

LOW TEMPERATURE PHOTON-CONTROLLED GROWTH OF THIN FILMS AND MULTILAYERED STRUCTURES

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Pulsed ArF (193 nm) excimer laser photolysis of disilane, germane, and disilane–ammonia mixtures has been used to deposit amorphous superlattices containing silicon, germanium and silicon nitride layers. Transmission electron microscope cross-section views demonstrate that structures having thin (5–25 nm) layers and sharp interlayer boundaries can be deposited entirely under laser photolytic control, using low reactant gas partial pressures and with the excimer laser beam parallel to the substrate. Growth of epitaxial films and structures under similar conditions is discussed.

1. Introduction

Low-temperature thin film deposition methods are needed because thermal equilibrium processes of defect generation and diffusion degrade the properties of many crystalline and amorphous materials, when they are grown at the temperatures needed for conventional pyrolytic (thermally driven) chemical vapor deposition (CVD) reactions. For example, when multilayered semiconductor structures contain highly doped adjacent layers, low temperatures are required to prevent dopant interdiffusion during growth. Low deposition temperatures also minimize in-diffusion of unwanted impurities from the surroundings and film–substrate interdiffusion. For crystalline films and superlattices (e.g., using II–VI compounds) lower growth temperatures are needed to minimize vacancy generation and interdiffusion that prevent the formation of high-quality crystalline layers with abrupt compositional changes and well-defined interfaces [1–3]. Low growth temperatures also provide unique access to crystalline metastable phases that can be stabilized by heteroepitaxial growth (pseudomorphism) [4–6]. However, conventional pyrolytic (thermally driven) CVD reactions usually are limited to very low film growth rates at low temperatures.

Photon-controlled dissociation of parent molecules is an attractive alternative to pyrolytic bond-breaking for low temperature film growth, because it

allows the film deposition rate to be controlled independent of the substrate temperature. By decoupling the bond-breaking process from substrate temperature, it becomes possible to choose the substrate temperature in order to obtain the best crystalline quality, or to optimize the electrical or optical properties of an amorphous film. In this paper we present results of recent experiments in which ArF (193 nm) excimer laser photolysis of parent gas molecules was used for deposition of hydrogenated amorphous silicon (a-Si:H), hydrogenated amorphous germanium (a-Ge:H) and Si_3N_4 , and to fabricate a-Si:H/a-Ge:H and a-Si:H/ Si_3N_4 superlattice structures.

In the course of this work we found that pulsed laser photolysis has several advantages for low temperature fabrication of amorphous superlattices: (1) High resolution can be obtained in layer thickness because film deposition is inherently "digital", with a single moderate-energy laser pulse increasing the average film thickness by only a fraction of a monolayer. (2) Nevertheless, high deposition rates that are nearly independent of substrate temperature [7-9] still can be achieved at low temperatures, because of the high pulse repetition rate and variable pulse energy of excimer lasers. Consequently, (3) the substrate temperature can be chosen to minimize impurity and/or dopant diffusion, resulting in well-defined layer boundaries, or to optimize electrical properties (e.g., via the temperature-dependence of the H content [10] in hydrogenated amorphous semiconductor layers). (However, at low temperatures we find there is a limiting deposition rate above which film properties and composition - e.g., of a-Si:H - depend sensitively on the deposition rate as well as the substrate temperature [8]. Studies of systematic variations in the optical properties of our a-Si:H films as a function of deposition conditions are the subject of a separate paper [8].) We note that localized Ar^+ ion laser heating was used recently [11] to thermally "crack" PH_3 , resulting in deposition of GaAsP layers on top of conventionally grown (pyrolytic) GaAs layers. However, the experiments reported here appear to be the first in which superlattice structures have been fabricated entirely under photolytic control.

2. Experimental

Superlattice depositions were carried out in a turbo-pumped chamber (base pressure $\sim 10^{-6}$ Torr) based on a six-way stainless steel cross and equipped with 5 cm diameter Suprasil windows. The ArF laser beam (~ 15 ns full-width half-maximum pulse duration) was unfocused but was passed through a rectangular slit before entering the chamber, to more precisely define its cross section (6×20 mm²). The excimer beam traveled parallel to the substrate with its lower edge set 1 mm above the substrate surface. Depositions were made onto 2.5 cm square (100) crystalline (c) Si substrates, whose temperature was monitored with an infrared radiation thermometer (IRCON Type W). A high

stability HeNe (632.8 nm) laser beam was reflected at near-normal incidence from the gas–film and film–substrate interfaces and the interference oscillations in the reflected intensity as the film was deposited were used for precise in situ film thickness monitoring. (Very similar techniques were used earlier by us and by others in studies of transient melting and solidification [12,13] and of film deposition [7,14,15] on the nanosecond and longer times scales.) The background noise level in our reflectivity measurements corresponded to $<10^{12}$ atoms deposited within the HeNe laser's ~ 0.5 mm unfocused spot diameter, so that deposition rates of <0.01 nm/min could be measured. Helium was used to flush the inside of the two windows through which the excimer laser beam entered and exited the chamber. It was found that a He flow in the range of 150–250 sccm per window (see below) would nearly eliminate deposition of Si or Si_3N_4 films on the windows; this otherwise results in a rapid decrease of laser power and prevents accurate monitoring of the laser power within the chamber. The He flush did not prevent a very gradual decay of laser power within the chamber due to Ge film buildup on the windows during photolysis of germane (GeH_4). However, the rate of Ge window-film buildup was found to depend strongly on the incident laser fluence and could be forestalled by operating the excimer laser at a relatively low fluence.

2.1. Laser photochemical deposition reactions

Previous work has established that single layers of either amorphous semiconductor (a-Si [7,16–18], a-Ge [9,19]) or dielectric (SiO_2 [20,21], Si_3N_4 [20–24]) materials can be deposited photolytically, and that deposition conditions can be found to produce films having properties that are similar to those obtained using other CVD-based techniques. In our experiments, hydrogenated amorphous Si was deposited by photolysis of disilane (Si_2H_6); Si_3N_4 was deposited by photolysis of mixtures of disilane and ammonia (NH_3); and hydrogenated a-Ge was deposited by photolysis of germane. However, there appear to have been no definitive studies published of the photochemical reaction paths that lead to film deposition.

Photolytic deposition of a-Ge:H from GeH_4 relies upon a very small ($\sim 3 \times 10^{-20}$ cm²) absorption cross section at 193 nm [25,26]. Consequently, structures containing relatively thin (<10 nm) a-Ge:H layers can be grown entirely by photolysis, but it is necessary to grow thicker layers by pyrolysis, in order to obtain reasonable growth times and to avoid eventual loss of laser power due to Ge film deposition on the windows. However, reasonable pyrolytic growth rates then may require working at higher temperatures. Photolysis of germane probably produces germylene (GeH_2) at the surface, and growth of hydrogenated a-Ge may involve a series of GeH_2 insertion reactions followed by desorption of H_2 [27]. In addition to GeH_2 , photolysis

of germane produces other intermediates such as GeH, GeH₃, Ge₂H₆ and atomic Ge [9], but there is not sufficient experimental data to determine the primary precursor for film deposition [27].

The chemical reaction paths resulting in deposition of a-Si:H from disilane pyrolysis and by 193 nm photolysis are discussed in other papers [7,8]. The 193 nm absorption cross section ($\sim 3 \times 10^{-18}$ cm²) for disilane is about 100 times larger than for germane [25]. Photolysis of disilane is believed to result in SiH₃SiH and H₂ as primary photoproducts, while secondary homogeneous and heterogeneous reactions involve intermediates such as SiH, SiH₃, and Si₂H₆, which have for their final stage (SiH₂)_n deposition on the surface, where $n = 1$ or 2. Above 200 °C, H₂ desorbs from the surface.

The reaction chemistry resulting in growth of Si₃N₄ by photolysis of ammonia and disilane mixtures is even more complex, since both constituents are strongly photodissociated at 193 nm. Ammonia by itself photodissociates ($\sigma_{193} = 1.7 \times 10^{-17}$ cm²) [28] predominantly to NH₂(\tilde{X}^2B_1) + H, with only about 2.5% going to NH₂(\tilde{A}^2A_1) [29]. The excited NH₂(\tilde{A}^2A_1) state can absorb another photon, dissociating to NH(A³Π) + H, but this is energetically impossible for the majority of NH₂ in the (\tilde{X}^2B_1) state [29]. Considering the variety of intermediates formed during photolysis of disilane in the absence of ammonia, the possible intermediates formed by their joint photodissociation are numerous and have not been determined. However, studies of Hg-sensitized reactions of SiH₄/NH₃ mixtures showed that silylamine (SiH₃NH₂), disilazane [(SiH₃)₂NH] and disilaneamine (Si₂H₅NH₂) were present [30]. These intermediates are believed to result in polymeric solids, with successive SiH₂ insertions producing films with higher Si and N content. These solids are known to transform to Si₃N₄ under prolonged heating.

2.2. Monitoring and controlling multilayer growth

Fig. 1 illustrates that essentially complete “photonic” control of deposition can be obtained at low temperatures, using the combination of laser photolysis and in situ reflectivity monitoring. Deposition ceases when the laser is turned off and quite small film thickness steps can be introduced by switching the laser on and off. Typical a-Si:H deposition rates were 0.15–0.3 nm/s using 40 mJ laser pulses at 40 Hz. At a mean deposition rate of ~ 0.2 nm/s and a laser repetition rate of 40 Hz, each laser pulse deposits only a fraction of a monolayer. At 350 °C and under our deposition conditions, the ratio of photolytic to pyrolytic deposition rates of a-Si:H from disilane is $\sim 10^3$, so that “background” thermal deposition is negligible.

Fig. 1 suggests two methods for in situ control of layer thickness in superlattice structures: (1) “open loop” control, in which the average laser power transmitted into the deposition chamber and the number of laser pulses

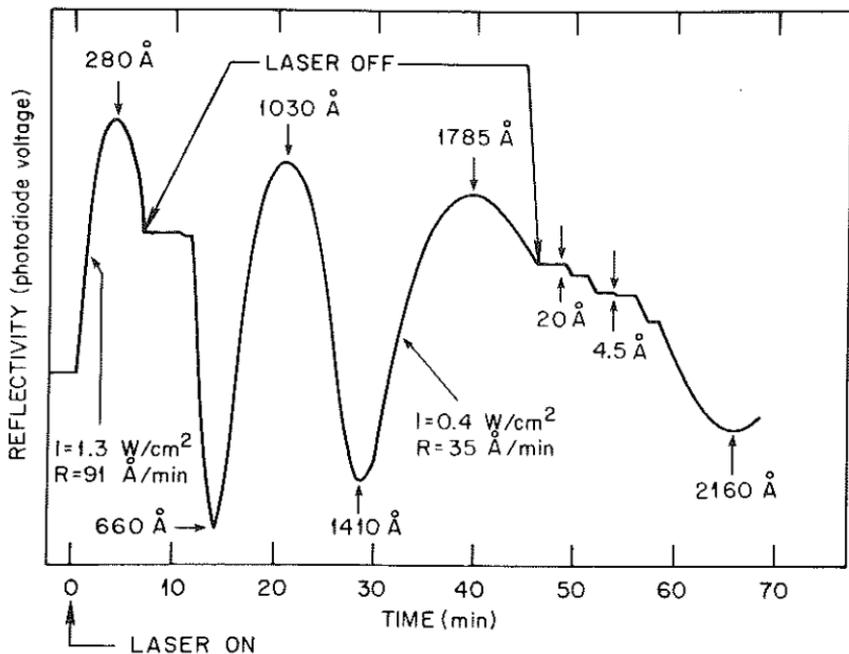


Fig. 1. Reflectivity signal obtained during photon-controlled deposition of a-Si on an oxidized c-Si substrate by ArF laser photolysis of disilane.

are monitored, and corresponding superlattice layers are deposited with the product of (power) \times (number of pulses) kept constant; and (2) "closed loop" control, in which the actual film thickness is monitored in real time and used to determine the end point for each layer. Open loop control requires that the deposition rate for each material be known in advance, in order to deposit layers of predetermined thickness. Its precision then depends upon accurate measurements of the excimer laser power and deposition time, and upon all other parameters, such as gas flows and pressure, remaining constant. Closed loop ("feedback") control requires accurate in situ monitoring of the actual film thickness. For this to be possible, the optical constants of the various layers must be known under actual deposition conditions, so that the reflectivity signal that will be obtained during deposition of a superlattice structure can be calculated. In this case, it also must be assumed that interlayer interactions (diffusion, chemical reactions, phase transformations) either do not occur during deposition or that they occur in ways that can be modeled. Thus, low substrate temperatures also have obvious advantages for optical feedback control.

3. Results

Our completely photolytic a-Ge:H/a-Si:H structures were deposited at 250 °C. The a-Ge:H layers used 140 sccm of a 10% germane/90% He mix at $p = 50$ Torr, 60 Hz laser repetition rate and 420 mW transmitted power. The a-Si:H layers used 40 sccm of a 10% disilane/90% He mix at $p = 5$ Torr, 40 Hz laser repetition rate and 200 mW of transmitted power. A 450 sccm (total) He window purge was used continuously. Fig. 2 shows a photolytically grown nine-layer a-Si:H/a-Ge:H structure, for which layer thickness was controlled by keeping the product of laser power and deposition time a constant for corresponding layers. The average thickness of the a-Si:H layers is 10.7 ± 0.4 nm, while for the first, third and fourth a-Ge:H layers the average thickness is 5.4 ± 0.2 nm. The second a-Ge:H layer was deposited with the power-time product increased by approximately 24%, resulting in a layer 30% thicker (7.0 ± 0.3 nm) than the average of the other layers.

Fig. 3 shows a 626 nm thick 32-layer (16-period) Si_3N_4 /a-Si:H superlattice that was deposited using ArF laser photolysis at 350 °C and $p = 5$ Torr. For the a-Si:H layers a 10% disilane/90% He mix was used at 20 sccm; for the Si_3N_4 layers, 60 sccm of NH_3 and 20 sccm of the disilane mix (30:1 NH_3 : Si_2H_6 ratio) were used. After the first layer, the a-Si:H layer thicknesses are 13.3 ± 0.4 nm, while the Si_3N_4 layer thicknesses are 26.6 ± 2 nm, as determined by TEM cross section views.

The results seen in figs. 2 and 3 demonstrate that experimental conditions exist (as described above) under which good control of layer thickness and sharp transitions between layers can be achieved using photolytic reaction chemistry, i.e., with no appreciable pyrolytic deposition. However, with in-

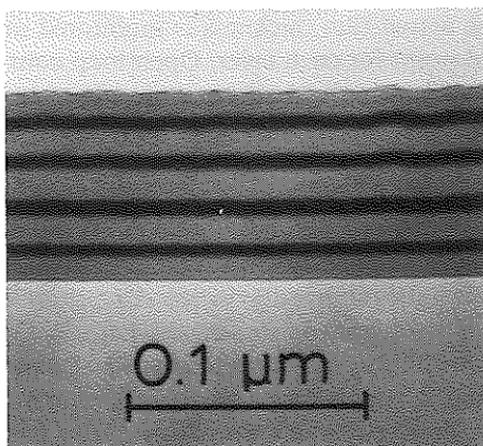


Fig. 2. TEM cross section view of a nine-layer a-Si(light)/a-Ge(dark) structure deposited on (100) c-Si by ArF laser photolysis at 250 °C.

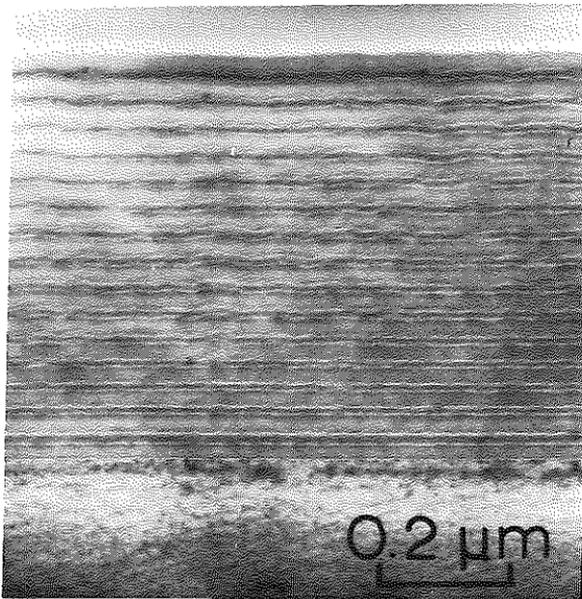


Fig. 3. TEM cross section view of a 32-layer Si₃N₄/a-Si amorphous superlattice deposited at 350 °C by ArF laser photolysis. The Si/Si₃N₄ interfaces show strong Fresnel fringe contrast.

creasing layer number occasional small (≥ 10 nm) lateral ripples were observed (fig. 3); because the layers are highly conformal, these ripples can propagate vertically into still higher layers.

During this work we also explored the feasibility of controlling amorphous superlattice deposition by monitoring the 632.8 nm reflectivity signal and comparing it in real time with pre-deposition model calculations of the reflectivity of the growing superlattice structure. The multilayer calculations were carried out using standard techniques for calculating the ellipsometry parameters Ψ and Δ for multilayered structures and graded interfaces [31]. The experimental and calculated reflectivities were found to be in good semiquantitative agreement, i.e., the relative heights of successive reflectivity peaks and valleys, in the relative complex reflectivity “signature” of a thin, growing, multilayered structure, were almost always in the calculated relationship. Post-deposition reflectivity calculations showed that the differences that did occur between the calculated and actual reflectivities for the a-Si:H/a-Ge:H structure were most likely due to small differences in the assumed and actual n and k values for the layers, i.e., the n and k values are sensitive to deposition conditions. However, the reflectivity model calculations for the Si₃N₄/a-Si:H structure were based on measured n and k values for single-layer films that had been deposited earlier, at substrate temperatures only slightly different than the 350 °C deposition temperature used for this

superlattice. Thus, we cannot rule out the possibility that at least part of the difference between the calculated and experimental superlattice reflectivity signals at 350°C was due to interactions (chemical reaction, interdiffusion) across the interfaces between layers during deposition.

4. Discussion

Most of our photon-controlled film deposition experiments were carried out at low reactive gas partial pressures (typically 40–500 mTorr), at low substrate temperatures ($\sim 250^\circ\text{C}$) and with the excimer laser beam parallel to the substrate so that only photolytic reactions were present. An intriguing question is whether epitaxial films can be grown under similar conditions, i.e., with minimal use of pyrolytic reactions of substrate heating.

Several groups recently have used normally incident pulsed laser radiation to grow epitaxial Ge [32], GaAs [33–35], and InP [36] films and to dope GaAs [37]. However, in most of these experiments, it was apparent that both photolytic and pyrolytic (surface heating) reactions were present, with pyrolysis probably dominating in some cases [33,37]. For GaAs and InP it was found that transient surface heating by a normally incident laser pulse was essential in order to obtain an epitaxially oriented film and to avoid serious ($\sim 2\%$) carbon contamination [35], which apparently results from secondary ArF laser (193 nm) photolysis of carbon-bearing ligands on metalorganic molecules. Normally incident laser fluences $\sim 0.1\text{ J/cm}^2$ were sufficient to reduce carbon contamination to $< 0.1\%$ and to obtain epitaxy, but interference between the normally incident laser beam and scattered surface waves resulted in periodic twin growth within the epitaxial film, with a spatial period equal to the laser wavelength [35]. An increase of the laser fluence to 0.2 J/cm^2 resulted in surface melting and decomposition (by As or P loss) [36]. When only photolytic reactions were used, by keeping the laser radiation parallel to the film surface, only amorphous or polycrystalline III–V compound films have been obtained.

However, quite different results were obtained recently by Zinck et al., who used KrF (248 nm) excimer laser photolysis of metalorganic molecules (dimethylcadmium and diethyltellurium) with the laser beam parallel to the substrate, for successful photoepitaxial growth of CdTe(111) on GaAs(100) at 165°C [38]. Growth rates up to $2\text{ }\mu\text{m/h}$ were obtained at low reactant partial pressures ($\sim 1\text{ mTorr}$). Auger analysis showed that the films were stoichiometric throughout, and that carbon and oxygen contamination was below Auger detection limits (0.1%). It appears that by using longer wavelength (248 nm) laser radiation, Zinck et al. were able to avoid secondary photolysis reactions of ligands, which do lead to carbon contamination of III–V compound films when 193 nm radiation is used [34,35].

Epitaxial growth of a $\text{Hg}_{0.2}\text{Cd}_{0.8}\text{Te}$ film on CdTe at 150°C by direct ArF (193 nm) laser photolysis of dimethylmercury, dimethylcadmium and dimethyltelluride was reported earlier by Morris [39]. Growth rates up to $4\ \mu\text{m}/\text{h}$ were obtained with the laser beam parallel to the substrate. However, film purity was not reported.

Our experiments demonstrating photon-controlled superlattice growth, and these recent experiments on epitaxial II–VI compound growth both used excimer lasers in the parallel beam geometry. Thus, the combined experiments point the way toward low temperature photon-controlled growth of epitaxial superlattice structures, using the spatial and spectral selectivity of pulsed excimer lasers to control film growth reactions, while retaining the practical advantages of vapor phase transport of parent molecules. Since the parallel-laser-beam geometry can be used, transient surface heating and film decomposition are not expected to occur, and photoepitaxial film growth should occur at sufficiently low substrate temperatures (at least for II–VI compounds) that thermal equilibrium vacancy generation and interfacial diffusion will be reduced to acceptable levels. Thus, high quality films and well-defined superlattice layers should result; our results imply that excimer laser photoepitaxial growth of superlattices with about monolayer precision is possible.

5. Summary

Our experiments demonstrate that pulsed excimer laser photolysis can be used to achieve high precision deposition of amorphous superlattice structures that have repeatable layer thicknesses and well-defined boundaries between layers. The use of pulsed excimer lasers results in “digital” control over film thickness with much less than a monolayer deposited per laser pulse, while their high repetition rate allows attractive overall deposition rates to be maintained. However, the reflectivity of a growing superlattice structure is sensitive to the n and k values for the individual layers. These values must be known under actual deposition conditions, and depositions must be carried out at sufficiently low temperatures that interfacial interactions do not occur, if optical control of superlattice deposition is to be achieved. We anticipate that the generality of the photolytic film growth concept will make it possible in the future to extend these results to low temperature fabrication of a wide variety of other materials, phases, and multilayered structures, including epitaxial structures.

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