatment.

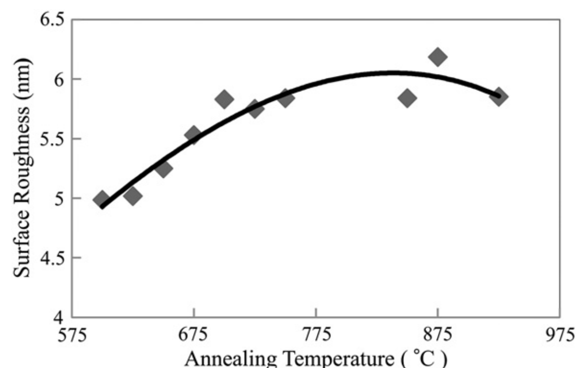

Fig. 2. Surface roughness data as a function of annealing temperature. The solid line acts as a visual aid.

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