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Chapter 1 Introduction

The project this report describes was a sub-project of Xylaur project. To distinguish this project from Xylaur project in this report, it is called Control Project. Xylaur project involves expertise from the following units: 1) Xylaur Enterprise Ltd. (Now Prosensys Inc.), whose business includes supplying services to semiconductor manufacturing industry, 2) Chemical Engineering Department, 3) Physics Department, and 4) Electrical and Computer Engineering Department of University of New Brunswick. In this chapter the motivation, organization and objectives of Xylaur project, as well as the goal of Control Project, will be described.

1.1 Motivation of Xylaur Project

Semiconductor manufacturing is a complicated process with many stages, Chemical Vapor Deposition (CVD) is one of the stages in which controlled chemical reactions are used to create layers of thin films on wafers.

Traditionally, fixed process recipes with open-loop control and off-line metrology are applied in semiconductor manufacturing [1]. The conventional metrology can only identify process excursions after yield loss [2]. As the size of the wafer is continually increasing (>200 mm in diameter [3]), the cost of yield loss is becoming prohibitively expensive, therefore *in situ* process control is considered to be a critical solution for the future semiconductor factory [2].

The process chosen in this project was TEOS(tetraethoxysilane)/Ozone-based deposition of silicon dioxide thin film at sub-atmospheric pressure. This process

yields silicon dioxide thin films with excellent properties for applications within ULSI (Ultra Large Scale Integration) device fabrication at unusually low process temperatures (350° C to 400° C [4]). However, there are some fundamental problems which have impeded the implementation of this process in ULSI fabrication schemes [4]: The as-deposited films tend to be hygroscopic with significant residual Si-OH and water, but annealing - a traditional route to remove silanols and water - is not viable for this process since it negates the process advantage of low thermal budget; also high temperature processing will accelerate parasitic side reactions, which reduces deposition rate and leads to particle growth. Thus, this process needs a fine balance of conditions to produce high quality films with low particle growth.

The literature shows that gas phase intermediates play important roles in the characteristics of TEOS CVD processes, and well-behaved IR (infrared) absorption characteristics under near-industrial process conditions has been recorded [4]. Therefore in Xylaur project a control system based on FT-IR (Fourier transform infrared) spectroscopy was developed, in which *in situ* analysis of the process chemistry was used for real-time process control of the deposition process in the reactor. The control variable was the absorbance at about 1117 cm⁻¹ in the spectrum of the process reaction, which was selected by Xylaur's chief scientist Dr. Thomas Whidden. Further discussion of the control variable will be presented in Chapter 5: Controller Design.

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1.2 Objective and Tasks of Xylaur Project

The overall goal of Xylaur project was to develop a prototype operator interface, monitor and control unit for real-time process control based on *in situ* analysis of the CVD reaction chemistry. In the first phase, the most important task was to prove the feasibility of using *in situ* IR data for real-time control of the absorbance in a specific area in the IR spectrum.

Xylaur supervised the overall project and coordinated activities among team members. The project team was divided into three groups and each group had its tasks and goals:

1) CVD process characterization group

This group's members were from Xylaur and Chemical Engineering Department of UNB. They determined the infrared signature of dominant intermediates in the TEOS/Ozone based deposition process, correlated the strengths of intermediate signatures with CVD process condition in individual processes, and correlated variations in intermediate concentrations with variations such as process deposition rates, particulate levels, thin film uniformity, and film conformity.

2) FT-IR spectrometer customization group

This group's members were from Physics Department of UNB. They customized and configured the interface between spectrometer and the CVD reactor.

3) Control system development group

This group's members were from Electrical and Computer Engineering

Department of UNB. They determined control system platform including hardware and software with Xylaur staff's help, developed control system software, designed and implemented the process controller.

The first phase of Xylaur project was successfully finished, so was the Control Project. Xylaur (Prosensys) is now planning to implement and test the prototype at industrial sites (beta sites) and further develop the control system.

Chapter 2 Base System

CVD reactions were performed in a customized reactor system, which mimicked actual industrial systems. Xylaur designed and constructed the CVD reactor. It also chose the model of FT-IR spectrometer and spectrum analysis software. All the work for the Control Project was based on this base system.

2.1 Base System Structure

CVD reactions take place in a chamber, Figure 2.1 [4] and Figure 2.2 [4] shows the outside view and inside structure of the chamber.



Figure 2.1 Reactor (outside view)

Figure 2.1 shows the chamber (on the right side of the figure), loadlock (which is used to load and unload wafer into and from the chamber) and the FT-IR spectrometer with its interfaces to the chamber: on both left and right side of the chamber. Figure 2.2 shows the schematic of the chamber interior. The chamber is configured for wafers of size up to 125-mm diameter. It operates in both high vacuum and sub-atmospheric pressure modes.



Figure 2.2 Reactor (interior structure)

The nitrogen, ozone and TEOS enter the chamber through a water-cooled showerhead. TEOS is carried by nitrogen, therefore in the figure there is TEOS/N2. Most of the deposition takes place on the substrate, while some also takes place slowly on the chamber wall. The substrate is heated by Tungsten-Halogen lamps. The quartz plate between the substrate and the lamps is used to minimize the deposition on the lamps. The thermocouples, which have direct contact with the substrate, are used for temperature control. The pressure of the chamber is controlled by the combination of an inline throttle valve and pumping speed reductions by the addition of nitrogen at the mechanical pump. The spectra are obtained by a customized Bomem MB series FT-IR spectrometer, which has interface to a desktop PC. On the PC there is a dedicated data acquisition board and a dedicated software package CAAP (continuous automated analysis program) Frame is run to analyze and manage the data from the spectrometer via the data acquisition board. The IR beam enters the chamber and exits through windows. It focuses directly above the substrate. The detector receives the IR beam and transmits it back to the FT-IR spectrometer for analysis.

There are also electrical and mechanical devices in this system, which are used for monitoring and controlling the reactions. Figure 2.3 shows the (simplified) system



Figure 2.3 System Diagram (Courtesy Xylaur Enterprise Ltd.)

diagram. The gases go through the mass flow controllers (MFCs) and pneumatic

valves (PVs) to enter the chamber. The MFCs are shown by their capacity, e.g., "1 slm N2". The inline throttle valve is controlled by a pressure controller. Finally, there is a temperature controller, which is connected to the thermocouples, to control the chamber temperature. Note that the temperature controller and the pressure controller are not shown in the figure. The MFCs, pressure controller and temperature controller are respectively self-contained, closed-loop control systems with on-board microprocessors. They all have interfaces for remote control (specification of set point). The MFCs have analog I/O interfaces (0 to 5 volts) through which a user can send set points to and receive the reading from the controllers. The PVs are connected to on-off relays, which can be controlled by digital signals: (logical) high or low voltage. Both temperature and pressure controller have the standard RS-232 interface through which a user can send setpoints to and receive the reading from the controllers. These devices make it feasible for automatic control of the basic CVD process variables (not including the absorbance at a specific spectrum area): temperature, pressure and gas flow rates.

2.2 Experiment Procedure

Typical deposition experiment in the system follows a standard procedure:

- 1. Load a wafer (substrate) into the chamber;
- 2. Evacuate the chamber to base pressure (between 10^{-7} and 10^{-6} torr);
- Condition the system with specific values of flow rates, pressure and temperature for individual experiments;

- 4. Deposition takes place for several minutes;
- 5. Put the system through three standard purge cycles;
- 6. Transfer the wafer out of the chamber.

The whole procedure takes about two to three hours, depending on the chamber condition and the purpose of each experiment. During the experiment, the IR spectrum of the reactor is obtained, data is stored and analyzed off-line or online. The thin film resulted from the deposition is studied using other dedicated devices so that correlation between spectrum and film quality can be obtained.

Step 1 and step 6 must be performed by an operator. A Control and Monitor Unit (CMU) was developed in the Control Project, which can perform step 2 to step 5 without the operator's interception. Further details about the experiment procedure and the CMU will be presented in next chapter.

Chapter 3 Control and Monitor Unit

3.1 Control System Architecture

As stated in section 1.1, the objective was controlling the absorbance at about 1117 cm^{-1} in the spectrum of the deposition process, which was sensed by the FT-IR spectrometer. The reaction condition is described by the following parameters: chamber pressure, temperature, flow rates of various gases and open-closed status of the pneumatic valves. Those parameters are controlled by the respective process controllers, which were described in last chapter. Thus the control system architecture can be represented by the following diagram:



Figure 3.1: Control System Architecture

In the Control Project, the absorbance at about 1117 cm^{-1} was chosen to be the control objective and measured variable, while the flow rate of one MFC (the 1 slm

N2 MFC) was chosen as the control variable using the feedback data from FT-IR PC (PC with CAAP Frame and dedicated data acquisition boards installed). Therefore the control system was a SISO (Single Input Single Output) control system. Detailed discussions about the control variable, measured variable and control variable will be presented in Chapter 5 Controller Design.

3.2 Platform of Control and Monitor Unit

3.2.1 Platform Selection

As shown in Figure 3.1, the CMU (Control and Monitor Unit) should fulfill the following objectives:

- 1) Receiving selected spectrum data from FT-IR PC;
- Sending set-points to and receiving process status from process controllers, and set open-closed status of digital valves;
- Displaying reaction status, such as chamber temperature, value of the control variable, etc.;
- Adjusting set-points of certain process variables based on feedback data from FT-IR PC automatically;
- 5) Logging all relevant reaction data for further analysis; and
- 6) Emergency shut down of the system. Since the experiments involve hightemperature chemical reactions, this is a necessary precaution.

The goal of the first phase of this project was to prove the feasibility of using *in situ* IR data for real-time control of the absorbance at a specific area in the IR spectrum. After this goal was accomplished, the goal of the next phase is to beta

test the concept on industrial sites, where the system set-up, such as different types of process controllers, will be different. So the CMU developed in Control Project was a prototype. The cost of the unit, including software and hardware, should be low and the development software should be relatively easy to learn.

It was determined that a desktop personal computer with data acquisition boards and off-the-shelf software should be used to build a PC-based data acquisition and control system as the CMU, because a PC uses an open architecture, its cost is low and it's powerful.

3.2.2 Hardware

There are three plug-in PCI-bus data acquisition boards in the CMU: one for analog inputs, one for analog outputs and one for digital outputs. They are the products of Intelligent Instrumentation [™]. Detailed description of the boards are presented in Appendix A. There was also one serial-port extension board, which provided the system with extra 8 serial ports.

There were three types of communication between the CMU and the reactor system: analog input/output, digital output and serial-port input/output. The analog output board was used to send set-points to three mass flow controllers as shown in Figure 2.3: "1 slm N2", "10 slm N2" and "10 slm O2" respectively, while the analog input board was used to receive status from those three controllers. The digital output board was used for opening (logical 0) and closing (logical 1) pneumatic valves as shown in Figure 2.3. One serial port was used for receiving data from FT-IR PC; one serial port was used for sending set-points and receiving

current status to and from the temperature controller; and one serial port was used for sending set-points and receiving current status to and from the pressure controller.

3.2.3 Software

The PC for CMU had Windows 95 installed. We used Visual Designer[™] 3.0 from Intelligent Instrumentation[™] as the development software. It has built-in support for the three data acquisition boards mentioned above. Visual Designer is "a block diagram-based application construction system. Visual Designer allows you to develop a custom data acquisition and control, signal processing, or monitoring system by drawing the program's data and control flow structure in the form of a diagram, or FlowGram" (quote from Visual Designer's help system). There are two steps for constructing a customized system: First the user draws the data flow and control diagram using function blocks provided by Visual Designer; then the user compiles the diagram into an executable form (flow code) for Visual Designer. Figure 3.2 shows an example. In Figure 3.2 there are three icons representing three blocks: "Set-point", "Digital Meter" and "AnalogOutput". The green arrows shows the data flow, for example a green arrow points from "Set-point" to "Digital Meter" means the data are sent from "Set-point" to "Digital Meter". The gray dialog is where the developer configures the parameters of "AnalogOutput" block; all other blocks have similar dialogs for configuration. Visual Designer has more than 100 types of blocks for functions such as display, data acquisition, simple digital signal processing, file read and write, etc. If the diagram contains function blocks

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involving data acquisition boards, such as "AnalogOutput" block in Figure 3.2, the corresponding board must have been installed on the PC, otherwise the resulted flow code won't be executable or won't run properly.

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Figure 3.2 Example: A Visual Designer Diagram

Once the developer finishes editing and compiling the diagram, the resulted flow code can be load and run. Usually the developer does not choose to run the flow code upon loading, since the user interface needs to be adjusted to desired appearance. Once that is done and the configuration is saved, the flow code will be run. Figure 3.3 is a screen shot of the user interface of a running flow code for above example diagram.



Figure 3.3 Example: The User Interface

3.3 System Operation

The base system was configured in such a way that it could be operated in manual mode and automatic mode. There was a switch in the system to change the mode of operation. As described in section 2.2 (Base System Operation), there were six major steps for operating the system. Under manual mode, the set-points of the process controllers (temperature, pressure and mass flow controllers) and the status (open/closed) of the pneumatic valves were manually set and changed by the operator as the experiment proceeded. Under automatic mode, except for step one (load the wafer) and step six (unload the wafer), the set-points of the process controllers and the status of the pneumatic valves were controlled by the CMU. In normal operations, the system was under manual mode during step one, in which operator loaded the wafer into the chamber. Once it was done, the system would be changed to automatic mode and the CMU took over for step two to step five. Then the system was switched back to manual mode and the operator unloaded the wafer from the chamber, which was step six.

3.3.1 Experiment Recipe

Here is the operation instruction for a typical experiment:

- Check the power of IR sources, gases, pressure and everything else. Turn on the turbo pump at the base pressure and turn on the UHP (ultra high pressure) nitrogen tank.
- 2. Do a wafer background scan. Store the spectrum data. Do other scans if it is required.
- 3. Load a wafer into the chamber.
- 4. Pump down the system for 5 minutes after the wafer is loaded: Open PV13, pump the system to below 0.5 torr; then close PV13, and open PV3; pump to base pressure (between 10^{-5} torr and 10^{-6} torr) for 5 minutes.
- 5. Precondition of the chamber: Close PV3, then open PV13, PV1, PV19, PV14 and PV4. Set the pressure set-point to 400 torr, MFC for nitrogen (100 slm) to 5 slm. Set temperature to 400 °C for 2 minutes. Turn the temperature down to room temperature; and pump the system with PV13 on with nitrogen flow through PV1, PV14, PV4 and PV9.
- Pump the chamber down with PV13 open until it is 0.5 torr in the chamber, then close PV1, PV4, PV14 and PV 19 if the temperature has gone down below 100 °C.
- Close PV13 when the chamber pressure is below 0.5 torr, then open PV3 for turbo pump and pump the chamber down to base pressure (between

 10^{-5} torr and 10^{-6} torr) and keep it for 5 minutes.

- 8. Turn on the IR detector and take a background scan of the chamber, then store the data.
- 9. Establish nitrogen flow rate and chamber pressure as planned for the experiment. Then take an IR scan of the chamber and store the data.
- 10. Set the temperature to the test point and stabilize it.
- 11. Take a background (test background) IR scan for the tests and store the data.
- 12. Set the reactant flow: first nitrogen, then ozone and finally nitrogen with TEOS flow. For ozone, open PV2, PV7, PV8, PV6 and PV18; For nitrogen/TEOS flow, open PV17 and PV16.
- 13. Wait for 1 minute for deposition and do the first IR scan and store data; then do another scan 3 minutes after the beginning of the deposition and another one 4 minutes after the beginning of the deposition.
- 14. Turn off reactant flow (nitrogen/TEOS, ozone) in reverse order of turn-on and lower the temperature.
- 15. Put the system through 3 purge/pump cycles: pump with mechanical pump with PV13 open; after the temperature goes down to 100 °C, turn off the liquid nitrogen; after the chamber pressure is below 0.5 torr for 1 minute, open PV1, PV4, PV14 and PV19; purge the system for 3 minutes.
- 16. Re-establish a nitrogen flow at the level used in the experiment, do a background scan and store the data.
- 17. Close PV13, then open PV3 to pump the reactor to base pressure (between 19

 10^{-5} torr and 10^{-6} torr). Take another background IR scan and store the data.

- 18. Start the procedure of transferring wafer to load lock: turn off PV3, turn on PV13 and PV12 to pump down the load lock; turn off PV12 and open the load lock gate; transfer the wafer away from the chamber; close the load lock gate; bring the load lock to atmosphere pressure.
- 19. Unload the wafer.

The instruction shows that the operation steps are similar for all experiments. The major difference among the experiments is the test condition (reactant flow, chamber pressure and temperature) for the deposition phase, which starts at step 9 and stops at step 13 in above instruction. So it is straightforward to set up a recipe for an experiment and it is easy to modify the recipe for different test conditions. Table 3.1 shows the format of a recipe that is used by the CMU software:

//Step	Step	Time	N2	Press.	N2	O2	Temp.	N2	 Not	Function
//Name	No.	(sec.)	High	(torr)	Low	(slm)	(oC)	Source	 in use	Select
//			(slm)		(sccm)			PV1	 PV20	
Idle	1	2	0	0	0	0	15	1	 1	0
//End of re	ecipe									

Table 3.1 Format of a Recipe

The actual recipe files are tab-separated ASCII text files. In the recipe, the first few rows are for comments, which must start with "//" string. In table 3.1, the first three rows are used for showing the meaning of each column. For example, "N2 Source PV1" shows this column specifies the status of PV1 for each step, where "1" means to close the valve and "0" means to open the valve. The last column is called "Function Select". In CMU software, for some steps certain

special functions need to be applied, for example: value 3 means to hold the procedure and prompt the operator to load the wafer. During the operation, it is not recommended to change the status of several pneumatic valves at the same time, so in the recipe in each step only one pneumatic valve's status is changed.

3.3.2 User Interface

M CAAP							<u>_ 0 x</u>
Cycle time 0.0	Data from	FTR PC 0	00E+00			Status 0	
Current Time	Absorptio	n (ca. 1117 cn	n*(-1) 0	00E+0	D		
Main .							_0 >
82 [PVI] 27.3 [t alm 142]	END IN ISOS	N2(100 slr	n) Set-p Value	ointh 0.0	0.00 stm stm		
MVI 100 dra H2 PM	E PRES CA	N2(1 sim) Set-poi		100 sccm		
02 PM2 FE2 10 da 01 FMS FM	avena Hold	02	Set-poir	x 0.00) sin		
	F919		Set-poin	e	0.00 tor		
- EVEL - F3		ACX	Press. Angle	0.00	torr degree		
	hamber Burger	a. 3	Working	Mode			
		Temp.	Set-poin	t O	degree	Absorption Setpoint 0.8	ca 1117 cm/(-1) Value 0.88E+00
	<u>- 0</u> 00		Value	a	degree	Controller outpu	t 0.000 vots
Cycle time 0 Data Logging	Spare 0.00	Function	0			Step 0	ţ.
Recipe c:/vylaur/test/march1-1.bd						Step Name	
Data File	Timer 0						

Figure 3.4 is a screen shot of the user interface of the CMU software.

Figure 3.4 Screenshot of User Interface

The CMU software consisted of two flow codes (*.fco): One for receiving spectrum data via serial port from FT-IR PC, and one for monitoring and controlling the experiment. The first one continuously received the data and sent them to the second one where the data could be used to modify set-points of relevant process controllers. The received data shows the absorption of bands at about 1117 cm^{-1} of the IR spectra of the reaction. Details of the process will be discussed in next chapter.

In Figure 3.4, the upper part is the interface for the first flow code. The "Cycle Time" shows the execution cycle time of the flow code. The "Status" shows the status of the serial port. The lower part of the figure shows the interface of the second flow code. The diagram at upper left corner is the system diagram previously shown in Figure 2.3. The color of the gray boxes for PV# will change as the status of the valve changes, i.e. gray color means the valve is closed and green color means the valve is open. The "Hold" button is used for suspending the operation. On the right side of the diagram, the set-points and status of the process controllers are shown. "ACX" is the name of the pressure controller. Also the absorbance circa 1117 cm⁻¹ of the IR spectra of the reaction, which is received from first flow code, is shown at the right side of the figure. There is a text field "Controller output" below the text fields for spectrum data. The controller it refers to is what adjusts the set-point of the relevant process controller to achieve absorbance set-point shown in the text field above. Details of this controller will be discussed in next chapter. The text fields at the lower right corner show which step of the recipe is being executed. The text fields at the lower left corner shows the recipe file, flow code execution cycle time and the data logging file. The log file records the status of all process controllers, pneumatic valves, absorbance set-point and value with time stamp. Only when the "Data Logging" button is pressed down will the flow code start recording data.

Chapter 4 Controller Design

As stated in section 1.1, the control variable was the absorbance circa 1117 cm^{-1} in the *in situ* spectrum of TEOS/ozone mixture in the CVD reactor. The CMU was designed to not only automatically operate the CVD system, but also control the absorbance at a desired value. This chapter presents the design process and experiment results of the controller.

4.1 Control Objective and Manipulated Variable

The basic thrust of the Control Project was to study the feasibility of controlling the CVD reaction based on *in situ* IR analyses and real-time manipulation of reagent concentrations and process conditions. The ultimate control variable(s) will be the concentration(s) of selected gas phase intermediates in the CVD reaction. The *in situ* spectra of the CVD process provide the absorption characteristic of the process, and the "individual absorption bands were found to vary in a well-behaved linear fashion with concentration" [5]. Thus, the absorbance of the individual bands can be the measured variables for the control system. As the first step towards accomplishing real-time control of the reagent concentrations, the Control Project focused on controlling one of the absorption bands instead of trying to directly control the reagent concentrations. Thus, in the Control Project the absorbance at about 1117 cm^{-1} of the spectra was the controlled and measured variable of the control system. As one of the absorption bands that are "common to all process gas analyses and can be assigned to overlapping

bands from the precursors and silicon-containing reaction intermediates" [6]. Figure 4.1 [6] is an example of the *in situ* spectra of the CVD process. In Figure 4.1, the unit of x-axis is wave number (cm^{-1}) , while the y-axis is the absorbance (arbitrary unit).



Figure 4.1 Example of In Situ Spectra

Once the controlled and measured variable was identified, the next step was to identify the manipulated variable. The process condition is described by the chamber temperature, chamber pressure and the flow rates of oxygen, nitrogen (with TEOS) and diluent nitrogen. Any change of the above process variables during the deposition can affect the reagent concentration. Figure 4.1 shows that the flow rate of nitrogen with TEOS has significant impact on the absorbance at about 1117 cm^{-1} . Thus, as a starting point, the flow rate of nitrogen with TEOS was chosen as the manipulated variable while other process variables were kept as constant values during the deposition. Note that the CMU did not control the flow rate of nitrogen with TEOS directly, since the CMU can only send set-points to the

MFC via analog output in voltage. The MFC then controls the flow rate according to the set-point. Therefore, the manipulated variable is the output voltage (between 0 and 5 volts) to the MFC.

4.2 System Identification

The objective of system identification is to find out the process model. It is an iterative procedure, which includes the following steps: experiment planning, selection of model structure, parameter estimation and validation [7]. In the Control Project, the objective was to find out the process model, which described the relation between the measured variable, the absorbance at about 1117 cm⁻¹ with arbitrary unit, and the control variable, the output voltage (between 0 and 5 volts) of the set-point of flow rate of nitrogen carrying TEOS. In following sections the control variable (measured variable) will be referred to as Absorbance, and the control variable will be referred to as TEOS.

4.2.1 Open-loop Test

The base system had been operated under manual mode before the development of CMU software. Preliminary experimental data indicated that the process model, which describes the relationship between Absorbance and TEOS, could be expressed as a low-order system with relatively large rise time. Thus using stepwise input signals, which covered the usual operating range of the control variable, should provide enough information about the system. The flow rate of the nitrogen with TEOS ranges from 0 to 1000 sccm (standard cubic centimeter per minute). The corresponding analog output is from 0 to 5 volts. The normal operation set-points were between 300 sccm and 600 sccm. The normal deposition time was around 4 minutes. The deposition not only happened on the substrate, but also on the wall and window of the reactor chamber, though with much slower rate. Thus the deposition should not last for too long, otherwise the deposition on the window would build up to a level that it decreased the accuracy of the spectral data.

In the test, except for the control variable, other process variables were kept constant during the deposition, their values were: temperature = 280 °C, pressure = 300 torr, flow rate of oxygen with ozone = 3 slm, flow rate of diluent nitrogen = 3 slm. The deposition lasted for 8 minutes. In the first 2 minutes, TEOS = 150 sccm; in the next 2 minutes, TEOS = 300 sccm; in the next 2 minutes, TEOS = 450 sccm; and in the final 2 minutes, TEOS = 600 sccm.

During the test, the data of the process variables were recorded on the Control PC with sampling time t = 1 second. The Absorbance was recorded on the FT-IR PC with sampling time t around 3.3 seconds. To maintain reasonable level of data accuracy, the sampling time for the Absorbance cannot be too short since the spectrometer needed several scans to obtain the spectrum. Both Control PC and FT-IR PC recorded the data with time stamp to each sampling, so the curve of Absorbance vs. TEOS against time could be drawn from the data.



Figure 4.2 Absorbance vs. TEOS

Figure 4.2 shows the curve: the straight line "stair case" represents the normalized TEOS set-points and the curve represents the Absorbance, which was expressed in arbitrary units. The deposition lasted for 8 minutes, during which TEOS was changed every two minutes. Other process variables were kept constant during the 8-minute deposition. After that, the systems entered the transition status towards the purge-pump cycles. Thus from then on the data were not used to identify the process model.

From the curve, the following observation were obtained:

1. Before the deposition the Absorbance was close to 0 (less than 0.03), while more than 10 minutes after the deposition, the Absorbance was still more than 0.06.

- During the period when TEOS was 450 sccm (the 3rd stair of the curve), the Absorbance did not oscillate around a steady-state value. Instead it oscillated around a ramp.
- 3. There was a time delay of the response of Absorbance during the first period, when TEOS was from 0 to 150 sccm.
- 4. The oscillation amplitude of the response when TEOS was 600 sccm was stronger than other periods.

Normally, the deposition phase of an experiment was no more than four minutes since as time goes on there will be deposition reaction occurs at the windows besides on the substrate, which decreases the accuracy of the spectrum data. In this open-loop test, the deposition phase was eight minutes. This may explain the observation one and two: after four minutes, the deposition on the windows had built up to a level that was strong enough to affect the spectrum data. For observation three, one possible explanation is that the reaction did not start immediately. For observation four, one possible explanation is that the reaction intermediate was less stable at this process condition.

Overall the step-wise open loop test indicated that the process can be modeled as a linear low-order system, not counting the window deposition side effect and the time delay in response in low range. This generally facilitates the control system design and implementation.

4.2.2 Least-squares Method

Least-squares method was used for estimation of model parameters. Least-square

method assumes the model structure is linear in unknown parameters [7].

In a general least-squares problem, it is assumed that the system can be approximated by the model:

$$\hat{\mathbf{y}} = \boldsymbol{\theta}_1 \boldsymbol{\varphi}_1(\mathbf{x}) + \boldsymbol{\theta}_2 \boldsymbol{\varphi}_2(\mathbf{x}) + \dots + \boldsymbol{\theta}_n \boldsymbol{\varphi}_n(\mathbf{x})$$
(4.1)

where $\phi_1, \phi_2, ..., \phi_n$ are known functions and $\theta_1, \theta_2, ..., \theta_n$ are unknown

parameters. A loss function is defined as: $J(\theta) = \frac{1}{2} \sum_{i=1}^{N} \epsilon_i^2$ where

$$e_i = y_i - q_j j_1(x_i) - q_j j_2(x_i) - \cdots - q_n j_n(x_i), y_i \text{ and } x_i \ (i = 1, 2, \dots, N) \text{ are}$$

observations obtained from an experiment. The problem is to determine the unknown parameters in such a way that the loss function is minimum. The solution to the problem is:

$$\theta = (\Phi^T \Phi)^{-1} \Phi^T \mathbf{y} \tag{4.2}$$

provided that $\Phi^T \Phi$ is nonsingular. [7]

Loosely speaking, if the input signal in the experiment is sufficiently rich, the least-squares method can be used to estimate the unknown parameters in dynamic systems. [7]

4.2.3 Model Structure and Parameter Estimation

Based on the curve in Figure 4.2 and to simplify the design, the process can be modeled as a first-order system. Its output-input relation can be expressed as [7]:

$$y(k) = -ay(k-1) + bu(k-1)$$
 (4.3)

where y(k) represents the kth sample of Absorbance, u(k-1) represents the (k-1)th sample of TEOS.

Using the least-squares method, the problem of finding a and b can be expressed as to find a and b in such a way that the loss function J(a,b) =

$$\frac{1}{2} \sum_{k=2}^{N} e(k)^2 \text{ is minimal, where } \varepsilon(k) = y(k) - \hat{y}(k) = y(k) + ay(k-1) - bu(k-1) .$$

Compare this system with the general case in section 4.2.2 gives:

$$y = \begin{bmatrix} y(2) \\ y(3) \\ . \\ . \\ . \\ . \\ y(N) \end{bmatrix}, \Phi = \begin{bmatrix} -y(1) & u(1) \\ -y(2) & u(2) \\ . & . \\ . \\ . & . \\ -y(N-1) & u(N-1) \end{bmatrix}, \varepsilon = \begin{bmatrix} e(2) \\ e(3) \\ . \\ . \\ . \\ e(N) \end{bmatrix}$$

and

 $\boldsymbol{\theta} = \begin{bmatrix} a & b \end{bmatrix}^T$

Hence, from (4.2) there is $\theta = \begin{bmatrix} a & b \end{bmatrix}^T = (\Phi^T \Phi)^{-1} \Phi^T y$ (4.4)

Equation (4.3) describes a discrete-time system, which approximates the actual continuous-time system. Since it can be modeled as a first-order system, its transfer function can be expressed as $G(s) = \frac{K}{1+T_s}$, or y = (Ku - y)/T (4.5)

Assuming that h is the sample time, y and u are constant over the interval

 $t_{k-1} < t < t_k$, then by using the standard solution there is [8]:

$$y(t_k) = y(t_{k-1})e^{-h/T} + K(1 - e^{-h/T})u_{k-1}$$
(4.6)

Comparing equation (4.6) and (4.1) there is [8]:

$$T = -h\ln(-a), K = a/(1+b)$$
 (4.7)

Thus both continuous-time and discrete-time system model are obtained.

Applying equation (4.3) to (4.7) to the data from open-loop test with h=3.3 second, there is:

$$y(k) = -ay(k-1) + bu(k-1) = 0.4891y(k-1) + 0.1772u(k-1)$$
(4.8)

and

$$G(s) = \frac{K}{1+Ts} = \frac{0.3468}{1+4.6145s} \tag{4.9}$$

4.2.4 Model Validation

To validate the obtained model, which was described by (4.8) and (4.9), the input for the open-loop test were used as the input to (4.9). To ignore the effect of time delay of response to low-end input (0 to 150 sccm), several samples of the original input were set to 0. Figure 4.3 compares the calculated output with the output from the open-loop test:



Figure 4.3 Model Validation

In the first graph of Figure 4.3, the values of K and T are those from equation (4.9). The graph shows that the model has better approximation to the actual system when the input is between 2.25 volts and 3 volts (between the third and fourth stairs). In the second graph of Figure 4.3, with smaller K and larger T, the model has better approximation to the actual system when the input is between 0 and 1.5 volts (the first stair). In the third graph of Figure 4.3, with K using the value between those used in first and second graph and T using the value from second graph, the model has good approximation to the actual system when the input is between 0 and 2.25 volts. If the window deposition side effect, which happens after a few minutes of deposition, is ignored, the model used in the third graph has good approximation to the actual system with the largest range: between 0 and 3 volts (first to the last stair). It has been observed that the least-square time constant T tends to be underestimated (low) when the y(k) data is noisy and a small number of data points are used [8].

Thus not counting the window deposition side effect and the time delay in response in low range, the system process model is:

In continuous-time domain:
$$G(s) = \frac{0.32}{1+6s}$$
 (4.10)

In discrete-time domain (from equation (4.7)):

$$a = -e^{-T/h} = -e^{-6/h}, \ b = K(1+a) = 0.32(1-e^{-6/h})$$
 (4.11)

where h is the system sampling time.

4.3 System Sampling Time

The sampling time of the control system was determined by two major factors: system limitation and performance requirement.

The sampling time for collecting CVD reaction IR spectrum is not directly configurable. Instead, the CAAP Frame software configures how many scans it takes to obtain a sample. By putting a time stamp to each of the sample data, which has the accuracy up to one second, the average sampling time can be calculated. The sampling time can be considered constant, thus base on the time stamps, the average sampling time is 3.3 second for the open-loop tests. Generally speaking, the more scans it takes, the more accurate the data are and the longer the sampling time is. The range of the sampling time during the Xylaur project was between 3 seconds and 8 seconds. Based on the information provided by Xylaur staff, to maintain a reasonable level of accuracy of the data, the sampling time should not be less than 3 seconds.

The sampling time for the CMU software on control PC can be set at any value with the accuracy up to 1 millisecond. However, the actual sampling time is also limited by the complexity of the running flow codes. The tests showed that one execution cycle of the main flow code would take at least 1.5 seconds. Therefore, the shortest sampling time for the CMU software is 1.5 second. Since there is always performance fluctuation of the operating system, there should be some room left in an execution cycle. Thus, the sampling time was set to be 2 second.

According to Shannon's sampling theorem, the sampling frequency of the

system should be at least twice the highest frequency component of the continuous system. In practice, the minimum sampling frequency can be set to the cutoff frequency of the system. The cut-off frequency is defined as the frequency where

the magnitude ratio of a system's frequency response is $\frac{\sqrt{2}}{2}$ of the value where the

frequency is 0. For a first order system $G(s) = \frac{K}{1+Ts}$ (K>0), the magnitude ratio of

its frequency response is $|G(jw)| = \frac{K}{|1 + T(jw)|}$, where w = 2pf. Since

|G(j0)| = K, thus for cutoff frequency f_c there is $|1 + T(jw)|^2 = 1 + (wT)^2 = 2$, so

$$f_c = \frac{1}{2pT}$$
. From (4.10), T=6, so $f_c = \frac{1}{2pT} = 0.0265$ second⁻¹. So the minimum

sampling frequency should be $f_{\min} = 2f_c = 0.053$ second⁻¹, thus the sampling time

$$h_{\text{max}} = \frac{1}{f_{\text{min}}} = 18.87$$
 second. In practice, h is usually much smaller than h_{max} is. As

rule of thumb, h can be chosen in the following way [7]: Define rise time T_r as the time required for the system response to a unit step input to rise from 10 to 90 percent of its final value, $N_r = \frac{T_r}{h}$ as the number of sampling periods per rise time, then h should be chosen in such a way that $N_r \approx 4-10$ (4.12). The solution to equation (4.5) is $y(t) = K(1 - e^{-t/T})u(t)$ where u(t) is the step input. Let t_0 is the time when the output is the 10 percent of the final value, t_1 is the time when the output is the 10 percent of the final value, thus $T_r = t_1 - t_0 \approx 2.20T = 13.2$ second.

Thus from (4.12), there is h = 1.3 - 3.4 second. In our system, the sampling time for spectrum data is 3.3 second while the sampling time for other process variables are 2 second, thus both are within recommended range. The sampling of spectrum data and other process variables were done independently and there was no efficient way to synchronize the sampling, so it was not proper to use the same value of sampling time for both system and declare that is the sampling time of the control system. Thus, the system was a mixed sampling rate computer-controlled system.

4.4 Controller Design

The general procedure of designing a controller is: 1.Obtain the design specification; 2.Choose the controller structure; 3.Obtain the parameter values of the controller; 4.Simulate the system with the controller; 5.Implement the controller; 6.Refine the controller if necessary. The procedure is an iterative process.

4.4.1 Design Specification

The main objective of the Control Project was to prove the feasibility of using *in situ* IR data for real-time control of the absorbance at specific area in the IR spectrum. It was not necessary to design a sophisticated controller for the closed-loop system. Thus, the specification for the closed-loop system with the controller was simple: 1) the system should be stable; 2) the steady-state error for step response should be zero; and 3) the settling time for step response should be less or

equal to that of the open-loop system without the controller.

For this system, the settling time T_s was defined as the time required for the response to a unit step input to reach and remain within 5 percent of its final value. The T_s for the open-loop system (in continuous-time domain) without the controller was obtained in this way: The solution to equation (4.5) is $y(t) = K(1 - e^{-t/T})u(t)$, where u(t) is unit step input. The solution indicates the system has no overshoot in the step input response. Thus, based on previous definition of T_s , at T_s there is $e^{-T_s/T} = 0.05$, hence $T_s = -T \ln(0.05) \approx 18 \sec(4.14)$. So the T_s of the closed-loop system with the controller should be less or equal to 18 seconds.

4.4.2 Controller Structure and Parameters

PID (proportional, integral and derivative) controller and its variation are among the most widely used controllers. They are easy to implement while still powerful enough to meet the requirement of many systems. The open-loop test data indicated that, assuming the system is a first-order system, the measured output data were noisy. For a noisy system, it is common to remove the D (derivative) component from the controller. Thus, the controller used for the system was a PI controller.

For continuous-time system, a typical PI controller can be expressed as:

$$u(t) = K(e(t) + \frac{1}{T_i} \int^t e(t)dt)$$
, where $u(t)$ is the output of the controller, $e(t)$ is the

input of the controller and is the difference between the set point and the process

output (measured variable). Since the system was a computer-controlled system, the PI controller was implemented in the form of difference equation. The implementation details are further discussed in section 4.5 *Controller Implementation*. Now that the controller structure is determined, the closed-loop system can be expressed using a Simulink model:



Figure 4.4 Closed-loop System with PI Controller

In this model, the sampling time for the integrator and step input was 2 second. The integrator used Backward Euler method: u(k)=u(k-1)+h*e(k), where u(k) is the integrator output, e(k) is the difference between set point and process output, h is the sampling time and its Z-transform function is $\frac{hZ}{Z-1}$, where h is the sampling time. The saturation block is used to limit the output of the controller according to the analog output board's limitation, which is between 0 and 4.99 volts. The uniform random number block is used to simulate the measurement noise of the system. Its output is between -0.05 and 0.05.

One basic assumption of conventional discrete-system design methods, such as

nyquist design, root-locus design, etc., is the system has one sampling rate or at least one sampling rate for a control loop (multi-rate sampling system), while the control system in this project had different sampling time within one control loop. This invalidated directly applying of conventional design methods to the controller design. The approach taken in the controller design of this system was trial and error through simulation: using different values of *K* and T_i during the simulation until the simulation response met the system specification. The result was:

 $K = 2, T_i = 5$.



Figure 4.5 Simulation of Closed-loop System Unit Step Response #1

Figure 4.5 is the simulation of the system unit step response with measurement noise, while Figure 4.6 is the simulation of the system unit step response without measurement noise. Both simulation show that the steady-state error of the system response is 0 and the settling time T_s is less than 18 seconds.



Figure 4.5 Simulation of Closed-loop System Unit Step Response #2 Because of the mixed sampling rate, the system is a nonlinear system. The stability criterion for linear system cannot be applied to this system. Lyapunov stability theorem provides stability criterion for nonlinear system [7]. The most important and difficult step of applying this method is to find a Lyapunov function for the nonlinear system. In this project, the simulation method was chosen to study the stability of the system. The approach was: In the system model, replace the unit step input with uniform random number, which generates outputs between 0 and 2 (the normal range of system input is between 0 and 1). The range of measurement noise output is set to be between –0.1 and 0.1 (the range in previous simulation is between –0.05 and 0.05). Figure 4.7 shows the simulation result. Figure 4.7 shows that even under large amplitude of noisy input and measurement, the system still produces bounded output with bounded input. Thus, it is believed that the closedloop system with the controller is a stable system.



Figure 4.7 Stability Study

4.5 Controller Implementation

Based on the model shown in Figure 4.4 and the simulation result, the z-transform function of the PI controller can be expressed as:

$$\frac{U(z)}{E(z)} = K(1 + \frac{hz}{T_i(z-1)}) = 2(1 + \frac{0.4z}{z-1})$$
(4.15)

where h is the sampling time. Generally speaking, the larger the K in (4.15) is the faster the system responds to inputs yet the less stable the system is. The open-loop test shows that if the system is modeled as a first-order system then the measurement data are quite noisy. The data also shows that within different operation ranges the system has slightly different dynamics, which indicates the model is an approximation assuming that the system is linear throughout the system

operation range. Based on these observations, K was set to 1 instead of 2 in the

implementation. Thus, (4.15) should be rewritten as: $\frac{U(z)}{E(z)} = 1 + \frac{0.4z}{z-1}$.

Based on the z-transform function, the difference equation of the controller is:

$$u(k+1) = u(k) + 1.4e(k+1) - e(k)$$
(4.16)

Equation (4.16) can be written in another form:

$$u(k+1) = u(k) + \Delta u(k+1)$$
(4.17)

where $\Delta u(k+1) = u(k+1) - u(k) = 1.4e(k+1) - e(k)$. Equation (4.17) emphasizes the incremental nature of the controller. This is important for the system, because the system had two control modes: open-loop and closed-loop. When the system switched from open-loop mode to closed-loop mode, the controller was introduced. To guarantee "bumpless" switch, the controller should "remember" the previous output to the process. Equation (4.17) indicates that the controller has this capability.

A closed-loop experiment was done to verify the controller. The recipe was setup in such a way that: In the experiment, the deposition occurs for five minutes. The system was open-loop system before the deposition. After the deposition started, the system was still open-loop system and TEOS was set to 300 sccm (1.5 volts output) for 60 seconds. Then the system was switched to closed-loop system and the PI controller was introduced. The set point of Absorbance was then set to 0.8 for 80 seconds, 0.3 for 100 seconds and finally 0.6 for 60 seconds. Figure 4.8 shows the Absorbance data during the deposition:



Figure 4.8

In Figure 4.8, the solid straight line represents the set points of Absorbance, the curve represents the measured values of Absorbance. The left and right vertical dotted lines are two timelines. The system was closed-loop system with a controller between these two time lines. The system was open-loop system outside these two time lines. The sampling time of the Absorbance data is 2 second.

A simulation was done to study the accuracy of the system model including the controller. In the simulation, the system model has the same structure as that in Figure 4.4, K is 1 and Ti is 5, which were the same values in the implementation; the simulation time is 300 seconds. The values and duration of the set point is the same as that in the closed-loop test except for the first 60 seconds. In the test, the system is open-loop system during the first 60 seconds. In the simulation, to

simplify the model structure, the system is closed-loop throughout the period and the set point is 0.45 for first 60 seconds. The period we want to compare is between 60 and 300 second, so the system response during the first 60 seconds can be ignored. The maximum and minimum values of Uniform Random Number are the same as in Figure 4.4. The simulation result and the closed-loop test data are drawn together in Figure 4.9:



Figure 4.9 Simulation Data vs. Deposition Data

The time period of interest is the time between 60 and 300 seconds. In Figure 4.9, the dotted-line curve is the actual deposition data while the solid-line curve is the simulated data. The figure shows that the two curves are very close to each other. This indicates that the system process model is a good approximation to the actual system and the controller behaved as expected.

Chapter 5 Summary

In this Control Project, the absorbance at about 1117 cm^{-1} was chosen as the control variable and measured variable; the flow rate of nitrogen carrying TEOS was chosen as the manipulated variable. A process model, which uses the control variable as the output and the manipulated variable as the input, was obtained. The closed-loop experiment indicated that the process model was a good approximation to the actual system within normal operation range.

According to Xylaur scientist Dr. Tom Whidden, the absorbance at about 1117 cm^{-1} was correlated against deposition rate of the CVD reaction. Therefore, the successful experiment in the Control Project provided suggests that it is feasible to use *in situ* infrared spectrum for real-time process control of the CVD reaction. Thus, it can be said that the objective of the first phase of Xylaur project has been accomplished.

Two major aspects of the control system need to be improved: First, the sampling of infrared spectra and other process variables were not synchronized, which led to mixed sampling time of the system. This caused difficulty to analyzing the system and designing the controller. Secondly, the accuracy of the measured data needs to be improved. The experiments and work from other groups of the Xylaur project suggests that the FT-IR spectrometer configuration needs further modification or re-design to effectively obtain the spectrum data of interest [6]. Both issues needed to be solved to design a more sophisticated and efficient

controller.

The first phase of Xylaur project has been completed. The next phase will be to find an industrial beta site to further explore using *in situ* infrared spectrum data for real-time process control of CVD reaction. The type of reaction process and system configuration might be changed. Thus, the process model and controller will be redesigned. One possible path of the project in terms of control system design would be to select absorbance at multiple bands in the infrared spectrum, which has close correlation with the CVD product quality, as control variables and multiple process variables as manipulated variables. In other words, a more sophisticated MIMO (multiple input multiple output) system might be developed.

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Appendix A: Data Acquisition Boards Specification (excerpted from Intelligent Instrumentä Product Catalog)

PCI-20428W-1 (for analog inputs)

PCI-20428W SERIES FEATURES & BENEFITS

- Waveform Generation, Transient Capture Capabilities
- Ideal for OEM Applications
- Completely Software Programmable
- 100kHz Multi-channel Analog I/O Throughput
- 16SE/8Diff 12-Bit Analog Inputs
- 2 12-Bit Analog Outputs
- DMA Transfer to/from Host Memory
- 8 Digital Inputs, 8 Digital Outputs
- One 16-Bit Counter
- Supported by Visual Designer™ Application Generator Software

PCI-20428W-1 HARDWARE SPECIFICATIONS

PARAMETER	CONDITIONS	SPECIFICATION
Analog Input		
Number of Channels, Resolution	Single-ended/Differential	16/8, 12 bits (1 part in 4096)
Voltage Ranges		0-5V, 0-10V, ±5V, ±10V
Software Programmable Gains	PCI-20428W-1	1, 10, 100
Over Voltage Protection	Power on/Power Off	±35V / ±20V
Common Mode Rejection	60Hz, 100 ohm imbalance, Gain = 100	-92dB (0.05lsb/V)
Linearity Error		±0.024%FS
Throughput, Multichannel	DMA, Triggered DMA	100KHz
Analog Output (PCI-20428W-1, -2)		
Number of Channels, Resolution		2, 12 Bits (1 part in 4096)
Voltage Ranges		0-10V, 5V, 10V
Throughput	DMA	100kHz
Digital I/O		
Number of Channels, Levels	Programmable	8 Inputs, 8 Outputs, TTL
Counters		
Number of Channels, Levels	Maximum	1, TTL
Clock Speed		16MHz
Rate Generators		
Number, Resolution		2 (1 analog input, 1 analog
		output), 125nS
Range		0.002Hz to 2MHz
Level		TTL

PCI-20093W-1 (8-CHANNEL ANALOG OUTPUT BOARD)

PCI-20093W-1 FEATURES & BENEFITS

- Waveform Generation, Simulation, Control
- Completely Software Programmable
- 8 Analog Outputs, 12-Bit Resolution
- Output Ranges of ±2.5V, ±5V and 0-5V
- Supported by Visual DesignerTM Application Generator Software

NARRATIVE DESCRIPTION

The PCI-20093W-1 is an analog output board featuring eight channels of voltage or current output. It plugs directly into one of the expansion slots in an ISA or EISA computer.

PCI-20093W-1 HARDWARE SPECIFICATIONS

PARAMETER	CONDITIONS	SPECIFICATION
Analog Output		
Number of Channels		8
Resolution		12 Bits (1 part in 4096)
Voltage Ranges		±2.5V, ±5V, 0-5V
Current Ranges	Jumper Selectable	0-20mA
Throughput		2000 outputs/second per channel
Accuracy		
Linearity Error	Voltage ranges	±1/2LSB
	Current range	±1LSB
Offset Voltage	Voltage and current ranges	±1LSB
Physical Characteristics		
Power Requirements	Vin = +5V	1.1A maximum
	Vin = +12V	45mA maximum
	Vin = -12V	50mA maximum
Size	Length x Height	7.2" x 4.2" (18.3cm x 10.7cm)

PCI-20087W-1 (DIGITAL I/O BOARD)

PCI-20087W FEATURES & BENEFITS

- 40 Digital I/O Channels with TTL-Compatible Levels
- Programmable as Inputs or Outputs in 8-Bit Ports
- Software Programmable; No Jumpers
- High Current-Sink (24mA) and Source (15mA) Capability
- Synchronization/Handshaking Capability
- Supported by Visual Designer[™] Application Generator Software

PCI-20087W HARDWARE SPECIFICATIONS

PARAMETER	CONDITIONS	SPECIFICATION
Digital I/O		
Number of Channels, Levels	Programmable	40 Channels (32 full-buffered), TTL
Output Levels		
Source Current	Vout=high	15mA
Sink Current	Vout =low	24mA
Interrupts		
Sources		Up to 3
PC Levels		2 through 7
Physical Characteristics		
Power Requirements	Vin = +5V	425mA maximum
Size	Length x Height	5.0" x 4.2" (12.7cm x 10.7cm)

Appendix B: Contents on the Floppy Disk

- Visual Designer diagrams and flow codes for the Control Project
- Data obtained from the open-loop test and closed-loop test
- Matlab m-files and Simulink models for system identification, simulation and other tasks