Temperature Control and Calibration Issues in the Growth, Processing and Characterization of Electronic Materials

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This paper summarizes the temperature control and calibration issues encountered in the growth, processing and characterization of electronic materials in the Microdevices Laboratory of JPL's Center for Space Microelectronics Technology. The primary problem area is identified as temperature control during epitaxial materials growth. While qualitative thermal measurements are feasible and reproducibility is often achievable within a given system, absolute calibration is essentially impossible in many cases, precluding the possibility of portability from one system to another. The procedures utilized in the MDL for thermal measurements during epitaxial growth are described, and their limitations discussed.

INTRODUCTION

The charter of JPL's Microdevices Laboratory (MDL) is the development of electronic materials and devices for space applications. The MDL, a 38,000 square foot facility opened in late 1988, contains class 100,000 area for materials growth and characterization, class 100 and 1,000 areas for deposition and device processing, and class 10 areas for e-beam and photolithography. The facilities also include a variety of deposition, processing and characterization capabilities, as detailed in Table I. In the area of semiconductor materials and device development, the main program thrusts include strained-layer superlattices, silicides and amorphous silicon, medium to far infrared silicon-compatible detectors, photonic devices, and neural network systems. There are also strong programs in conventional and high-temperature superconducting materials, SIS mixers, superconducting electronics, and miniature electron-tunneling based sensors. Low-temperature STM capabilities, and a new technique developed in the MDL, ballistic electron emission microscopy, which enables microscopic imaging of the electronic properties of subsurface interfaces, enhance these programs.

Among the wide variety of deposition, processing and characterization methods utilized in the MDL, the primary area where temperature control remains an unsolved problem is in the epitaxial growth of electronic materials. Three important epitaxial growth techniques, molecular beam epitaxy (MBE), metal-organic chemical vapor deposition (MOCVD), and laser-assisted chemical vapor deposition (LACVD) have been selected for the focus of this paper. While thermal control is

critical for the reproducible fabrication of high-quality films by any of these techniques, in each case the growth environments interfere with accurate temperature measurements of the growth surface. For each deposition technique, I discuss the thermal control requirements, the growth environment, the methods currently utilized for temperature measurement and control, and the limitations of these methods.

MOLECULAR BEAM EPITAXY (MBE)

MBE places the most demanding requirements on thermal control of any of the epitaxial growth techniques. Reproducible growth of high-quality films requires reproducible sample temperatures controlled to within 1 K. Sample homogeneity requires even better uniformity across the growth surface, which is typically 1 to 3 inches in diameter. This is because the mobility of the deposited atoms is thermally activated, and thus depends exponentially on the local ambient temperature. Thus small variations in temperature across the growth surface can result in large changes in surface atom kinetics, causing significant sample inhomogeneity. MBE growth is carried out in ultra-high vacuum, typically ~ 10^{-11} Torr, and temperatures of 400 - 600 °C for the growth of III-V materials, and room temperature and up for group IV samples. Growth occurs when a beam of the desired atoms or molecules impinges on a substrate. For growth homogeneity the sample mount is often rotated during deposition, making good thermal contact difficult or impossible. In addition, the thermal capacity, and surface emissivity evolve as deposition occurs, so that the sample temperature can vary greatly even when a fixed heater current is provided. In addition, the deposition of material within the chamber is not totally limited to the substrate surface, and any surfaces in the vicinity of the growth surface will eventually become coated with sample material.

An approximate measurement of the temperature in the growth chamber is normally obtained using a thermocouple. In order to escape contamination during sample growth, the thermocouple must be placed under the sample mount where it is shielded from the beams of atoms/molecules which are impinging on the growth surface. If the sample mount is stationary during growth, the thermocouple can be inserted in a well carved out at the back of the mount for better thermal contact. However, if the sample mount is being rotated, which is frequently done for better deposition uniformity, then the thermocouple must be placed below the mount, and cannot be effectively heat sunk to provide an accurate measure of the sample mount temperature. Even when an accurate measure of the sample mount is obtained, there can also be large thermal gradients between the back of the sample mount and the sample surface, so an estimate of the temperature at the back surface may

not provide a realistic measurement of the growth surface temperature. Since the discrepancy depends on the instantaneous sample emissivity, it can be strongly dependent on the evolving (and usually unknown) sample characteristics during growth, and consequently cannot be effectively calibrated.

Another method of calibrating the growth temperature takes advantage of an easily observable eutectic critical point. For example, the sharp transition at ~ 570 °C for the interdiffusion of aluminum on silicon, which can be identified by a large change in reflectivity, is often used to determine the control settings required to achieve this temperature in a given growth chamber. Such calibration measurements are normally carried out in separate runs with no gas flow, rather than during an actual sample deposition, and are especially useful in establishing consistent growth temperatures within a given system over long periods of time. This method offers the advantage of measuring the temperature at the *front* surface of the substrate where growth actually occurs, rather than at the back of the substrate holder, and thus avoids problems associated with thermal gradients in the sample mount and substrate. Inherently, it also provides a solid thermal contact with the substrate. However, since the surface temperature of a growing sample depends on the instantaneous surface emissivity, this calibration cannot provide an absolute measure of the growth surface temperature for an arbitrary sample, and as mentioned earlier, fixed heater current may not be equivalent to a fixed sample temperature if the surface emissivity changes during growth.

Standard IR pyrometry, in which one measures the difference in brightness of the sample emission at two IR wavelengths in order to determine the equivalent black-body temperature, offers an alternative approach. Since it is inherently a contactless measurement, it bypasses the problems associated with poor heat sinking. In principle it can also be used during the deposition process, offering the possibility of active feedback for temperature control during growth. However, this method suffers from its own set of problems. First, the emission flux from the sample is often rather low, because the deposition is carried out at relatively low temperatures. Coupled with the small solid angle for optical access available in typical MBE systems, this weak emission can be difficult to observe with enough sensitivity for an accurate determination of the temperature. In addition, since the samples are usually rather imperfect black-body emitters, a two-point determination may not provide enough information for an accurate determination of the temperature. However, the measurement of the entire spectrum is not usually practical given the low flux and the changing emissivity of the sample surface. Finally, in most cases the sample and substrate materials are transparent to the IR emission, so that the desired signal from the sample surface may be

completely overwhelmed by the emission from the sample mount and heater elements.

Finally, an additional contactless optical method, photoreflectance, offers many advantages. In this technique, the optical gap of the deposited material is determined from the phororeflectivity spectrum, which in turn is used to determine the temperature from previously determined data on the band gap of the material as a function of temperature. This technique samples only the material within the optical absorption length of the sample surface, and for heteroepitaxy can even be used to determine the temperature of the growing layer without interfering signals from the substrate. The measurement can be made using light of relatively high intensity travelling in an optical fiber within the deposition chamber, offering more readily observable optical signals than available with IR emission pyrometry. However, a wide enough range of frequencies must be scanned to permit a fit to the complex derivative spectra. Since the temperature dependence of semiconductors is typically on the order of 0.1 to 1.0 meV per degree Kelvin, this method will not provide accurate temperature readings if the spectrum is greatly broadened by crystal imperfections or by thermal effects at high growth temperatures. In addition, this method is not useful for the deposition of alloys, because the band gap depends not only on the temperature, but also on the precise alloy composition, which cannot be determined independently. The effects of confinement on the electron states will also tend to cause calibration difficulties for thin-layer structures. Of course this method also fails for the deposition of gapless materials such as metal overlayers. Nevertheless, it offers distinct advantages over the other methods described, and is likely to become more widespread in the future.

METAL-ORGANIC CHEMICAL VAPOR DEPOSITION (MOCVD)

As with MBE deposition, temperature control is critical for the growth of high-quality films by MOCVD. The most stringent requirements are actually for the source temperatures (± 0.1 K), but fortunately these demands are relatively easily met, since the sources can be immersed in constant-temperature baths. In reality, control of the substrate temperature to a desired uncertainty of ± 2 K is considerably harder to achieve. The vacuum used for MOCVD deposition is not as extreme as for MBE, typically only in the range 0.1 to 1 atmosphere, and the growth temperatures range from 600 - 750 °C for III-V materials, and 350 - 450 °C for II-VI growth. The primary additional constraint on techniques for thermal control in MOCVD growth is the presence of toxic, corrosive and almost opaque vapors in the growth chamber during deposition. Their presence requires that any in situ sensor be encapsulated for protection

from the environment, and precludes any optical measurement during growth. In addition, the relatively high density of these gases in the vicinity of the sample surface results in significant convective cooling of the growing surface, as well as continuous deposition on all nearby exposed surfaces.

The two primary techniques used in the MDL for temperature measurements in MOCVD growth are the thermocouple and IR pyrometry approaches described in the previous section on MBE growth. The corrosive gas environment requires that the thermocouple be encapsulated, normally in a glass tube, which further degrades the thermal link between the sensor and sample. The thermocouple must also be placed behind or inside the sample mount to keep the device away from the area of heavy materials deposition. IR pyrometry capabilities are also limited by the MOCVD growth environment. The relatively opaque gases present in the chamber during growth preclude the use of this technique during actual growth cycles. IR pyrometry can only be used to precalibrate the control settings before growth is commenced, and the additional convective cooling which occurs under growth conditions limits the accuracy of such pregrowth calibrations.

LASER-ASSISTED CHEMICAL VAPOR DEPOSITION (LACVD)

Since LACVD growth is not a heater-driven process, temperature control of the sample environment is less critical than for the previously-described growth techniques, and $\pm$ 25 K is considered acceptable. The growth environment is basically the same as for MOCVD, with the exception of the ambient vacuum, which is typically held at $\sim 10^{-6}$ Torr for LACVD growth, intermediate to that of MOCVD and MBE. LACVD is an ultra-low temperature growth technique, typically utilizing temperatures close to room temperature. Growth is stimulated at these low ambient temperatures by pulsed laser excitation of the surface.

The only technique currently employed for monitoring the temperature during LACVD growth within the MDL is thermocouple based. The IR emission signals are too small for the IR pyrometry approach due to the low growth temperatures. As with MBE growth, the thermocouple is placed inside the sample mount, rather than at the growth surface where material deposition is occurring. Unfortunately, this does not provide a good measure of the temperature at the laser-stimulated growth surface. The discrepancy has been measured directly using a second thermocouple at the surface with all conditions identical to those during growth, but without initiating the gas flow, and temperature gradients as large as 100 K have been observed between the back of the sample mount and the substrate surface. In addition this discrepancy depends on the ambient temperature and sample parameters, and thus cannot easily be calibrated

and corrected for. Finally, the laser pulses may cause transient local excursions in the temperature which are not reflected by the thermocouple measurement which has a rather slow response. The ability to measure these transient surface effects would not only allow growth temperature specifications which would be portable from one system to another, but would also be useful in elucidating the underlying mechanisms responsible for growth by this technique. Unfortunately, there is currently no method capable of obtaining such measurements.

SUMMARY

This manuscript has summarized the temperature measurement and control issues important in the growth, processing and characterization of electronic materias in JPL's MDL. Although accurate thermal control is required for a variety of processes, the major obstacles remain in the area of materials growth. In this paper these issues have been examined in detail for MBE, MOCVD and LACVD growth. In all three cases, the existing temperature measurement technologies are marginally adequate. In general it is possible to reproduce consistent growth conditions for the growth of a particular type of sample in a given growth chamber. However, sample-to-sample differences due to effects such as surface emissivity (MBE), convective cooling (MOCVD) and laser heating effects (LACVD) cause unknown variations in the temperature at the growth surface during the deposition of an arbitrary new material or structure. The inability to measure the temperature of the growth surface itself precludes the ability to compare the actual growth temperature among different growth chambers. As a result, portability of precise growth conditions is unfortunately still beyond existing capabilities, and the optimal growth parameters must be determined empirically for each growth chamber and for each new material or structure.

TABLE I. <u>CAPABILITIES OF THE MICRODEVICES LABORATORY</u>

MATERIAL DEPOSITION	SURFACE / INTERFACE CHARACTERIZATION
• evaporation • molecular beam epitaxy (Si and III-V) • liquid phase epitaxy (LPE) • metal organic chemical vapor deposition (MOCVD) • laser assisted chemical vapor deposition (LACVD)	• STM / BEEM • ESCA / SAM • TEM • SEM
LITHOGRAPHY / DEVICE FABRICATION • electron-beam and optical lithographies • diffusion and oxidation furnaces • wet and dry etching • reactive ion etching	BULK MATERIALS AND DEVICE CHARACTERIZATION • transport • optoelectronic