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The Workshop on Intelligent Procesing for MBE and MOCVD was held in Santa Fe, New Mexico on January 27 & 28, 1993. It assembled approximately fifty(50) researchers in the field of advanced sensors and controls to address issues in realization of intelligent control of MBE and MOCVD for III-V and II-VI devices. The workshop consisted of open sessions and presentations from researchers in the field of growth, sensors and control theory. MBE and MOCVD, presently being the two primary epitaxial growth technologies for quantum structured devices, were discussed to identify issues and directions for sensor and control algorithm development toward the realization of future electronic and photonic devices. Issues discussed were the present status of growth technologies, sensors for in-situ diagnostics, algorithms for real time control and cost of sensor implementation. Possibilities for future intelligent epitaxial systems and the insertion of the sensor/control systems into the technology base were also considered.

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RE: Grant #N00014-93-1-0354
Workshop on Intelligent Processing for MBE and MOCVD
Principal Investigator - George Maracas
Final Technical Report

It has come to our attention that you may not have a copy of the final report for grant #N00014-93-1-0354 titled "Workshop on Intelligent Processing for MBE and MOCVD" that was originally sent out at the end of March 1993. Attached is an additional copy for your records.

Sincerely,

Holly Jordan
Administrative Assistant/Maracas

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Final Report

for

Workshop on Intelligent Processing for MBE and MOCVD

Hotel Santa Fe
Santa Fe, New Mexico

January 27-28, 1992

by

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Abstract

The Workshop on Intelligent Processing for MBE and MOCVD was held at the Hotel Santa Fe, Santa Fe, NM on January 27&28, 1993. It assembled approximately fifty (50) researchers in the field of advanced sensors and controls to address issues in the realization of intelligent control of MBE and MOCVD for III-V and II-VI devices. Presented here is a summary of the technical program and discussions held during the workshop.

Introduction

It is generally agreed that compound semiconductor epitaxial growth technology has had a rapid development over the past twenty years. Advanced devices such as modulation doped field effect transistors (MODFETs), resonant tunneling transistors (RTTs), heterojunction bipolar transistors (HBTs), quantum well lasers, detectors and modulators have been realized and continue to be developed. While successes in these areas have achieved the present state of III-V technology, future generations of devices require even more control of critical dimensions and materials properties than is presently available.

A one and one-half day workshop was held to discuss these issues. The workshop consisted of open sessions and ten minute presentations from researchers in the field of growth, sensors and control theory. MBE and MOCVD, presently being the two primary epitaxial growth technologies for quantum structured devices, were discussed to identify issues and directions for sensor and control algorithm development toward the realization of future electronic and photonic devices. Issues discussed were the present status of growth technologies, sensors for in-situ diagnostics, algorithms for real time control and cost of sensor implementation. Possibilities for future intelligent epitaxial systems and the insertion of the sensor/control systems into the technology base were also considered.

Requirements for future electronics and photonics manufacturing

Jane Alexander (DARPA) opened the workshop with an overview of the requirements for future manufacturing systems. There is a strong sentiment in moving away from the ever-increasingly expensive silicon fabrication lines toward smaller ($50M), flexible lines which can manufacture high-end products. Present fab line utilization is only approximately 35% and considerable amount of time is spent in inspection and cleaning. Cost will be the overriding factor in determining the success of any future fabrication technology.

Ray Balcerak (DARPA) then spoke specifically about infrared focal plane array project and the requirements for epitaxy of II-VI compounds required to realize their goals. One of the major problems in II-VI epitaxy is temperature control. He stressed that temperature measurement in MBE needs to be significantly improved. He recommends that models be developed that describe the growth process and that cost of any intelligent sensor be reduced to approximately $10K.

Growth

The degree of growth process control depends on the nature of the growth mechanisms of a particular technology. For instance, in MBE (an unstable process) several parameters (group III flux, group V flux and temperature) need control to realize reproducible, high quality structures. The process control surface can be considered sharp and thus requires active feedback to maintain process control. Atomic layer epitaxy (ALE), on the other hand is a self assembling process characterized by a flat control surface and thus does not require active control. MOCVD lies somewhere between the two because of its chemically assisted growth nature. It was generally agreed that the different epitaxial growth technologies will require different levels of control.

There are many variables in the growth of III-V and II-VI compounds by MBE and MOCVD. Measurable parameters can be grouped into three categories depending on their relationship to the desired target epitaxial structure. Different materials systems (e.g. AlGaAs and HgCdTe) will have different tolerances on the growth conditions (listed as tertiary variables) and thus sensors for these may require different sensitivity and accuracy.

The process variables for MBE and MOCVD were grouped in order of importance for controlling growth of currently popular heterojunction devices.

Primary variables:
Alloy composition
Thickness

Secondary variables:
Doping
Alloy ordering (including defects)
Interface properties (abruptness)
Chemical state of initial growth surface (substrate properties)

Tertiary variables:
Growth temperature
Ambient pressure, temperature
Precursor species and fluences (beam fluxes, gas flow rates and composition)

Sensors

Sensor requirements for integration into epitaxial process control

In order for an intelligent sensor to become successfully integrated into the III-V and II-VI MBE and MOCVD growth community, it must satisfy certain requirements. Some of the requirements that were discussed are listed below. Atomic layer epitaxy (ALE) was also discussed since, because of its self-limiting, single atomic layer nature, does not require in-situ growth control of thickness or composition. It was suggested that ALE could be used to obtain calibration samples for in-situ or ex-situ sensors. The in-situ diagnostics discussed here can also be applied to ALE.

Sensors should:

provide information on the growing material or the sources in real time
be adaptable to existing growth reactors
not limit the reliability of the growth system
not alter the growth process
be easy to align or calibrate
be compatible with substrate rotation

not significantly affect the cost of the total growth system (be inexpensive)

Depending on the application, the sensor should have the capability to:
operate intermittently to provide process calibration information
or
operate continuously for logging of process parameters or failure alert

A brief description of sensors that have, or possibly may be applied to in-situ epitaxial growth are presented here. The discussion is not comprehensive and only limited to sensors that were discussed at the workshop.

Thermocouples (TC)

This is the most commonly used temperature sensing device because of wide temperature range (typically 0°C to 1200°C) and its low cost (~$1K). A junction between dissimilar metals produces a temperature dependent voltage which is converted to temperature from a calibration table. A (p-i-d) temperature controller produces feedback power to the control point. Accuracy limitations in epitaxial reactors (10°C - 200°C) are a result of TC placement far from the desired control surface (substrate).

Optical pyrometry

The black body radiation (at fairly high temperatures) of a material with a certain emissivity is measured by a photodetector. The choice of detected wavelength depends on the bandgap of the material to be measured. Temperatures above 450°C can be measured for III-Vs. Multiple wavelength pyrometry increases the accuracy of temperature determination and it was shown that, in II-VIs, temperature control of ±1°C was possible. The cost of a multiple wavelength system is $20K - $22K while a single wavelength unit is approximately $5K.

Projection Moiré thermometry

A diffraction grating (e.g. 1μm pitch, L=3mm) is etched into the surface of a substrate of which temperature is to be measured. The amount of wafer expansion (a priori knowledge of the thermal expansion coefficient of material is required) is proportional to the temperature. The optical collection system consists of a beam splitter, lens and CCD camera to observe the Moiré fringes produced by the first order grating diffraction. The lens performs an optical 2D FFT which produces two spots on the CCD camera. The distance between the spots is proportional to the grating size change and thus the temperature. Temperature resolution is less than 1°C and cost is approximately $10K.

Energy dispersive x-ray (EDX)

X-rays induced by an incident electron beam can be used to determine the chemical composition of a material. It was proposed that glancing angle EDX could be used in an MBE system to monitor surface lattice constant and composition. The cost of such a system would be approximately $75K.

Reflection high energy electron diffraction (RHEED)

Diffraction of an electron beam (~10keV) from a crystalline surface onto a phosphorescent screen enables the measurement of surface reconstruction and lattice constant. This is commonly used in ultrahigh vacuum (UHV) reactors (standard equipment on MBE machines) to monitor growth rate and V/III ratio, for example. Growth rates are measured in a layer-by-layer growth mode by observing the intensity variation of a diffracted spot (RHEED oscillations). Using a video camera and synchronizing pattern acquisition with substrate rotation enables surface structure monitoring under substrate rotation. Cross-sectional analysis of streaks can extract surface step distributions. The cost of a RHEED system is approximately \$20K-\$30K plus cost of any video image acquisition equipment.

Optical reflectance

Optical radiation at normal incidence is reflected from the growth surface and detected versus time. Assuming that the optical thickness or optical constants are known versus wavelength, alloy composition, growth rate or thickness can be measured. This has been extended to the growth of microcavity optical devices such as vertical cavity surface emitting lasers (VCSELs) where the Fabry-Perot cavity thickness is adjusted after growth interruption. A basic, one wavelength (HeNe laser) system is \$5K.

Reflectance difference spectroscopy (RDS)

A variation on the optical reflectance technique takes the difference in polarization induced by surface dipoles (for instance) to obtain chemical information of the surface and growth rate. Polarizers and synchronous detection instrumentation are required. The cost is approximately \$30K-40K.

Spectroscopic ellipsometry (SE)

The polarization of light reflected from a surface is analyzed as a function of wavelength. The response is fitted to (predetermined) optical constants of the material and a specific layer structure to extract alloy composition, thickness, growth rate and temperature. Two main types of ellipsometers were discussed: photelastic modulator SE and rotating analyzer SE. SE development is still continuing which is producing commercially available systems costing between \$15K and \$150K.

Infrared transmission/absorption

The temperature dependence of a semiconductor bandgap edge can be used to extract the substrate temperature in MBE and MOCVD. The band edge is determined spectroscopically in the temperature range $100°C<T<800°C$. The position of the band edge can be obtained more accurately by differentiating the absorption spectrum and accuracy of $\pm 2°C$ has been stated. Limitations are on thick, absorbing multilayer structures. The cost of a commercially available system (Intevac) is \$42K.

Ultrasonic gas flow sensing

Accurate determination of gas flows in MOCVD source lines can be obtained by measuring the acoustic velocity of the gases. A commercially available system (Epison) provides a gas velocity signal which has been used to control composition in MOCVD to 0.25%. The units cost \$5K per gas line. Multiplexing one unit among four lines has been demonstrated.

Ultraviolet (UV) absorption and fluorescence

The fluorescence and absorption spectra of excited molecules can be used to identify chemical species and their concentrations. UV lamp excitation is used for absorption and tunable lasers for fluorescence while dispersive optics are used for detection. These techniques can be used near the growth surface or near the sources to obtain information in the gaseous state. An absorption/fluorescence system has been built which can be fitted to existing MBE systems without modifying the growth chamber (Martin Marietta). This system uses light conduits to bring the light parallel to the substrate surface. Typical costs of basic systems is ~\$10K-20K.

Second harmonic generation (SHG)

Laser pulses are incident onto the surface of a semiconductor. The discontinuity in the potential at the interface generates a second harmonic of the incident radiation which is detectable by standard optical techniques. This second harmonic light is surface reconstruction sensitive. The cost of such an instrument is approximately $50K.

Sensor	measurable param	accuracy/sensi.	cost
Thermocouple	T	10 - 200°C	$1K
Optical pyrometer (multi λ)	T	± 1°C	$20K-22K
Projection Moiré	T	<1°C	$10K
X-ray diffraction	t	<1%	$120K
EDX	surface composition	-	$75K
RHEED	R_g, surf. reconstr.	~5%	$20-$30K
Spectroscopic ellipsometry	t, x, T, Refl.	few %	$10 - 150K
Optical reflectance	t, x		$5K
Reflectance difference spectr.	surf. chemistry		$30K-40K
IR transmission/absorption => E_g	T	±2°C	$42K
Ultrasonic gas flow sensing	gas flow	±0.25%	$15K (X4)
UV absorption and fluorescence	chem. species		$10K-20K
Second harmonic generation	surf.state, R_g		$ 50K

Algorithms/control

Efficient analysis and control algorithms need to be developed toward the goal of attaining intelligent processes which will reliably produce target epitaxial structures. This may require concurrent development of growth models and their coupling to sensor models. Several talks on real time parameter tracking and control were presented which addressed possible directions and approaches.

Two approaches are possible when analyzing the growth of a complicated multilayer structure. One is to use all the information gained during the entire growth (history dependent) and the other is to use only the information obtained in the changing surface (history independent). The latter technique as been used to achieve control of the near-surface MBE AlGaAs composition by SE on extremely small structures such as quantum wells. The prior technique is more appropriate for VCSEL mirror structures since the optically active region is several quarter wavelengths of light thick.

Tracking of parameters such as MBE growth rate by SE require algorithms that can adapt to changes in related parameters such as temperature as the optical constants drift with temperature. incomplete data problem algorithms have been successfully used to track growth rate in the presence of varying temperature and surface roughness.

Model based control and sensing has been used in MOCVD by extended Kalman Filtering techniques. A suggestion was to couple sensor models with growth models. Calibration of growth models still needs to be achieved. The number of variables required for some detailed growth models is large. Calibration could require monitoring all aspects of growth which could easily create unmanageably large quantities of data.

It was suggested that describing a complex system such as an MBE requires a nonlinear dynamical system model. Specifically, a decentralized, hierarchical control strategy, including adaptation should be necessary to attain control. A requirement on the sensor response is that the system parameters be sampled five times faster than the response time of the MBE system.

On the subject of system response, an effusion cell flux transient was modeled toward the goal of reducing the transient. The response was approximated by a simple transform in attempts to minimize state information for the cell.

Another approach to the intelligent processing problem involved applying neural networks. There were no presentations on this subject.

Other Issues:

Three other issues were mentioned but, due to time constraints, were not discussed in detail. They are briefly described here and would be appropriate to address more completely in a future workshop.

Is it feasible to have one epitaxial reactor grow all (device) structures?

Such a reactor might be complex, versatile and capable of several in-situ controls. Some discussion ensued about the possibly high cost of such a system in comparison to a dedicated "single-structure" system.

III-V sensor development should be transferable to silicon processes.

This discussion was motivated by the large portion of the semiconductor market presently held by the silicon industry in comparison to compound semiconductors. Epitaxy of III-Vs and II-VIs is more difficult than that of silicon so sensors should be easily adaptable to silicon processes. The evolution of SiGe has put the silicon industry into the compound semiconductor field. Therefore, materials technologies developed for III-Vs and II-VIs could be adapted to SiGe. Such a connection might spur the silicon industry to fund advanced sensor and process control development for non-Si based compounds.

To what extent should detailed growth models be developed?

This question came up because there was very little discussion on first-principles growth models. One view was that efficient development of a growth technology could not occur without deep physical insight into the process. It was generally agreed that some physical models must be available for this purpose and for analyzing situations where the process mysteriously fails to meet specifications. The extent of the required physical model depth was mentioned but not discussed in detail due to time limitations.

Final statements

The Intelligent Processing Workshop spurred a considerable amount of discussion on sensor, analysis and control algorithm and materials growth issues. Comments from the participants, during and after the workshop, were generally positive and there was much interest in holding future workshops. Many constructive and useful suggestions were made on improving the organization and exchange of ideas. These will be implemented if future workshops occur.

Program

Workshop on Intelligent Processing for MBE and MOCVD
January 27 & 28, 1993
Hotel Santa Fe Santa Fe, NM
PROGRAM

Wednesday, January 27, 1993

8:00 - 9:00	**Continental breakfast**
9:00 - 9:15	
George Maracas (ASU)	Introduction, purpose of meeting
9:15 - 10:00	
Jane Alexander (DARPA)	Manufacturing directions for future electronics and photonics
10:00- 10:40	**Growth** - George Maracas
Salah M. Bedair (NCSU)	Film Thickness and Composition Control by ALE Growth
Andrew Purdes (TI)	Sensor-Based MBE - Promise and Limitations
Dick Shealy (Cornell)	Real Time Control of Advanced OMVPE Processes
Ronald Gale (Kopin)	Intelligent Processing of Epitaxial Materials using OMCVD materials
10:40 -11:00	**Coffee break**
11:00- 12:10	**Growth (cont.)** - Manijeh Razeghi
John Dinan (USNVL)	Issues for real-time control of MBE of HgCdTe
John Jensen (Hughes)	HRL activities related to MBE/MOMBE of III-V and II-VI
	Sensors
Doug Collins (CalTech)	Applying RHEED to MBE Process Control
Haluk Sankur (Rockwell)	Laser Reflectance as an in situ monitor for MOCVD of III-V epilayers
Paul Luscher (Intevac)	Accurate, real-time substrate temperature measurement control
Colin Wood (U. MD)	Real-time surface composition control by energy dispersive x-ray analysis
12:15 - 1:30	**Lunch**
1:30 - 2:30	**Sensors** - Andrew Purdes
Walter Duncan (TI)	High-speed Spectral Ellipsometry for In-Situ Diagnostics and Control
John Woollam (U.Nebr.)	Design and Performance of a Low Cost Multiple Wavelength in situ Ellipsometer
George Maracas (ASU)	Spectroscopic ellipsometry for III-V MBE
Manijeh Razeghi (NW U.)	In Situ Investigation of MOCVD Deposition of Semiconductors by RDS
Jeff Tsao (Sandia)	VCSEL growth by reflectance feedback
Ken Bacher (Stanford)	In-situ corrections to vertical cavity optical devices grown by MBE
2:30 - 2:50	**Coffee break**
2:50 - 3:50	**Sensors (cont.)** - Salah Bedair
Steven DenBaars (UCSB)	Composition control of epitaxial films by real time ultrasonic sensing of metalorganic vapor concentration variations

Steve Brueck (U. NM)	Real-time temp. measurement using projection Moiré techniques
Glenn Westphal (TI)	Multi-λ pyrometry for II-VIs on MBE
Tom Pearsall (U. Wash.)	In-situ monitoring of epitaxial growth using UV absorption and fluorescence spectroscopies
Stefan Svensson (Mart.Mar)	Optical beam flux monitoring for MBE
Stuart Irvine (Rockwell)	Integrated in-situ monitoring for II-VI MOVPE
3:50 - 4:40	**Discussion** - Bob Trew
4:40 - 5:00	**Nanofabrication Network** - Jane Alexander

Dinner - everyone for themselves

Thursday, January 28, 1993

8:00 - 9:00	**Continental breakfast**
9:00 - 9:30	**Discussion #1 - recap of previous day** - Maracas
9:30 - 10:20	**Algorithms** - Walter Duncan
Dave Aspnes (NCSU)	A History-Independent Algorithm for Determining Outer-Layer Dielectric Responses from Ellips. Data: Application to Closed-Loop Feedback Control of AlGaAs Epitaxy
Guy R.L. Sohie (GE)	New algorithms for dynamic tracking of temperature and growth parameters in MBE ellipsometry
Ted Schulman (ISI)	Model-based sensing and control for MOCVD
Kostas Tsakalis (ASU)	Control of MBE by spectroscopic ellipsometry
Jim Vlcek (EPI)	Effusion cell flux transient compensation

10:20 - 10:40 **Coffee break**

10:45-12:15 **Discussion #2 - directions** - Dave Aspnes

What are the variables that need to be controlled?
What variables can be measured and controlled? What tolerances are required?
Which variables are presently under control and which need improved control?
What sensor control technologies presently are being used?
What are the algorithmic speed requirements for real time control?
What sensors and controls can reasonably be developed? (the big payoff)
Which sensors have a near term market?
Where do we go from here?

12:15 **Lunch and adjournment**

NOTE:

Presentations are 10 minutes long including questions.
Please be prepared to discuss the questions listed in the discussion sessions (this includes attendees not giving presentations).
Attire is casual.

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