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Effects of hydrogen surface pretreatment of silicon dioxide on the nucleation and surface roughness of polycrystalline silicon films prepared by rapid thermal chemical vapor deposition

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It is well known that Si surface treatment is crucial for low-temperature Si epitaxy. Although considerable work exists which is aimed at elucidating the effects of Si surface pretreatments on Si epitaxy, little is known about the effects of SiO_2 surface pretreatments for polycrystalline silicon (poly-Si) growth. We report on a study of SiO_2 surface pretreatment effects on poly-Si nucleation and film surface roughness using a low energy hydrogen ion beam (200 eV) and H_2 gas annealing (850 °C) in a rapid thermal chemical vapor deposition system. *In situ* real-time ellipsometry was used to monitor the surfaces during pretreatment and observe the nucleation. The microstructure and surface roughness of the deposited poly-Si films are determined by analysis of *in situ* spectroscopic ellipsometry (SE) and atomic force microscopy (AFM) measurements. Hydrogen ion beam pretreatment was found to produce higher nuclei density and a smoother poly-Si surface than nonpretreated substrates, and the opposite was found for hydrogen gas annealing giving lower nuclei density and rougher poly-Si. [S0003-6951(96)03330-X]

It is well known that Si surface treatment is crucial for low-temperature Si epitaxy (see for example, Refs. 1–3). However there are few reports on SiO_2 surface pretreatment for polycrystalline silicon (poly-Si) depositions. The use of poly-Si films in applications involving integrated circuit and thin-film transistor (TFT) technology for active matrix liquid crystal displays has received much attention.[4] For this application, glass substrates must be used with limit the temperature at which the silicon layers are formed to around 620 °C, the softening point of glass.[5] Smooth poly-Si films with large grain size are desirable, in order to achieve a high mobility (10–100 cm^2/V s) TFT.[6] It has been found that the grain size of recrystallized films formed from Si_2H_6 chemical vapor deposition (CVD) is larger than that formed from SiH_4.[7] Good quality poly-Si films have been obtained by deposition in the amorphous phase and subsequent crystallization via low-temperature annealing.[8–11]

In this letter, we report on the surface roughness and microstructure of poly-Si films using a low energy hydrogen ion beam to pretreat SiO_2 covered Si substrates before the CVD from Si_2H_6. *In situ* ellipsometry was used for real time observation of the surface change during pretreatment and nucleation. *In situ* spectroscopic ellipsometry (SE) and atomic force microscopy (AFM) were used to characterize the film roughness and microstructure. For comparison with the ion beam pretreatments, results of H_2 gas pretreatment at 2 Torr and 850 °C are also presented.

Surface pretreatments and poly-Si film depositions were performed *in situ* in a custom built rapid thermal processing system consisting of a water cooled, turbopumped vacuum chamber (base pressure of 10^{-8} Torr) with a rotating analyzer *in situ* ellipsometer that was described in greater detail elsewhere.[12,13] The samples are heated from the back side, and the ellipsometric measurements are made on the sample front side. The SE measurements were made with photon energies between 2.0 and 5.0 eV and at room temperature after cooling the sample. The real time single wavelength

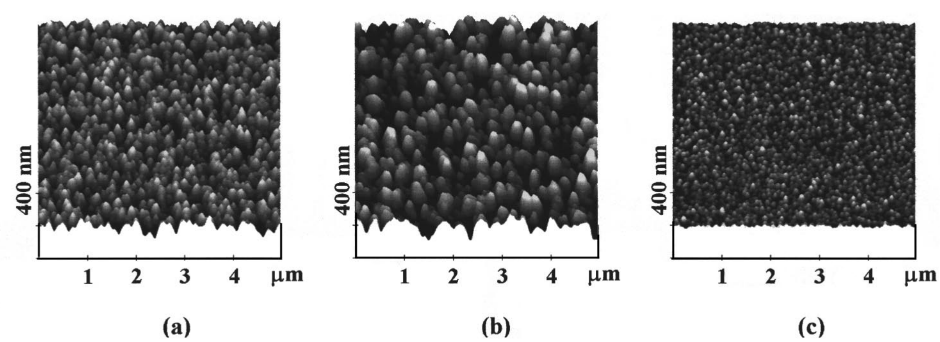

FIG. 1. AFM results of the poly-Si film surface deposited on *in situ* pretreated SiO_2 substrates using Si_2H_6 (5% in He) at 0.02 Torr and 650 °C. rms: root mean square, Z_{max}: maximum height, D_g: average grain size. (a) Nonpretreated sample, rms=22 nm, Z_{max}=140 nm, D_g=270 nm. (b) H_2 gas bake pretreated sample, rms=33 nm, Z_{max}=190 nm, D_g=440 nm. (c) H ion beam pretreated sample, rms=6 nm, Z_{max}=58 nm, D_g=180 n

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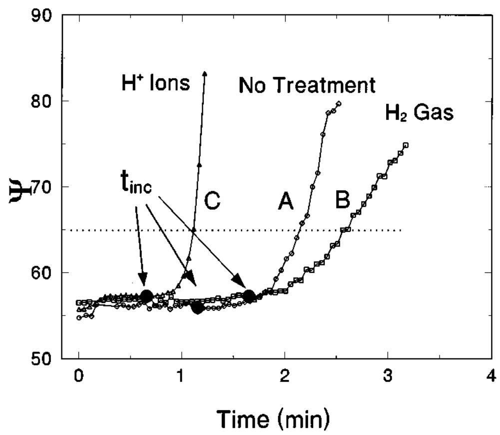

FIG. 2. Initial evolution of ψ as a function of time from real time single wavelength ellipsometry ($\lambda = 340$ nm) measurements at 650 °C and 0.02 Torr of Si_2H_6 (5% in He) for different surface pretreated samples.

ellipsometry (SWE) was carried out at 340 nm (3.65 eV), since this wavelength is relatively insensitive to temperature for Si.[14]

The samples were silicon wafers, with about 50 nm thick thermally grown SiO_2. The sample pretreatments used in this study consisted of a H_2 bake (2 Torr and 850 °C) and H^+ ion beam bombardment with low ion energies (100–1000 eV) at 650 °C for 5 min. Hydrogen ion beam pretreatment was performed using a 3 cm Kaufman ion beam source operated at 1–10 mA, with calibrated doses of about 10^{16}–10^{17} ions/cm^2 for 5 min. The ion beam was directed normal to the substrate. Substrates were heated using quartz-halogen lamps. A shutter located in front of the substrate is used during H^+ ion beam stabilization and during rapid thermal CVD (RTCVD). The poly-Si films were deposited on the pretreated samples using Si_2H_6 (5%, diluted with He) at a pressure of 0.02 Torr and 650 °C in the RTCVD system. Immediately after pretreatment the substrates were subjected to Si deposition by RTCVD using Si_2H_6 under the same conditions (temperature of 650 °C and pressure of 0.02 Torr for 5 min). The SE spectra were obtained at room temperature after cooling the samples, because of the unavailability of reliable optical data for all the components present at the higher temperatures. Aspnes[15] has shown that the interband peaks in the ϵ_2 spectrum at about 3.4 and 4.3 eV are particularly sensitive to the semiconductor surface condition, and that a maximum value for the peaks indicates the best surface. We found featureless ϵ_2 spectra from the poly-Si film deposited on the substrate pretreated by H_2 bake which is indicative of a rough surface while the film deposited on H^+ ion beam pretreated substrate shows the Si interband peaks indicating a higher quality and smoother film. The SE data were analyzed using the well-established linear regression technique employing the Bruggeman effective medium approximation.[16] Previously, we have found that the best-fit optical model for poly-Si film deposited on SiO_2 is a two layer model.[17] The top layer is a roughness layer and the bottom layer is the poly-Si film. The best-fit models reveal that the films are mixture of amorphous and crystalline phases, and that the films deposited from H_2 gas baked samples have more voids, less c-Si component and a much thicker roughness layer (47 ± 5 nm) than that formed from ion beam pretreated samples (3.7 ± 0.3 nm). A series of pretreatment experiments with different ion energies and ion doses have also been done and show that reducing the ion doses from 9.8×10^{16} to 2.4×10^{16} ions/cm^2 increases poly-Si roughness from 4 to 8 nm, and increasing ion energies from 200 to 1000 eV the roughness only changes by a fraction of a nm. In order to confirm the SE results, the film surfaces were examined using AFM. Figure 1 displays AFM images with the root mean square (rms) and maximum roughness values of the surface roughness. The trends of the values are similar to the results from SE models.

In order to understand the effect of the surface pretreatments on the deposited poly-Si film structures, we monitored both the surface changes with a real-time SWE during pretreatment, and then with SE after pretreatments. Real-time SWE at 340 nm did not yield observable changes in ψ and Δ for both the 5 min H_2 gas and H^+ ion beam pretreatments. *In situ* SE results before and after the pretreatments show only small changes. AFM revealed no changes after the substrate pretreatments. We have found from our previous thin-film nucleation studies[11,12,17,18] that nucleation is very sensitive to the substrate surface conditions. Therefore, a series of nucleation experiments after *in situ* surface pretreatments were performed. The ψ-time curves in Fig. 2 show that the time prior to observable nucleation, the incubation time (t_{inc}), for

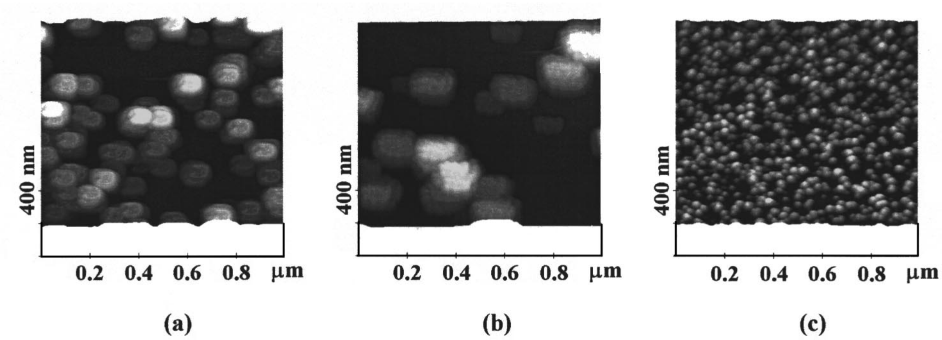

FIG. 3. AFM images of initial nucleation stage of RTCVD Si nuclei on different pretreated SiO_2 surfaces. In order to make a good comparison the CVD were stopped at the certain time corresponding to $\psi = 65°$ for all samples from Fig. 2. t: deposition time, n: nuclei density, r: mean nuclei radius, h: average nuclei height. (a) Nonpretreated sample, $t = 2$ min, $n = 4.2 \times 10^9$ cm^{-2}, $r = 67$ nm, $h = 20$ nm. (b) H_2 gas pretreated sample, $t = 2.5$ min, $n = 1.5 \times 10^9$ cm^{-2}, $r = 91$ nm, $h = 33$ nm. (c) H ion beam pretreated sample, $t = 1$ min, $n = 7.0 \times 10^{10}$ cm^{-2}, $r = 12$ nm, $h = 9.3$ nm.

TABLE I. A comparison of effect of different SiO_2 surface *in situ* pretreatments on the nucleation and poly-Si film surface roughness using RTCVD from Si_2H_6.

Parameters of film and nucleation	Non-pretreatment	H_2 gas annealing pretreatment	H^+ ion beam pretreatment
Incubation time (min)	1.2	1.7	0.7
Nucleation point	A	B	C
Nuclei density (cm^{-2})	4.2×10^9	1.5×10^9	7.0×10^{10}
Nuclei radius (nm)	67	91	12
Nuclei height (nm)	20	33	9.3
Film roughness (nm)			
SE (model)	22	47	4
AFM (rms)	22	33	6
Grain size (nm)	270	440	180

the H^+ pretreated substrate is the shortest at 0.7 min, compared to 1.7 min for H_2 gas treatment and 1.2 min for no treatment. We stopped poly-Si CVD processes at about $\psi=65°$ (see dashed line in Fig. 2) from ellipsometric monitoring for AFM observations shown in Fig. 3. Nucleation parameters for these samples, such as nuclei density n, mean nuclei height, h, and average nuclei radium r (or average nuclei area, where $A=\pi r^2$) are summarized in Table I. Relative to the nonpretreated sample there is at least a two-fold decrease in nuclei density for H_2 gas pretreatment, and a factor of ten increase for H^+ ion beam pretreatment. Also, Table I shows that the surface roughness and grain size of the poly-Si films deposited on SiO_2 are dependent on the nucleation parameters and that films with smooth surfaces are obtained from a high nuclei density. It is well known[19] that during Si nucleation on the SiO_2 surface, two mechanisms contribute to the Si nuclei growth: direct impingement of atoms from the gas phase on the growing nuclei, and lateral surface diffusion of adsorbed atoms and attachment to the nuclei. For the high nuclei density the nuclei coalescence occurs faster due to the smaller average separation distance, and thus a flat shape (disk) dominates the morphology of the nuclei layer. The difference of pretreatment mechanism between H_2 gas annealing and H^+ is not completely understood. However, we believe from the SE and AFM results (i.e., the inability to observe significant changes) that both pretreatments before deposition only strongly influence the top surfaces.

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