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In situ real time measurement of the incubation time for silicon nucleation on silicon dioxide in a rapid thermal process

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Real time ellipsometry and atomic force microscopy (AFM) were used to measure critical nucleation parameters for polycrystalline silicon deposition on an amorphous SiO_2 layer by rapid thermal chemical vapor deposition (RTCVD) using disilane (5% in helium). A particularly important parameter for selective epitaxial deposition is the time for nuclei to form, the incubation time. Quantitation of the nucleation parameters, such as the nuclei density, nuclei growth rate, nuclei coalescence, and an operational incubation time were determined from the real time ellipsometric measurements and confirmed by AFM. For a substrate temperature of 700 °C and at a chamber pressure of 0.2 Torr, the nuclei densities of 1.4×10^{10} nuclei/cm^2, incubation time of 26 s and nuclei layer growth rates of 20 nm/min were obtained. © *1995 American Institute of Physics.*

Interest exists for the selective epitaxial growth (SEG) of silicon, because this process offers a number of advantages,[1,2] such as a via hole filling method for planarization and a diffusion microsource for attaining accurate dopant profiles. The conventional approach in chemical vapor deposition (CVD) processes to obtain selective deposition is the use of dichlorosilane with H_2 and HCl.[3] However, the employment of ultrahigh vacuum chemical vapor deposition (UHV\CVD) systems restricts the use of chlorine-containing Si source compounds in favor of silane or disilane (Si_2H_6) as the reactants with H_2.[4] The use of Si_2H_6 in Si selective epitaxy by CVD is motivated by[4] low process temperatures and high growth rate (ten times higher than that of the SiH_4 process[5]). The effectiveness of SEG is determined largely by the nucleation of Si on SiO_2. The key parameters that characterize the nucleation process such as the time for stable nuclei formation, the incubation time, the coalescence point, and the nuclei saturation density are difficult to measure, and a technique that allows real time monitoring of these parameters is valuable for process development. The conventional *in situ* surface probes cannot be used outside the ultrahigh vacuum environment (i.e., at process pressures) or in the highly reactive and high-temperature ambient of a CVD reactor. However, ellipsometry has been shown to be capable of making measurements of nucleation, with monolayer sensitivity and quantification of microstructure.[6–13]

In this work, we investigated the initial rapid thermal CVD, RTCVD, nucleation, and growth regime for Si on SiO_2 film covered Si, from disilane using real time ellipsometry and atomic force microscopy (AFM). The results are presented in terms of an operational incubation time, the coalescence point, nuclei density, size, and growth rate. The RTCVD system operational incubation time (hereafter called the incubation time) is measured from the turn on of the heating system. An extrapolation method described later is used to obtain the onset of nucleation. This incubation time includes the system heat-up time as was done by others[14] for Si RTCVD. Thus, the incubation times we report are longer than the time to form a critical nucleus.

The custom built rapid thermal processing (RTP) system consisting of a vacuum system and an ellipsometer was described in greater detail elsewhere.[15] Essentially, the vacuum system consists of a load lock and a turbopumped, water-cooled stainless-steel RTCVD chamber with a base pressure of 10^{-8} Torr. A Baratron and a quadrupole mass spectrometer are used to monitor the total and partial pressures, respectively. The ellipsometer is also a custom built rotating analyzer ellipsometer similar to the system described by Aspnes.[16] The ellipsometry measurements are made in an inverted configuration (i.e., measured from below the sample). This configuration is advantageous for RTP, since with the wafer inverted and heated from above, the wafer blocks much of the direct radiation from the lamps. The *in situ* spectroscopic ellipsometry (SE) measurements were made with photon energies between 2.5 and 5.0 eV and at room temperature after cooling the sample. The real time single wavelength ellipsometry (SWE) was carried out at 288 nm (4.3 eV), since this wavelength is near a Si interband absorption and has been found to be sensitive[17–19] to Si nucleation. All data are expressed as trajectories in the Δ, Ψ ellipsometric measurement plane. In modeling Si nucleation, the measured ellipsometric Δ, Ψ data are compared to the calculated values as obtained from models to be discussed below. The nuclei are assumed to grow in a hexagonal network. Upon coalescence of the nuclei, voids may form at the interface, due to incomplete coalescence and/or on top of the film to represent surface roughness.

Nucleation experiments were performed on SiO_2 (thermally oxidized in dry O_2 to 24–28 nm thick) covered single-crystal Si (*c*-Si) wafers [*p*-type, (100) oriented]. A mixture of 5% Si_2H_6 in He was used. A low substrate temperature of 700 °C was chosen, since low-temperature selectivity is a current issue in RTCVD processes.

In order to study the initial stage of Si deposition on SiO_2 covered Si, we carried out a series of depositions at different times at constant substrate temperature after ramp up, gas pressure, and flow rate. In principle, the deposition of a new layer on an optically distinct substrate induces a

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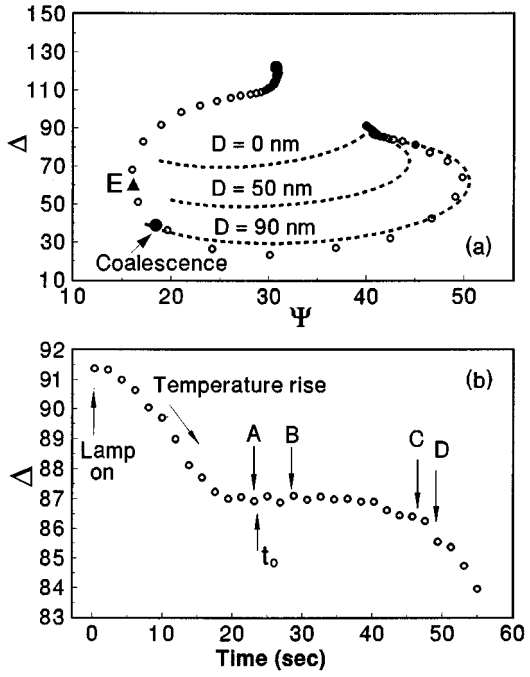

FIG. 1. (a) Δ, Ψ trajectories from real time single wavelength ellipsometry (λ=288 nm) measurement (symbols) at 700 °C and 0.2 Torr of Si_2H_6 (5% in He) along with simulations (dashed lines) assuming a spherical segment nuclei shape model (growth rate ratio: $K=0.35$). (b) Initial evolution of Δ as a function of time.

movement of the Δ, Ψ point as the layer thickens. It is known[6] that the growth interface between deposited polycrystalline silicon (poly-Si) and SiO_2 becomes rough with the deposition temperatures higher than about 615 °C. When the roughness is much smaller than the wavelength of light, an effective medium approximation (EMA) has been found to give an adequate description of the rough layer optical properties.[16] The Bruggeman EMA with isotropic screening[17] was used to calculate the effective dielectric function of the layer (at each thickness) from the volume fractions of the two components, *c*-Si and voids.

Figure 1(a) shows the Δ, Ψ trajectories measured by real time ellipsometry for a Si film deposited onto a SiO_2 covered Si substrate from Si_2H_6 (5% in He) at 700 °C. The data deviate significantly from the layer-by-layer film growth model ($D=0$ nm, dashed line). The optical model used for simulations of Si nucleation on amorphous oxide consists of a hexagonal network of spherically shaped nuclei having the bulk Si—Si bond density with an average distance D between nuclei, the radius of the nuclei $r(t)$ increases with time[10,20] up to the point where the nuclei come into contact, i.e., coalescence. AFM shows the nuclei geometry to be spherically shaped but smaller than a hemisphere, and based on these observations the model used for this study is also shown in Fig. 1(a). In this model, the growth ratio K, which is defined as the ratio of the growth rate in the vertical (h) to the lateral growth (r) direction, is 0.35 as obtained as an average from the AFM observations ($K=0$ and 1 correspond to layer-by-layer and hemispherical models, respectively). From this model, the Si volume fraction is given as: $f_{Si}=\pi h^2(3+K^3)/3\cdot 3^{1/2}K^2D^2$, where h is the thickness of the nucleation layer. Modeling requires a prior knowledge of the dielectric function of the Si nuclei which is approximated from spectroscopic ellipsometry measurements of a thick deposited poly-Si film. Using this model, the Δ, Ψ trajectory for the progressive growth of the Si nuclei on the SiO_2 covered Si substrate can be calculated. Some modeling results are plotted in Fig. 1(a) for mean distances between nuclei (D) of 0, 50, and 90 nm (dashed lines). The data correspond closely to the $D=90$ nm which yields a nuclei density of 1.4×10^{10} nuclei/cm^2, which as shown below is close to AFM values. It should be mentioned that assuming purely hemispherical nuclei, i.e., without the benefit of AFM measurements, we obtain N_n of 9.6×10^{10} nuclei/cm^2 or a factor of 6 larger.

Once critical Si nuclei form, their size increases with time whereas the density remains constant up to coalescence. This assumption has been justified by Claassen *et al.*[21] at higher growth temperatures using scanning electron microscopy to analyze the nucleation step. We believe that we do not observe the critical nuclei, because within a few seconds of the appearance of nuclei, the saturation density is reached. We can then consider a schematic model of the nucleation process where the first step is the formation of critical nuclei separated by a distance D, and in order to render the calculation simple, the nuclei are assumed to be distributed on a hexagonal network. The mean nuclei density which is readily calculated from the nuclei distance D is 1.4×10^{10} nuclei/cm^2. The incubation time is one of the important parameters for Si selective epitaxy and it is difficult and tedious to measure using *ex situ* observations. However, real time ellipsometry monitoring of Δ versus time as is shown in Fig. 1(b) provides an efficient method to measure this time. From Fig. 1(b) the minimum change of Δ which can be observed is about 0.5°. From simulations of an optical model for the caplike shape ($K=0.35$) at a wavelength of 288 nm this value corresponds to about 1, 3, or 5 nm nuclei layer thicknesses for the distances between nuclei of 10, 50, or 90 nm, respectively. This means that by ellipsometry we could not detect a change of up to 5 nm nuclei layer thickness for a 90 nm nuclei separation. The AFM sensitivity is (conservatively) less than 1 nm vertically and 2–3 nm horizontally. The nearly linear evolution of the nuclei layer thickness with deposition time, obtained from the experimental ellipsometric trajectory and the best-fit optical model is shown in Fig. 2(a). Assuming a linear relationship from the beginning of the deposition, we can extend the line to the time axis and obtain t_0 corresponding to the zero thickness, in order to obtain the incubation time which for our present purposes is from the start of the lamp power which includes warm up and obtain $t_0=26$ s. This extrapolation somewhat alleviates the problem of the sensitivity of ellipsometry to small sparse nuclei.

Observation of the nuclei layer morphology by atomic force microscopy at progressive time steps in the nucleation process gives parameters, such as nuclei density N_n average nuclei height, h_n, and mean nuclei area A_n (where $A_n=\pi r_n^2$). Figure 3(a) is the AFM image corresponding to the point A in Fig. 1(b) at $t=25$ s (less than the incubation time t_0), and it shows no nuclei. Figure 3(b), corresponding to the point B in Fig. 1(b) at $t=31$ s (5 s after t_0) which shows no change in Δ

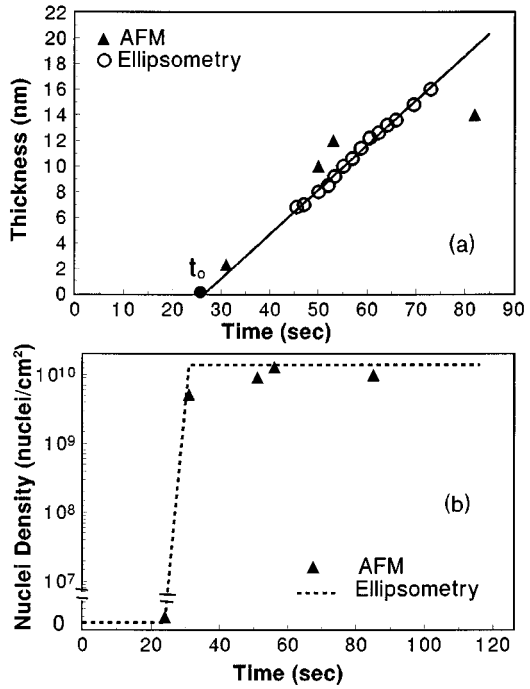

FIG. 2. (a) Initial nuclei layer thickness as a function of time from real time ellipsometry and comparisons with AFM. (b) Comparison of nuclei density from real time ellipsometry and AFM.

from the ellipsometry measurement, gives $N_n=5.2\times10^9$ nuclei/cm^2, $h_n=2.3$ nm and $A_n=7.2\times10^2$ nm^2. The smallest nucleus observed in Fig. 3(b) is about 2 nm in height and 15 nm base diameter. This size is much larger than that expected[22] for a critical nuclei in CVD which often consists of only a few atoms. This may imply that there is rapid lateral growth of nuclei from the critical nuclei size by lateral attachment to the nuclei from surface diffusion. Figures 3(c) and 3(d) are the AFM images corresponding to the points of C(50 s) and D(53 s) in Fig. 1(b) which yield nuclei densities of 9.2 and 13.0×10^9 nuclei/cm^2, respectively. The average heights are about 10 and 12 nm and mean nuclei areas are 2.7×10^3 nm^2 and 2.9×10^3 nm^2 ($r=30$ nm), from which we have deduced the average nuclei geometry to be a spherical segment or cap with $K=0.35$. Figure 3(e), corresponds to the point E(82 s) in Fig. 1(a) which was about 4 s after nuclei coalescence as was calculated from the optical model shown in Fig. 1(a). The mean nuclei area of 5.9×10^3 nm^2 ($r=43$ nm) is very close to the ellipsometry result of 45 nm. The good comparison of nuclei density and average nuclei height as a function of time from AFM and ellipsometry optical modeling are shown in Figs. 2(a) and 2(b).

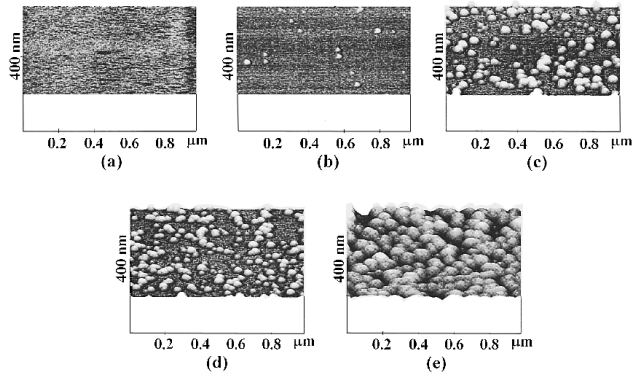

FIG. 3. AFM image of initial nucleation stage of rapid thermal CVD Si nuclei on SiO_2 surface (a), (b), (c), (d), and (e) corresponding to A, B, C, D in Fig. 1(b) and E in Fig. 1(a). (a) $t=25$ s, none; (b) $t=31$ s, $N_n=5.2\times10^9$ nuclei/cm^2, $h_n=2.3$ nm, $A_n=7.2\times10^2$ nm^2; (c) $t=50$ s, $N_n=9.2\times10^9$ nuclei/cm^2, $h_n=10$ nm, $A_n=2.7\times10^3$ nm^2; (d) $t=53$ s, $N_n=1.3\times10^{10}$ nuclei/cm^2, $h_n=12$ nm, $A_n=2.9\times10^3$ nm^2; (e) $t=82$ s, $N_n=1.0\times10^{10}$ nuclei/cm^2, $h_n=14$ nm, $A_n=5.9\times10^3$ nm^2.

In conclusion, we have shown that *in situ* real time ellipsometry can be used to characterize the nucleation of Si on SiO_2 in a RTCVD system using Si_2H_6 (5% in He). With the use of AFM we have been able to measure several nucleation parameters which are then used in the Bruggeman effective medium approximation for optical data analysis. The main nucleation parameters such as: prenucleation incubation time, saturation nuclei density, mean nuclei size, and nuclei layer growth rate are obtained. The best-fit model for the poly-Si nucleation on SiO_2 at 700 °C and 0.2 Torr is a cap-shaped optical model with ratio of $K=0.35$ and nuclei distance of about 90 nm. From this model a incubation time of 26 s, nuclei layer growth rate of 20 nm/min, coalescence nuclei size of 45 nm, and nuclei coalescence were obtained.

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