Ultrahigh vacuum glancing angle deposition system for thin films with controlled three-dimensional nanoscale structure

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An ultrahigh vacuum apparatus for the deposition of thin films with controlled three-dimensional nanometer-scale structure is described. Our system allows an alternate, faster, cheaper way of obtaining nanoscale structured thin films when compared to traditional procedures of patterning and etching. It also allows creation of porous structures that are unattainable with known techniques. The unique feature of this system is the dynamic modification of the substrate tilt and azimuthal orientation with respect to the vapor source during deposition of a thin film. Atomic-scale geometrical shadowing creates a strong directional dependence in the aggregation of the film, conferring control over the resulting morphological structure on a scale of less than 10 nm. Motion can create pillars, helixes, zig–zags, etc. Significant features of the apparatus include variable substrate temperature, insertion and removal of specimens from atmospheric conditions without venting the deposition system, computer controlled process parameters, and *in situ* analysis capabilities. The deposition system was successfully employed for the fabrication of a variety of nanostructured thin films with a wide range of potential applications. © *2004 American Institute of Physics.* [DOI: 10.1063/1.1667254]

I. INTRODUCTION

Optical or electron lithographic patterning is today's dominant technology for creating nanometer-scale structure in thin films.[1] The drawbacks of this process are high costs, long processing time, and severe constraints on suitable materials, primarily related to etchant availability.

An alternate way of obtaining controlled three-dimensional (3D) nanoscale structured thin films is the glancing angle deposition (GLAD) method, which employs oblique angle deposition and substrate motion.[2–4] GLAD provides advantages over conventional patterning procedures, being an *in situ* ''sculpting'' technique. Depending on the deposition rate and film thickness, three-dimensional nanostructured thin films can be fabricated in as little as 30 min. Many of the nanostructures obtainable with GLAD cannot be achieved by lithographic or other patterning techniques. GLAD enables unique structures, and offers an alternative to lithography for fabricating certain nanostructures in a shorter time, with few material restrictions, and with lower costs. Films obtained with this method are often called sculptured thin films (STF), described by Lakhtakia *et al.*[5] and Robbie *et al.*[6]

The versatility of form in films fabricated with GLAD is complemented by the wide range of suitable materials, including insulators, metals, semiconductors, and others, vaporized by resistive or electron beam evaporation,[3] sputtering,[7] or pulsed laser ablation.[8] The myriad attainable microstructures, together with the plethora of depositable materials, make these films remarkable candidates for application in electronics, optics, magnetics, medicine, and chemistry. The GLAD process also has the advantage of being a one-step fabrication method with promise for industrial scale-up.

GLAD nanostructure morphologies are composed of atomic-scale filamentary structures that result from the competitive aggregation process of atoms accumulating on the substrate. The structure might be described as pillars, or columns, that can be bent into various forms with various sizes: as chevrons, zig–zags, helical columns, superhelical with varying diameter, and very complex 3D nanostuructures including multilayers. Some structures clearly exhibit the power-law scaling structures characteristic of fractals. A few examples of attainable nanostructures are shown schematically in Fig. 1.

This method of thin film deposition is relatively new, therefore the field and applications have still to be explored. Many applications are at an early stage of development: magnetic media high density information storage,[9] liquid crystal display technology,[10] photonic crystals,[11,12] optical rotators, polarization beam splitters,[13] optical filters,[14–19] polarization inverter,[16] gas and liquid sensors,[20,21] catalysis of constituents of automotive exhaust.[22]

Many applications have yet to be explored.[2,23] Proposed applications of GLAD films include: bioluminescence sensors, electroluminescent devices, optically transparent conducting films sculptured from pure metals, multistate electronic switches based on filamentary conduction, optical sensors that can detect and quantify various chemical and biological fluids, microsieves for the entrapment of viruses, porous materials for growing biological tissues (e.g., prosthesis coating to help promote bone attachment), chemical sensors, catalytic reaction surfaces, optical coatings, thermo-

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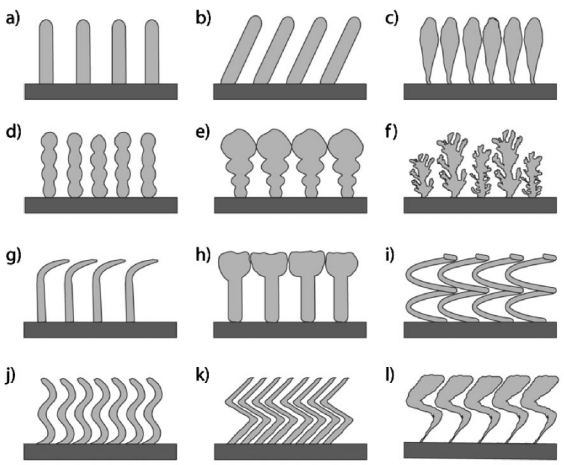

FIG. 1. Schematic depiction of various nanostructures obtained in GLAD thin films: (a) straight pillars, (b) inclined columns, (c), (d), (e) pillars with variable diameter, (f) fractal-like structures, (g), (h) columns with capping layers, (i) helix, (j) waved columns, (k) zig–zag, (l) zig–zag pillars with varying diameter.

electric materials, quantum effect devices, field emitters, and solar cells.

As thin films fabricated with GLAD owe their nanoscale structure to atomic scale geometric shadowing during the aggregation of vapor atoms onto the substrate, and as the shadow geometry depends on the orientation of the substrate with respect to vapor source, controlled substrate motion allows sculpting of film nanostructure into predictable forms. Near glancing incidence, with vapor arriving nearly parallel to the substrate plane, the shadows become extreme, producing porous structures of columns inclined towards the vapor source.[24,25]

The GLAD technique combines substrate rotation, $\omega(t)$, with oblique angle incidence, $\alpha(t)$ (see Fig. 2). The controlled variation in time of both angles leads to the formation of various microstructured thin films.

The present article describes the design, construction, and operation of an ultrahigh vacuum (UHV) GLAD deposition system employing an electron beam evaporation source. The computer control system for the deposition apparatus was developed to allow great freedom in the specification of substrate tilt and rotation during deposition. Elementary microstructures (helix, zig–zag, slanted, etc.) combined with interpolation through user specified tables of film thickness (or equivalently, time) and substrate position, enable synthesis of films with nearly unlimited complexity. The deposition performance of our apparatus has been confirmed by the fabrication of thin films with a large variety of nanostructure morphologies, demonstrating potential for numerous applications.

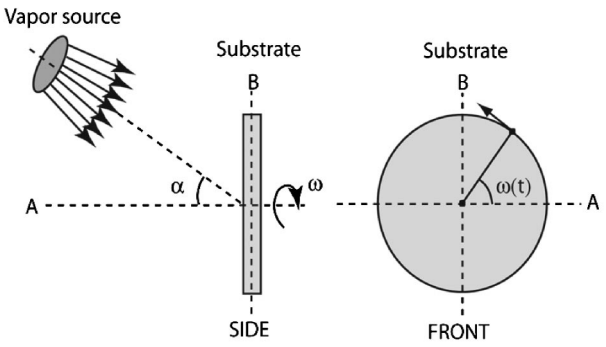

FIG. 2. Schematic description of the GLAD process. Angles of substrate tilt α and rotation $\omega(t)=$const are controlled by stepper motors situated outside the vacuum system operating through a feedthrough and gear system.

II. GLAD SYSTEM DESIGN CONSIDERATIONS

The basic requirements for glancing angle deposition of high quality thin films with controlled nanostructure are the following:

(1) Control of substrate rotation and tilt orientation relative to the vapor source to allow synthesis of thin films with specific nanostructure. In addition, there is a clear requirement for good control over all deposition conditions that affect the microstructure of deposited thin films.
(2) A highly collimated or point-like vapor source that can provide a narrow angular distribution of flux arriving on the substrate. As geometrical shadowing is critical to the GLAD technique, directionality in the arriving vapor is necessary. A small source such as an evaporator or an effusion cell is best, and background gas pressure must be reduced to minimize gas scattering of the vapor.
(3) Variable substrate temperature in order to enable variation of film nanostructure by controlling heat input to the nucleation and aggregation processes.
(4) Synthesis of thin films in a controlled environment. The deposition has to be performed at a vacuum better than 10^{-6} Pa (10^{-8} Torr) in order to minimize the effects of thin films contamination.
(5) The existence of a fast and easy transfer mechanism of specimens from atmospheric conditions to an UHV environment at a specific sample orientation. An efficient UHV chamber design allows the introduction and removal of samples or substrates without venting and pumping the main chamber, saving considerable time.
(6) The load-lock chamber has to be equipped with several sample holders to make the introduction and removal of specimens efficient.
(7) The deposition parameters must be controlled by computer and all process parameters must be monitored throughout the deposition process.
(8) The system should be equipped with several ports for future improvements.

While designing the GLAD UHV deposition system, the above aspects were taken into consideration. In addition, the chambers design, selection of pumping system, and provisions for incorporating various features and accessories were decided based on the above requirements. Care has been taken for the mechanical strength and for selection of UHV compatible materials in the construction of the system.

III. UHV SYSTEM DESCRIPTION

A photograph of the UHV system is shown in Fig. 3. The GLAD UHV chamber is constructed of type 304 stainless steel (SS). All demountable flange ports are of conflat type

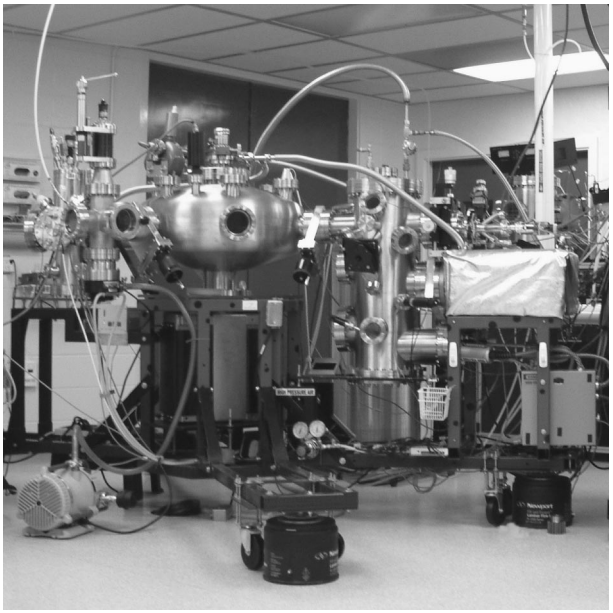

FIG. 3. Photograph of the GLAD UHV deposition system.

(Ref. 27), and use oxygen-free high-conductivity (OFHC) copper gaskets. The other vacuum components such as motion feedthroughs, electrical feedthroughs, gate valves, are specified as UHV compatible. The system consists of four main components: the load-lock chamber, distribution chamber, the GLAD deposition chamber, and the STM chamber. A three-dimensional drawing of the GLAD deposition system is shown in Fig. 4. The schematic drawing of the top view of the system is shown in Fig. 5. The system is situated in a 110 m^2 (1200 sq. feet) clean room, also housing structure and surface analysis capabilities.

A. Load-lock chamber

The load-lock chamber is used for introducing the samples into the UHV system from atmospheric pressure. The load lock contains an elevator onto which three sample platens are first loaded. The sample docks are attached to a vertical shaft capable of 100 mm vertical translational travel and full 360° rotation. The receiving docks can translate vertically and rotate in the horizontal plane. A dry mechanical scroll pump, and a turbomolecular pump, are used to achieve vacuum conditions of 10^{-4} Pa (10^{-6} Torr) in approximately 10 min, when a gate valve is opened and a rack-and-pinion transfer arm is extended from the distribution chamber into the load lock. The arm transfers substrate platens between the load lock, the STM, and the GLAD deposition chamber. More details on the pumping system are presented in the ''UHV conditions'' section below. When required, the system can be vented to atmospheric pressure with dry nitrogen gas through an inlet in the load-lock chamber.

B. STM chamber

The STM chamber is equipped with an RHK Technology, Inc.[26] scanning tunneling microscope. The STM stage design employs heavy, compact sample pucks (required to minimize vibrations), whereas the GLAD deposition system requires small thermal mass, low profile sample platens (required to allow oblique deposition and maximum temperature variation). As a result, the RHK STM and GLAD transfer systems operate on different principles, and a custom sample transfer system was needed to integrate the two. Several devices were designed, including sample platens for GLAD and STM, a platen fork to mount on the extendable transfer arm, sample platen docks, and various mating pieces.[11] In addition, a STM tip exchange piece was designed to enable the replacement of a STM tip from atmosphere onto the scanning head in UHV. Optimization of the transfer system design is ongoing.

C. Distribution chamber

The distribution chamber from Kurt J. Lesker Co.[27] contains a rack-and-pinion-transfer arm capable of 760 mm translational and 360° rotational movement. This chamber

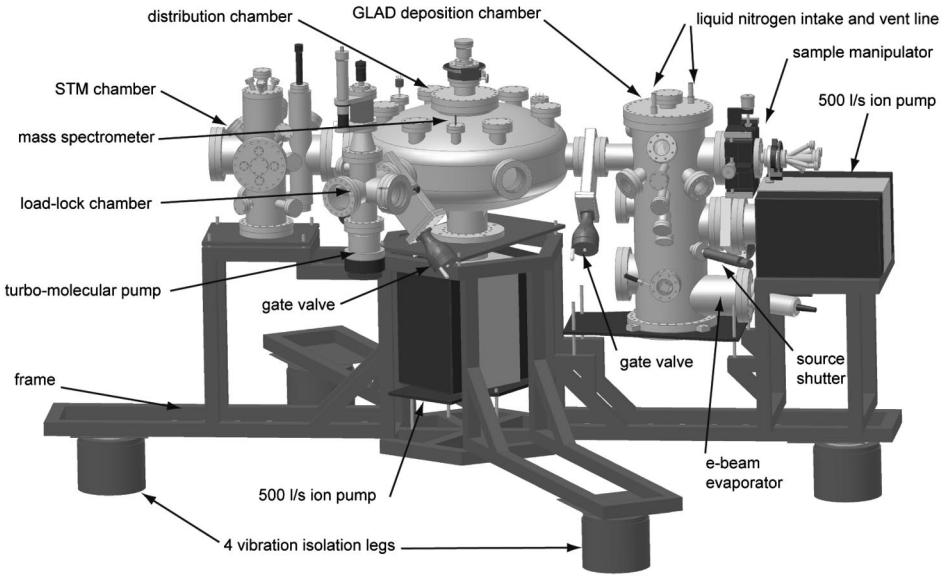

FIG. 4. Three-dimensional layout of the GLAD UHV deposition system. To scale.

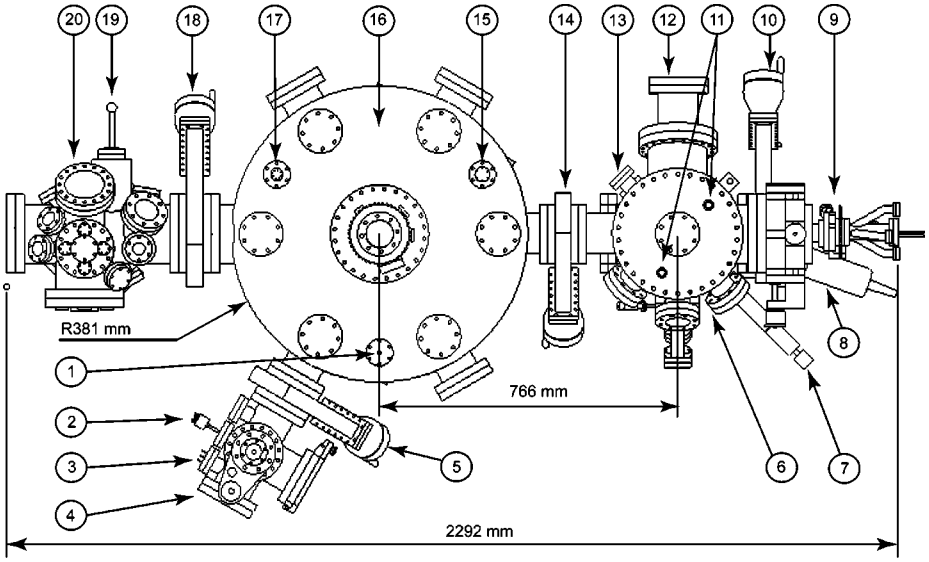

FIG. 5. Schematic drawing of the GLAD UHV deposition system: (1) mass spectrometer, (2) thermocouple gauge, (3) Bayart–Alpert ion gauge, (4) load-lock chamber, (5) gate valve, (6) GLAD deposition chamber, (7) source shutter, (8) electron beam evaporator, (9) sample manipulator with stepper motors, (10) gate valve, (11) liquid nitrogen intake and vent line, (12) port for RHEED screen, (13) crystal thickness monitor, (14) gate valve, (15) Bayart–Alpert ion gauge, (16) distribution chamber, (17) thermocouple gauge, (18) gate valve, (19) STM transfer wobblestick, (20) STM chamber. To scale. A dry scrool pump (not shown) is located below the load-lock chamber.

includes six 6 in. (15 mm) conflat flanged ports positioned on the circumference at 60° intervals, and smaller ports used for a mass spectrometer and pressure gauges. Three of the larger ports are connected via gate valves to the deposition, STM, and load-lock chambers.

D. GLAD deposition chamber

Figure 6 shows a schematic drawing of the GLAD deposition chamber. The chamber is cylindrical with a height of 724 mm and diameter of 273 mm with a wall thickness of 3 mm. The GLAD deposition chamber is equipped with a Telemark[28] 4-pocket electron beam evaporator, a Thermionics[29] sample manipulator, a Telemark[28] quartz crystal microbalance deposition-rate monitor, ports for ellipsometry source and detector, infrared (IR) thermometer, a reflection high-energy electron diffraction (RHEED) port, thermocouple and ion gauges,[30] and electrical and motion feedthroughs. An interior cryo-shroud annulus cools the chamber when filled with liquid nitrogen, and prevents vapor from coating most of the interior walls. Chamber cooling minimizes the outgassing pressure rise that results from heating by the evaporator during deposition.

The upper section of the GLAD chamber houses the substrate holder on the sample manipulator (Fig. 7) with heating and rotation capabilities. This section of the chamber has three view ports for sample transfer and monitoring of the substrate during deposition, one situated normal and two at 20° with respect to the substrate holder. Several other ports at various angles with the substrate holder will be used for *in situ* measurements and characterization of thin films. The deposition rate can be monitored with a quartz crystal microbalance monitor, via a Telemark[28] deposition controller. While the deposition controller has the capability to feedback control the evaporator power, and therefore the deposition rate, coordination with the GLAD substrate motion con-

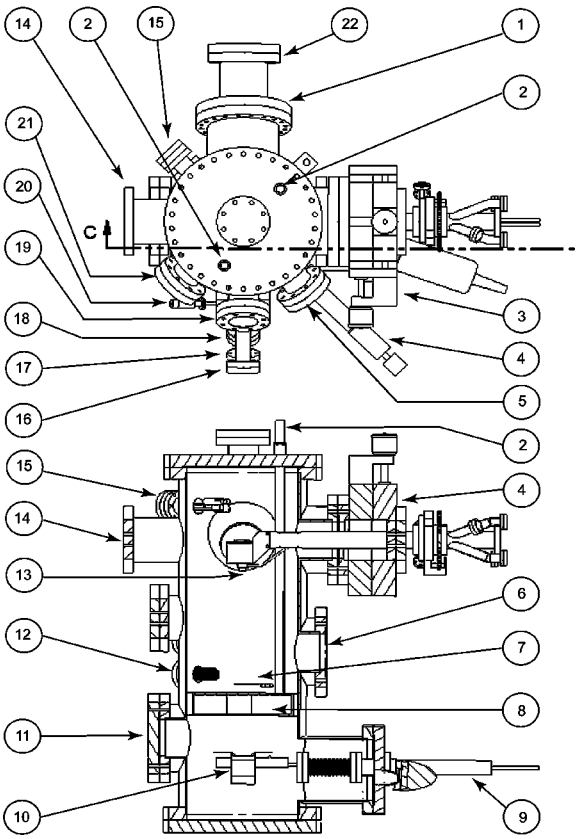

FIG. 6. Schematic drawing of the UHV GLAD deposition chamber. The upper part is a top view and the lower part is a section. Its components are (1) RHEED adapter flange, (2) liquid nitrogen intake and vent line, (3) sample manipulator with stepper motors (not shown), (4) source shutter, (5) viewport, (6) ion pump port, (7) source shutter, (8) liquid nitrogen filled cryo-puck, (9) electron beam control and power (7 kV, 6 kW), (10) electron beam evaporator with copper hearts, (11) evaporator reload port, (12) ion gauge, (13) substrate, (14) distribution chamber, (15) quartz crystal thickness monitor, (16) RHEED electron source, (17) infrared thermometer port, (18) ellipsometry source port, (19) viewport, (20) viewport shutter, (21) sample transfer viewport, (22) evaporator viewport. To scale.

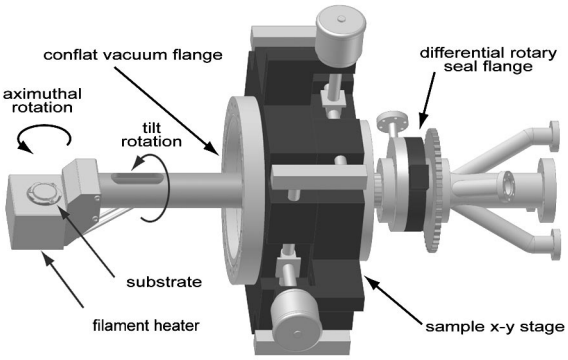

FIG. 7. Schematics of GLAD manipulator. The azimuthal and tilt rotations are executed by two stepper motors (not shown). To scale.

trol system has been problematic, and the evaporation rate is currently manually stabilized. A pneumatic shutter allows the crystal to be shielded from vapor. The control software can periodically close the shutter to prolong the life of the crystal for thick film depositions.

The lower section of the deposition chamber houses the electron beam evaporator[31] with four crucibles, electron beam emitter, high-voltage feedthroughs with a safety interlocked cover, a shutter, and an annular liquid nitrogen dewar, or ''cryo-puck.'' A shuttered viewport offers visibility of the crucible and source material bombarded by the high-power electron beam. Optical filters (welding glass) are used to view the evaporator source during deposition as most molten materials are too bright for direct viewing.

The Telemark[28] e beam evaporator source is located 500 mm below the substrate, providing a stable source of vapor during the deposition. The incident vapor flux has a maximum divergence angle of 2.9° for the geometry described in Fig. 6, in the case of substrates situated at normal angles of incidence. The electron beam emission occurs with the extraction of electrons from a hot tungsten filament that is biased to approximately 4 kV and emits a current of up to 1 A. The electrons are accelerated by the voltage toward a solid piece of the thin film source material melting in a carbon, refractory metal, or ceramic crucible in one of four 7.0 cubic centimeter (cc) pockets in the water-cooled copper hearth. The electron bombardment heats the source material to evaporation. A pair of electromagnet coils controls the beam profile, and the beam emission current is controlled manually, or automatically by the deposition controller, holding the deposition rate constant at a desired value. A pneumatic shutter can shield the evaporation substrate from the source. In addition, angled ports are provided at various angles with respect to the substrate, to be utilized for *in situ* measurements.

The Thermionics[29] sample manipulator was specified to be suitable for GLAD deposition, which requires continuous substrate rotation together with oblique incidence. The sample manipulator is mounted on a horizontal stage positioned with two precise micrometers, and is capable of $x-y$ translations of 12 mm perpendicular to its primary axis. A typical substrate is a 25 mm diameter disk of silicon or glass, 250 ± 50 microns thick, and polished on one or two sides. Platens for other substrates are available, up to 25 mm diameter and including 1 cm square. The substrate is mounted on a sample platen that is used to move the substrate around the chamber, and manipulate it during deposition. The sample dock on the manipulator is a copper ring with three molybdenum clips on the perimeter for holding the sample platen, and it is capable of continuous azimuthal rotation $\omega(t)$ about the substrate normal with simultaneously heating or cooling. The entire manipulator body is capable of 280° tilt rotation α about its primary axis, which passes through the surface plane of the substrate. Both rotations are automated by geared stepper motors, enabling simultaneous and dynamic control of the tilt and azimuthal angle of the substrate with respect to the vapor source. A substrate heater is situated directly beneath the platen, within the copper support ring. It is formed from a tungsten filament tightly wound in a 20 mm diameter serpentine pattern around alumina insulators wired to a molybdenum support plate. Behind the molybdenum plate, a set of thin tantalum foils provide a contact position for the internal *K*-type (chromel–alumel) thermocouple, and create a temperature gradient to the manipulator body. An annular liquid nitrogen dewar surrounds the heater assembly, and employs an explosion-bonded weld to connect the copper cooling ring (in contact with substrate platen) to the stainless steel cooling lines that transport liquid nitrogen through the manipulator differential rotary seal. Liquid nitrogen or water cooling must continuously cool the manipulator when the substrate heater is used. A maximum current of 15 Amps can be passed through the heater filament, radiatively heating the sample holder and the substrate. The reading of an internal thermocouple fixed on the rear of the heater gives a reference temperature that was calibrated to the measured temperature of the substrate. For calibration, a fine-wire thermocouple was placed in contact with various substrates on sample platens, mounted on the manipulator, its reading giving the substrate surface temperature. Temperature measurements from both thermocouples were recorded as the filament was slowly heated and cooled. A calibration graph of substrate temperature versus the heater temperature indicates a linear dependence for heater temperatures above 200 °C (or 30 °C substrate surface temperature). The filament heater is capable of maintaining the substrate temperature up to approximately 1000 °C for extended periods while the substrate is simultaneously rotated and tilted. Liquid nitrogen cooling can lower the substrate temperature to approximately -100 °C.

The sample platen material was carefully chosen to be suitable for use in the GLAD ultrahigh vacuum system. As many experiments require a heated substrate, the sample platen must withstand high temperatures. Refractive metals with high melting temperatures are suitable for this purpose, and include tungsten, tantalum, and molybdenum. Primarily for mechanical properties and ease of machining, we chose molybdenum as the material for substrate platens and screws, and tantalum foil for the thin retaining clips.[32] The platens are also designed to have small thermal mass in order to achieve effective heating and cooling of the substrate. Unheated parts, including the sample transfer dock and clips, are made of SS. Finally, to ensure cleanliness of the pro-

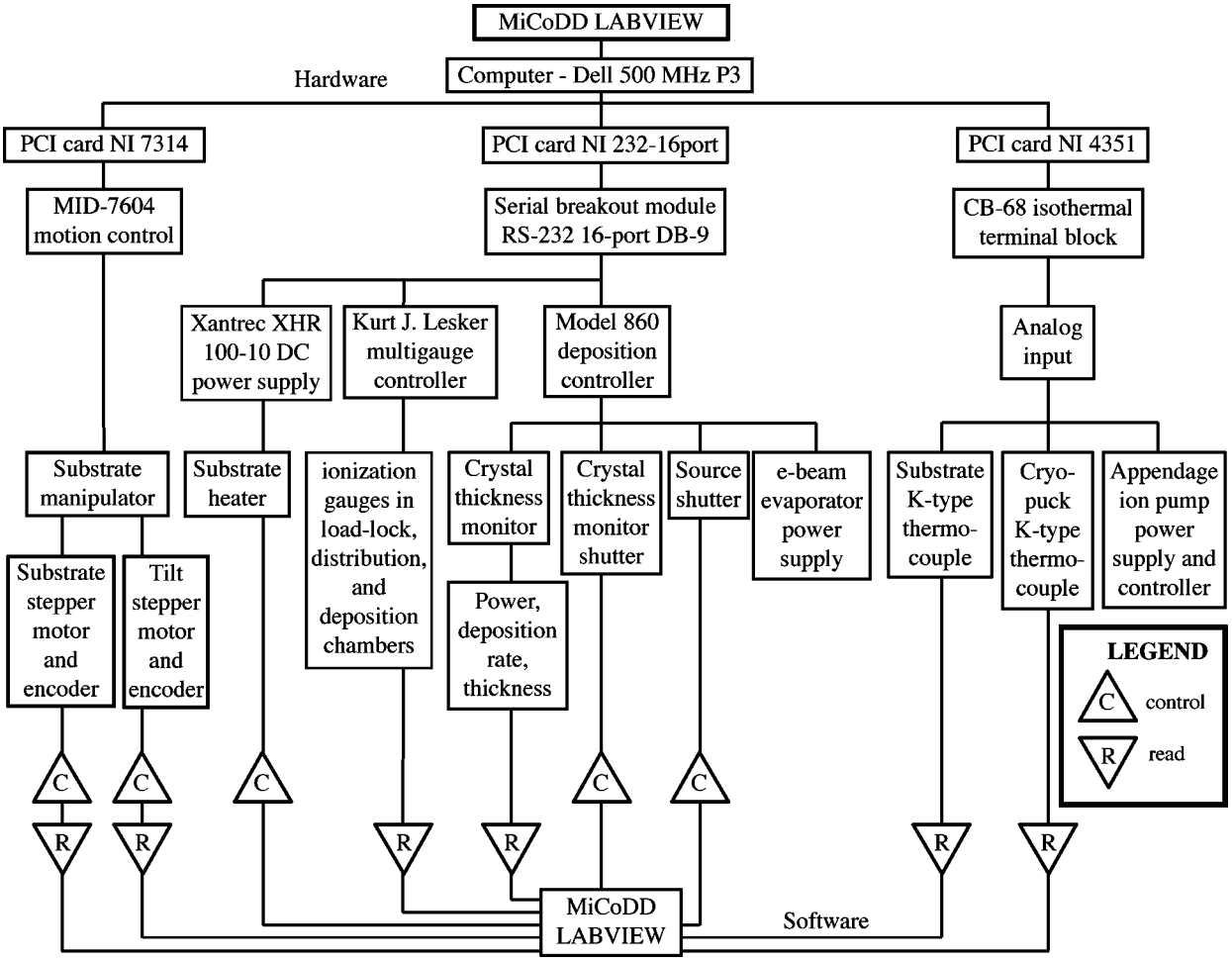

FIG. 8. Schematic diagram of communication interface for the LabVIEW deposition program.

duced films, ultrapure materials were used for the active part of the deposition system.

As deposition is performed at varying orientation of the substrate relative to the vapor, shadowing from clips or external objects must be minimized for the growth of any GLAD film. For this reason, the design of the sample platen requires an elevated substrate with low profile retaining clips. We use three thin tantalum foil clips (0.125 mm thickness) screwed to the molybdenum platen with #0–80 molybdenum screws. In addition, special care is taken in acquiring specimens for characterization away from clip positions (for optical spectra or electron microscope images).

Rotational movement of the sample manipulator and transfer arm is made possible by two differentially pumped rotary seal flanges.[29] The differentially pumped rotary seals provide rotation freedom through the wall of the UHV system ensuring that the low pressure of the UHV system is maintained. The first stages (atmosphere side) of the two rotary seals are connected to the load-lock chamber through a valve, and are periodically pumped with the turbomolecular pump. The second stages (vacuum side) are connected together to a 2 1/s appendage ion pump. When the pressure reaches 10^{-4} Pa (10^{-6} Torr), the appendage ion pump is turned on and the two stages are isolated from each other. The pressure of the appendage ion pump is measured and logged, and is typically 6×10^{-4} Pa (5×10^{-6} Torr).

E. LabVIEW deposition control program

Scientific investigation of thin films fabricated with GLAD requires the reliable control and monitoring of the many film growth parameters: substrate tilt and azimuthal orientation, substrate velocity, evaporator power and deposition rate, accumulated thickness, shutter positions, gas composition and pressure, substrate, cryo-puck, and chamber temperatures. The GLAD deposition experiment is controlled with a customized LabVIEW[33] control and data collection program—MiCoDD (pronounced "my cod")—Microstructure Construction by Dynamic Deposition, running in Windows 2000[34] on a 500 MHz Dell[35] Pentium 3 computer with 128 MB of RAM. A schematic diagram of the communication interface for the MiCoDD deposition program is shown in Fig. 8. The computer controls and monitors the deposition chamber through three PCI cards purchased from National Instruments.[33]

The stepper motors on the Thermionics[29] substrate manipulator are controlled with a National Instruments[33] MID-7604 controller interfaced via a NI7314 PCI card. Alumel–chromel (type *K*) thermocouples are placed behind the heater on the manipulator, and inside the cryo-puck, and are read by a National Instruments[33] NI4351 PCI card, connected through a CB-68T isothermal terminal block configurable connector accessory enclosure. Two Bayard–Alpert gauges,

one in the distribution chamber and one in the load lock, and a UHV ion gauge in the deposition chamber, are monitored through an RS-232 connection to a Lesker[27] multigauge controller. The electron beam evaporator can be operated remotely through the Telemark[28] Deposition Controller model 860, communicating with MyCoDD with an RS-232 connection. MyCoDD reads the evaporator power from the deposition controller, together with the deposition rate and accumulated thickness, and monitors the crystal thickness monitor shutter, the source shutter, and the cryo-puck fill valve. A National Instruments[33] serial breakout module (16-port model DB-9) is the go-between for all RS-232 connections to computer PCI card in the system.

The National Instruments[33] NI4351 PCI card also monitors the appendage ion pump power supply and controller, three thermocouple gauges, and thermocouples distributed over the body of the vacuum chambers.

The deposition program is a dynamic control and monitoring program able to display every one of the described system parameters. All information is logged for future validation. Film microstructure can be chosen from a combination of predefined elementary microstructures. These include: zig–zags, polygons and helixes. User defined structures are also accepted in a text format, specifying the tilt and azimuthal positions for increasing film thickness. This feature is useful for engineering thin films with very complicated microstructures, such as rugate filters.[19] Film parameters are ultimately limited by the finite velocity and acceleration of the stepper motors. Maximum thickness of a film is limited by the finite volume of the four electron beam crucibles (4×7 cc).

F. UHV conditions

The major deciding factors in selecting the pumping system to obtain UHV conditions are: the process pressure required, pumping speed at process pressure, the gas load, and tolerable contamination levels. The gas load is the result of a combination of the volume of the system, outgassing, and gas leaking. The leaks can be made negligible by adopting proper sealing and welding techniques. Thus, the main gas loads are due to outgassing from the internal surfaces of the chamber and the volume of the system.

Considering the above points, to achieve UHV conditions, our system employs two 500 l/s Duniway[36] ion pumps, a BOC Edwards[37] 250H 200 l/s turbomolecular pump and a BOC Edwards[37] XDS10 10 l/s oil-free mechanical scroll pump. The mechanical pump serves as a roughing pump for the entire system and a fore pump for the turbomolecular pump, due to its excellent capabilities for pumping large gas loads. Turbomolecular pumps have the disadvantage that they can cause vibrations in the system and must exhaust to atmosphere through the mechanical pump; consequently they are shut down when the desired pressure is achieved and the ion pumps can be used to maintain vacuum, and continue pumping.

The two ion pumps are suitable for continuous operation of the GLAD UHV system, as they do not need to exhaust to atmosphere, do not cause vibrations and they can achieve vacuum conditions of 10^{-9} Pa. Because the ion pumps do not operate above 10^{-2} Pa and have long pumping times at pressures higher than 10^{-4} Pa, our system is always pumped below 3×10^{-4} Pa with the turbomolecular pump before the ion pumps are turned on.

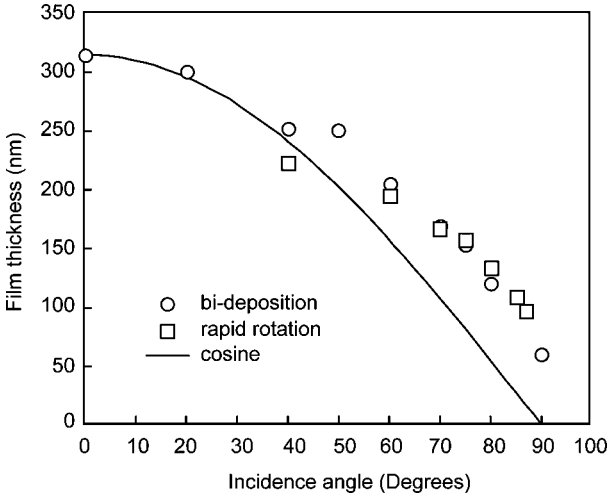

FIG. 9. Calibration of the film thickness profile–film thickness versus incidence angle.

The UHV system includes provisions for bakeout, necessary to achieve vacuum of 10^{-7} Pa or better. Although we do not routinely carry out a bakeout and degass of the UHV system, these operations have been performed successfully to achieve a base pressure of 3×10^{-8} Pa (2×10^{-10} Torr). The typical base pressure reached without bakeout is 10^{-7} Pa (10^{-9} Torr), and the pressure during deposition is always less than 10^{-5} Pa (10^{-7} Torr).

G. Thickness calibration

As the materials we fabricate are deposited under various angles of incidence, the calculation of film thickness from the deposition rate is not sufficiently accurate. Two factors influence the thickness departure from the nominal thickness, defined as the thickness of the film deposited from vapor arriving normal to the substrate.

(1) The magnitude of vapor flux arriving on a unit area of the substrate is proportional to the cosine of the incidence angle. Films deposited at high angles of inclination of the substrate from the direction of vapor will theoretically have decreased thickness compared to nominal thickness.

(2) The porosity increases for higher incidence angles due to self-shadowing under conditions of limited adatom mobility.

The combination of these two factors gives the profile of the thickness dependence on the angle of incidence. In Fig. 9 is shown a calibration of film thickness versus angle of incidence for films of silicon deposited on rapidly rotating substrates. Measurements from scanning electron microscope (SEM) are in agreement with those from spectroscopic ellipsometry. For the ellipsometric analysis we used a Woollam[38] M-2000, fixed angle, spectroscopic ellipsometer with a spectral range 370–1700 nm. The scanning electron microscopy measurements were performed with a LEO 1530 SEM.[39]

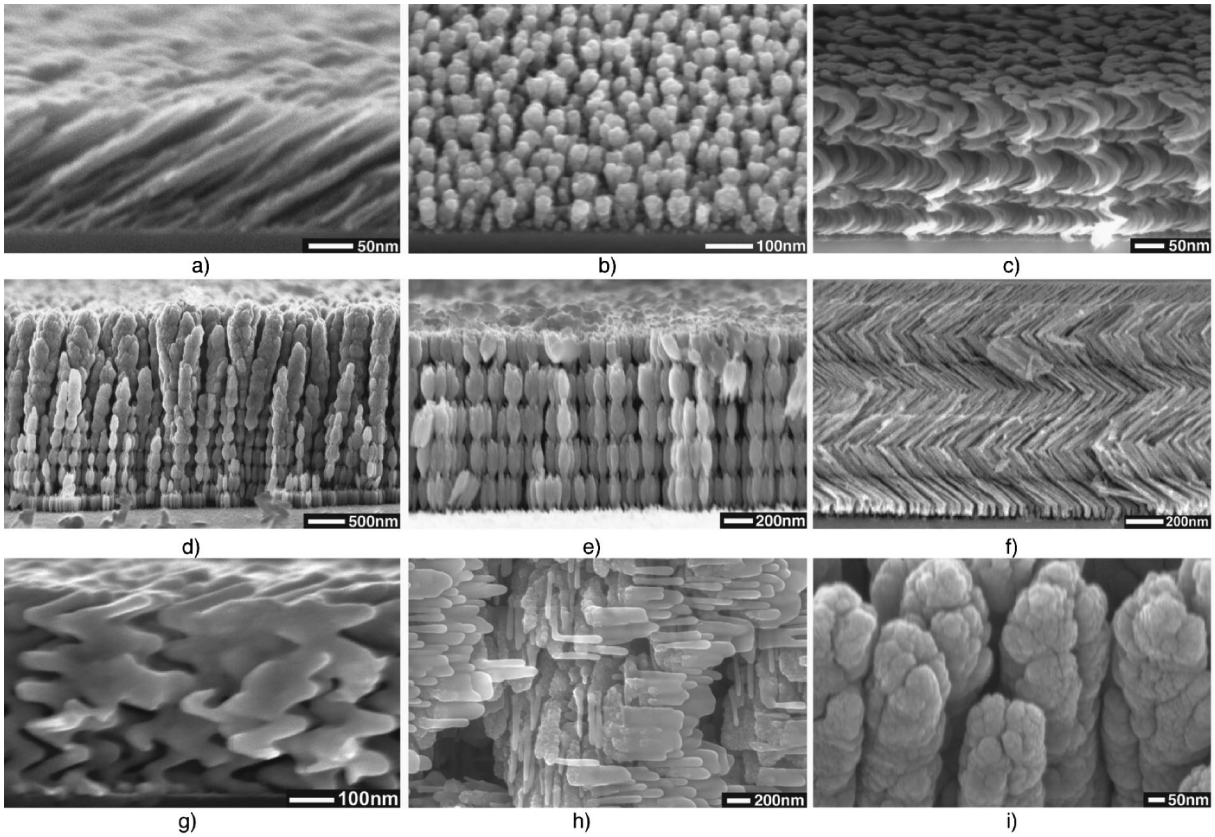

FIG. 10. SEM micrographs of thin films deposited with the UHV GLAD system (a) Si on Si(100) oblique columns, incidence angle of 83 degrees, 200 nm thick, base pressure of 10^{-8} Pa, (b) Si pillars on Si(100) deposited at 85 degrees tilt, 200 nm thick, base pressure of 1×10^{-7} Pa, (c) Si spirals, (d) Si rugate film on glass substrate, 4000 nm thick, base pressure of 4×10^{-6} Pa, (e) Si rugate film with an antireflection layer on glass substrate, 2400 nm thick, base pressure 1×10^{-6} Pa, (f) Si square helix film on glass substrate at 80 degrees tilt, (g) Cu zig–zag film on Si(100), deposited at 80 degrees tilt, 1350 nm thick, 7×10^{-7} Pa base pressure, (h) Si square spiral film on glass substrate, 83 degrees tilt, 3500 nm thick, 1×10^{-6} Pa base pressure, substrate temperature of 560 °C, (i) Si envelope corrected rugate on glass substrate, 4000 nm thick, base pressure of 4×10^{-6} Pa.

IV. OPERATION

To operate the GLAD system, cleaned substrates are mounted on sample platen. The sample platens are transferred onto receiving docks in the load-lock chamber. The load-lock chamber is closed, and the pressure is reduced 3×10^{-4} Pa. The valve between the distribution chamber and load lock is then opened, and the transfer arm is extended into the load-lock chamber. A sample platen is transferred onto the transfer arm fork, and the arm is retracted. The gate valve between the distribution chamber and load-lock chamber is closed. Confirming that the pressure in the distribution and GLAD deposition chambers are similar, the gate valve between the two is opened. Finally, the sample platen is transferred from the transfer arm onto the Thermionics[29] sample manipulator inside the GLAD chamber and the arm fork is retracted. The manipulator is then centered and the deposition is performed. After the deposition, the sample is removed by repeating the procedure in reverse.

To begin the deposition procedure, the cryo-puck is filled with liquid nitrogen. After the reservoir has cooled, the electron beam evaporator power is increased (with the source shutter open) to achieve a stable desired deposition rate. Once the rate is stable, the deposition procedure begins, using the MyCODD program to control and monitor.

V. DEPOSITION PERFORMANCE

The UHV GLAD deposition system has the ability to grow various controlled thin films nanostructures in a wide range of substrate temperatures. The combination of substrate rotation and variation of the angle of incidence facilitates a wide range of deposition conditions leading to various microstructures.

We have successfully deposited Ag, Al, Au, Cu, Co, Cr, Ni, Si, Ti, Mo, MgF_2, SiO_2, on substrates of glass, Si, SiC, and SiO_2. Depositions of films of different thickness have been carried out at constant rates of 0.2–0.5 nm/s onto substrates with various orientation and rotation programs.

In Fig. 10 we present some SEM images of films deposited with the UHV GLAD deposition system: (a) oblique film formed of slanted columns (b) pillar film obtained by tilting the substrate and continuously rotating it during deposition, (c) spirals, (d) rugate film,[40] (e) rugate film with antireflection coating,[19] (f) square helix, (g) zig–zag film, (h) square spiral film at high temperature, (i) envelope corrected rugate.[40] These structures represent only a fraction of the possible attainable nanostructures with this deposition system.

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[28] Telemark, 51 Whitney Place, Fremont, CA 94539, http://www.telemrk.com/telemrk/index.htm
[29] Thermionics Vacuum Products, 231-B Otto Street, Port Townsend, WA 98368, http://www.thermionics.com/index.shtml
[30] The ion gauge near the evaporator in the deposition chamber is sometimes inoperable while the evaporator is energized due to some form of electrical interference. When this occurs, the pressure at the ion gauge in the distribution chamber is monitored. As the chambers are connected with a high-conductance tube, the pressures are usually nearly equal.
[31] The evaporator is slightly misaligned; rotated about its central axis. The chamber was designed with American-style flanges, with a bolt at the top of each, while the Telemark evaporator is designed with a European-style flange with a between-bolt space at the top of the flange. To mount the evaporator in the chamber, it was rotated one-half a bolt position toward the front of the chamber (9 degrees on the 20 bolt circle). Fortunately, the evaporator performance is essentially unaffected by the slight tilt (about a 1% decrease in deposition rate and ultimate film thickness).
[32] As some of our experiments required heating up to 1000 °C, SS could not be chosen because nickel (Ni), one of its constituents, evaporates from SS at high temperatures, compromising the purity of the films.
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[35] Dell Computer Corporation, One Dell Way, Round Rock, Texas 78682, http://www.dell.com
[36] Duniway Stockroom Corp., 1305 Space Park Way Mountain View, CA 94043, http://www.duniway.com/
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[38] J. A. Woollam Co., Inc., 645 M Street, Suite 102 Lincoln, NE 68508, http://www.jawoollam.com/
[39] LEO Electron Microscopy Ltd., Clifton Road, Cambridge CB1 3HQ, England, http://www.leo-em.co.uk
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