UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

AD-A208 861

1a. REPORT SECURITY CLASSIFICATION: UNCLASSIFIED	1b. RESTRICTIVE MARKINGS
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT: Approved for public release; distribution is unlimited.
4. PERFORMING ORGANIZATION REPORT NUMBER(S)	5. MONITORING ORGANIZATION REPORT NUMBER(S): AFOSR-TR-
6a. NAME OF PERFORMING ORGANIZATION: Univ of Washington	6b. OFFICE SYMBOL (If applicable)
7a. NAME OF MONITORING ORGANIZATION: AFOSR/NP	
6c. ADDRESS (City, State, and ZIP Code): Seattle, WA 98195	7b. ADDRESS (City, State, and ZIP Code): Building 410, Bolling AFB DC 20332-6448
8a. NAME OF FUNDING/SPONSORING ORGANIZATION: AFOSR	8b. OFFICE SYMBOL (If applicable): NP
9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER: AFOSR-87-0034	
8c. ADDRESS (City, State, and ZIP Code): Building 410, Bolling AFB DC 20332-6448	10. SOURCE OF FUNDING NUMBERS

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.
61102F	2917	A6	

11. TITLE (Include Security Classification)

(U) MULTILAYERED ELECTRONIC MATERIALS AND DEVICES BASED ON III-V COMPOUNDS

12. PERSONAL AUTHOR(S)
Dr Larry C. Olsen

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
FINAL	FROM 1 Oct 86 TO 30 Sep 88	December 1988	12

16. SUPPLEMENTARY NOTATION

17. COSATI CODES		
FIELD	GROUP	SUB-GROUP
	20.12	

18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

The objectives of this program were to purchase and install a Metal Organic Chemical Vapor Deposition (MOCVD) system in the Electronic Materials Laboratory at the Tri-Cities University Center of the University of Washington. This system would be used to grow films of III-V compounds. In order to install the MOCVD system, it was necessary to modify the facilities so that the system could be operated safely. Facility modification was done with University of Washington funds.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT: ☒ UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT ☐ DTIC USERS	21. ABSTRACT SECURITY CLASSIFICATION: UNCLASSIFIED
22a. NAME OF RESPONSIBLE INDIVIDUAL: Lt. Col. B. L. Smith	22b. TELEPHONE (Include Area Code): (202) 767-4908
22c. OFFICE SYMBOL: AFOSR/NP	

DD FORM 1473, 84 MAR — 83 APR edition may be used until exhausted. All other editions are obsolete.

SECURITY CLASSIFICATION OF THIS PAGE

89 6 02 034

UNCLASSIFIED

1. INTRODUCTION

The objectives of this program were to purchase and install a Metal Organic Chemical Vapor Deposition (MOCVD) system in the Electronic Materials Laboratory at the Tri-Cities University Center of the University of Washington. This system would be used to grow films of III-V compounds. In order to install the MOCVD system, it was necessary to modify the facilities so that the system could be operated safely. Facility modification was done with University of Washington funds.

The grant was awarded under the Department of Defense-University Research Instrumentation Program for FY87. Beginning in October, 1986, specifications for the MOCVD reactor were formulated. Requests for bids were sent to potential vendors in January, 1987. Spire Corporation was selected in early March, 1987, to provide a MOCVD system designated as the Model 500XT. Facility modification efforts also began in March, 1987. This process involved engineering design and creation of architectural drawings. Facility modification did not actually begin until February, 1988. The work was completed in July, 1988. Installation of the Spire 500XT system occurred during the months of July, August and September, 1988. By September 30, 1988, the system was ready for film growth. Actual film growth had to wait until new research contracts were received to provide funds for source gases, GaAs wafers for substrates, and labor required to operate the system. Before shipping the MOCVD system, Spire grew GaAs films with the system delivered to TUC. Results obtained by Spire are discussed in Section 3.

2. UW/TUC MOCVD SYSTEM

The MOCVD system purchased from Spire Corporation was designed to grow exceptionally well controlled III-V compounds in a research environment. This reactor will be used to grow layered structures based on GaAs, $Al_xGa_{1-x}As$, $In_yGa_{1-y}As$ or $GaAs_xP_{1-x}$. A picture of the reactor is shown in Figure 1. Other components of the system not visible in the picture include an RF power supply, two three-compartment gas cabinets, a pumping module, and the exhaust treatment facilities. These items are located in a chase adjacent to the Electronic Materials Laboratory, but outside of the building as shown in Figure 2. Also shown in this figure is the arrangement of the Electronic Materials Laboratory, and the location of the MOCVD reactor.

The 500XT is a more compact yet enhanced successor to Spire's successful 450 series reactor. It features four metalorganic (MO) and five hydride channels, all injected directly into the vertical barrel reaction chamber by a radial manifold operating on the run/vent principle (see Figure 3 for schematic diagram). Each MO consists of a temperature controlled bubbler fed with palladium-diffused hydrogen and provided with a separate hydrogen boost line to speed reactant transport, and to prevent condensation in the piping. Each bubbler is independently pressure controlled during low pressure operation. Two hydride lines are designed for arsine and phosphine, and include liquid metal purifiers to scrub water and oxygen from the feed gas. The remaining three hydride lines are intended for dopants and are therefore equipped with dilution networks which allow controllable flows ranging from .05 sccm to 200 sccm.

The silicon carbide coated graphite susceptor is RF heated and can be set to any temperature in the range 200-1000°C. Five two-inch or three three-inch wafers can be processed at one time. A ferrofluidic seal transmits rotary motion to the susceptor which can turn at 0-10 rpm in either direction or in an oscillatory manner. The reaction vessel is made of double wall quartz and is cooled by water between the walls to minimize parasitic reactions. Both ends are sealed by double concentric O-rings and the space between the O-rings can be evaluated by a quadrupole mass spectrometer to detect leaks in the seals.

Low pressure processing allows greater purity and more abrupt interfaces. The 500XT can grow films in the pressure range of 10 torr to atmospheric pressure. It is also equipped with a turbomolecular pump that will initially be used for system cleaning and substrate conditioning, but which eventually could allow chemical beam epitaxy at much lower pressures. A second turbomolecular pump in the quadrupole mass spectrometer allows sampling of atmospheric pressure gas streams including the various feed gases or the reactor exhaust, thereby characterizing impurities and reactor efficiencies.

Particulates on the substrates will be minimized by cleaning the reactor vessel after each use to minimize arsenic buildup (which readily flakes). The particulate problem is further minimized because the reaction compartment is built as a glovebox with an airlock for transferring wafers, and air is supplied through a HEPA filter.

An IBM AT computer with a Siemens controller, a hard disk, floppy disk, printer, and two video displays, controls all reactor functions. Over a thousand growth steps are allowed, with each step defining gas flows (to both run and vent), susceptor temperature, chamber pressure, susceptor rotation, and time. Temperature, flows, and pressure can be ramped linearly or functionally. The video screens report actual values of all parameters, and the computer can log these values to memory. These features will be very useful for growing strained layer super lattices.

The computer also monitors many safety interlocks and can warn the operator of a questionable condition or automatically shut down the reactor into a safe mode in the case of a serious condition. This shutdown does not depend on house electricity, so it can proceed even during a complete power failure.

Two features are specifically incorporated for indium compound growth. A heated gas line between the indium bubbler and the reactor chamber is necessary to prevent condensation of the power vapor pressure of indium metalorganic compounds. The pre-reaction problem mentioned earlier is reduced by the radial injection manifold which eliminates contact between the indium compound and the hydrides until they enter the reaction chamber. It is reduced still more by transporting the indium with a gas such as argon which has a larger molar specific heat than hydrogen. This cools the gas approaching the susceptor, inhibiting reactions. The 500XT has the capability to use such an alternate carrier gas.

Most MOCVD systems, including the 500XT, are constructed of stainless steel and quartz, materials with a strong affinity for a monolayer of water. A single exposure to atmospheric water vapor can result in a long period of slow desorption of water into the reactor atmosphere. To combat this problem, the 500XT is constructed with high vacuum components. It is anticipated that routine use will utilize the turbomolecular pump to evacuate to 10^{-6} torr with a hot reaction chamber prior to every growth run. Furthermore, periodic cleaning will include evacuating all gas supply lines while applying external heat. Elimination of water by these tactics will increase the electronic quality of the films.

As indicated by the above discussion, the TUC MOCVD system has many unique features. Some of these features are listed in Table 1.

3. INSTALLATION AND CHECK-OUT OF SYSTEM

Installation of the several major components of the MOCVD system was performed in July and August of 1988. These components are: the main reactor, module, two compressed gas cylinder cabinets, the vacuum pump and waste treatment module, the radio frequency power supply, the hydrogen purifier, and the toxic gas monitor. As shown on Figure 2, the RF generator, pumping module, and gas cabinets were placed in the new service chase. The reactor module and hydrogen purifier were installed in the Fabrication Lab. The toxic gas monitor has units in both the Fabrication Lab (Lab 3) and the Characterization Lab (Lab 4) and the service chase, with the the main control in Lab 4.

The high purity gas plumbing required to connect the various components were formed and welded by two Spire technicians, chempolished by TUC personnel, and installed by the Spire technicians in cooperation with TUC personnel. All of this plumbing is 1/4" stainless steel tubing with welded VCR fittings. The lines installed are represented on Figure 3 as those joining the "Hydride Gas Cabinets" to the "Hydride Flow Control", all lines from the "Palladium Hydrogen Purifier", and the line from the "Liquid Nitrogen Boiler". The Spire technicians spent one full week at TUC.

Exhaust plumbing was fabricated in a local machine shop and installed by TUC personnel. Stainless steel tubing of 1/2", 3/4", and 1 1/2" sizes was used for the various exhaust lines, welded to quick-flange fittings. These lines join the main reactor module to the pump module in the service chase.

Helium leak testing of all gas plumbing within the various components and the new plumbing linking the components commenced with the assistance of Spire's installation engineer. He also installed the reaction chamber pieces, including susceptor supports, susceptor, thermocouple, and the bell jar and RF coil. All electronic sensors were checked and the mass flow controllers zeroed. Inert gas flows were initiated to test pressure and flow control, and to familiarize the TUC operators with the system.

After all systems were operating satisfactorily, the RF generator was turned on to heat the susceptor in an inert gas atmosphere. Since no problems developed, heating in hydrogen was performed. Finally, the first two metalorganic sources, trimethylgallium and trimethylaluminum, were loaded into their refrigerated baths and the MO plumbing was purged. Arsine, silane, and dimethylzinc compressed gas cylinders were placed in the gas cabinets and the plumbing was purged. Arsine and hydrogen were flowed through the system at room temperature, testing the toxic waste treatment system. At this point, the system was ready for film growth.

4. FILM GROWTH RESULTS

Prior to shipping the MOCVD system, Spire personnel grew GaAs films on GaAs substrates with the TUC 500XT reactor. Excellent results were obtained. Films grown on two-inch wafers exhibited a thickness uniformity of better than 2% and a doping uniformity better than 4.5%. With three-inch wafers, films were characterized by a thickness uniformity of 4% and doping uniformity of 8%. Film structures were typically characterized by a low density of interface states at the substrate/epi-layer interface. They also found that the AlGaAs composition uniformity was better than 2% for AlGaAs films grown on two-inch wafers. Finally, the background doping in epitaxial GaAs films grown with the TUC reactor was determined to be less than $1.5 \times 10^{14} cm^{-3}$. These performance figures represent a significant improvement in GaAs epitaxial film growth. The results obtained by Spire personnel with the TUC reactor are summarized in Table 2.

5. FUTURE WORK

The TUC MOCVD system will be utilized during FY89 to conduct an AFOSR program concerning high efficiency monolithic, multibandgap solar cells. The planned two-year effort will involve investigations of a monolithic three-cell stack based on an AlGaAs cell (E_g = 2.0 eV), a GaAs cell (E_g = 1.42 eV) and an InGaAs cell (E_g = 1.03 eV). Research will focus on the growth and characterization of a two-cell combination of AlGaAs/GaAs on top of an InGaAs cell. The potential efficiency of the three-cell system is greater than 35% (AMO).

Other research efforts will also utilize the TUC MOCVD system. In addition to other photovoltaic research, we expect to initiate activities concerning photodetectors and light emitters utilized for optical communications.

TABLE 1

FEATURES OF THE TUC MOCVD SYSTEM

1. The Spire 500XT is a compact new generation reactor evolved from Spire's Model 450.

2. Vertical barrel reaction chamber capable of processing five 2" wafers or three 3" wafers on a rotating susceptor.

3. Four metalorganic channels with separate controlled temperature baths for the MO bubblers and independent pressure control.

4. Five hydride channels.

5. Radial reactant injection manifold mounted directly on the reactor chamber.

6. Low pressure processing, and high vacuum capability (turbopumped) for cleaning, substrate pre-treatments, leak checking, and eventual vacuum MOCVD/chemical beam epitaxy.

7. IBM AT controlled processing utilizing two video displays for text and graphics. The computer allows constant, step, ramped , and functional growth profiles of both stoichiometry and doping.

8. Each MO channel has a hydrogen boost circuit for reactant dilution or to accelerate a low flow for improved interface sharpness. Two MO's can be supplied by an alternate carrier gas, and one has heated reactant plumbing (for growth of indium compounds).

9. Three hydride channels have hydrogen dilution networks allowing a concentration range of each gas of 1 to 1/2500.

10. Run-vent design for optimum stability and interface sharpness.

11. Palladium diffused hydrogen supply.

12. HEPA filter protected compartment for the reaction chamber.

13. RF heating.

14. Exhaust conditioning includes charcoal adsorption plus burn-off.

15. Thoroughly integrated safety interlocks and sensors.

16. Quadrupole mass spectrometer for leak checking, sampling of incoming reactants, and sampling exhaust.

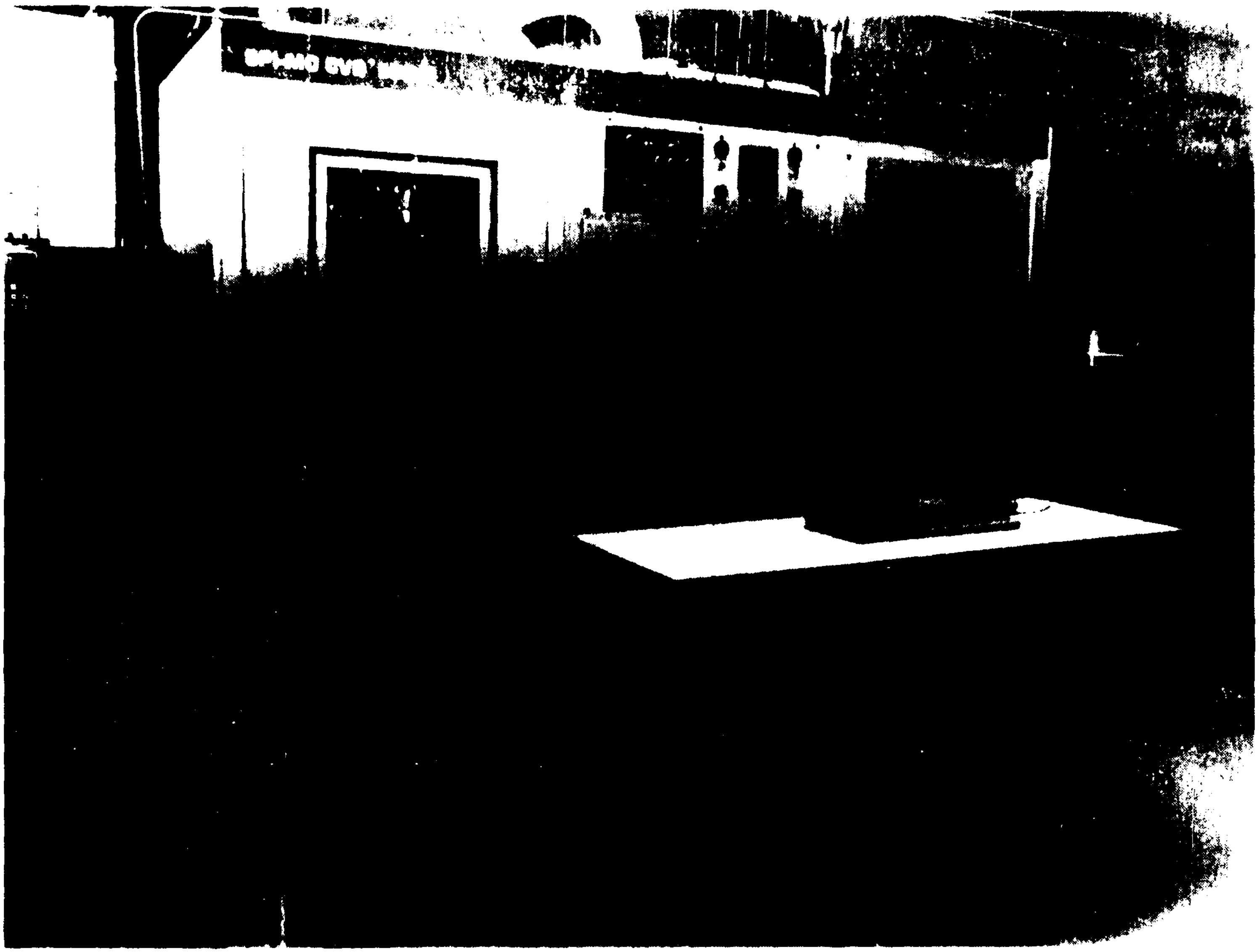


Figure 1. Spire 500XT MOCVD System Installed in the Electronics Materials Laboratory, Tri-Cities University Center.

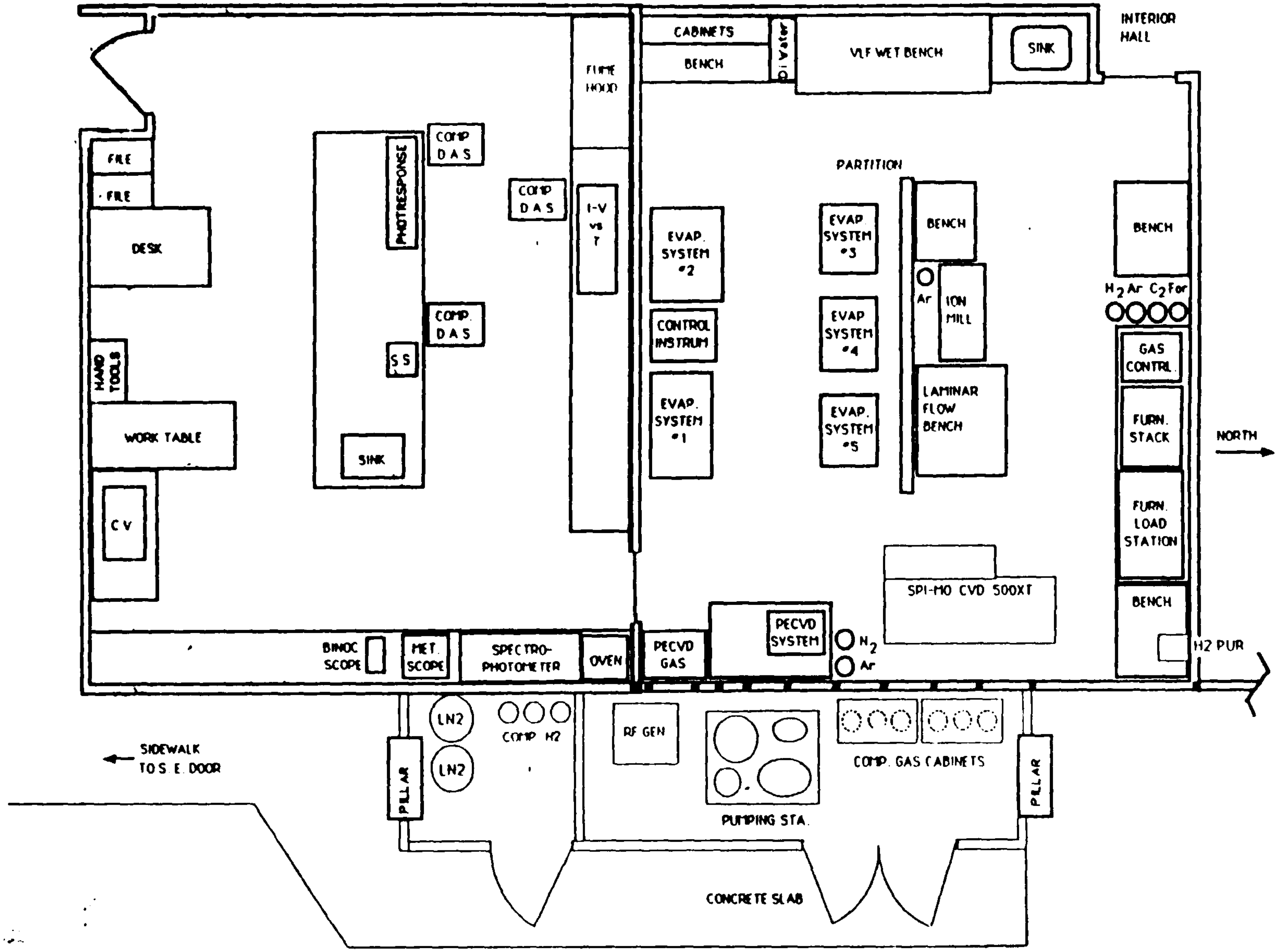
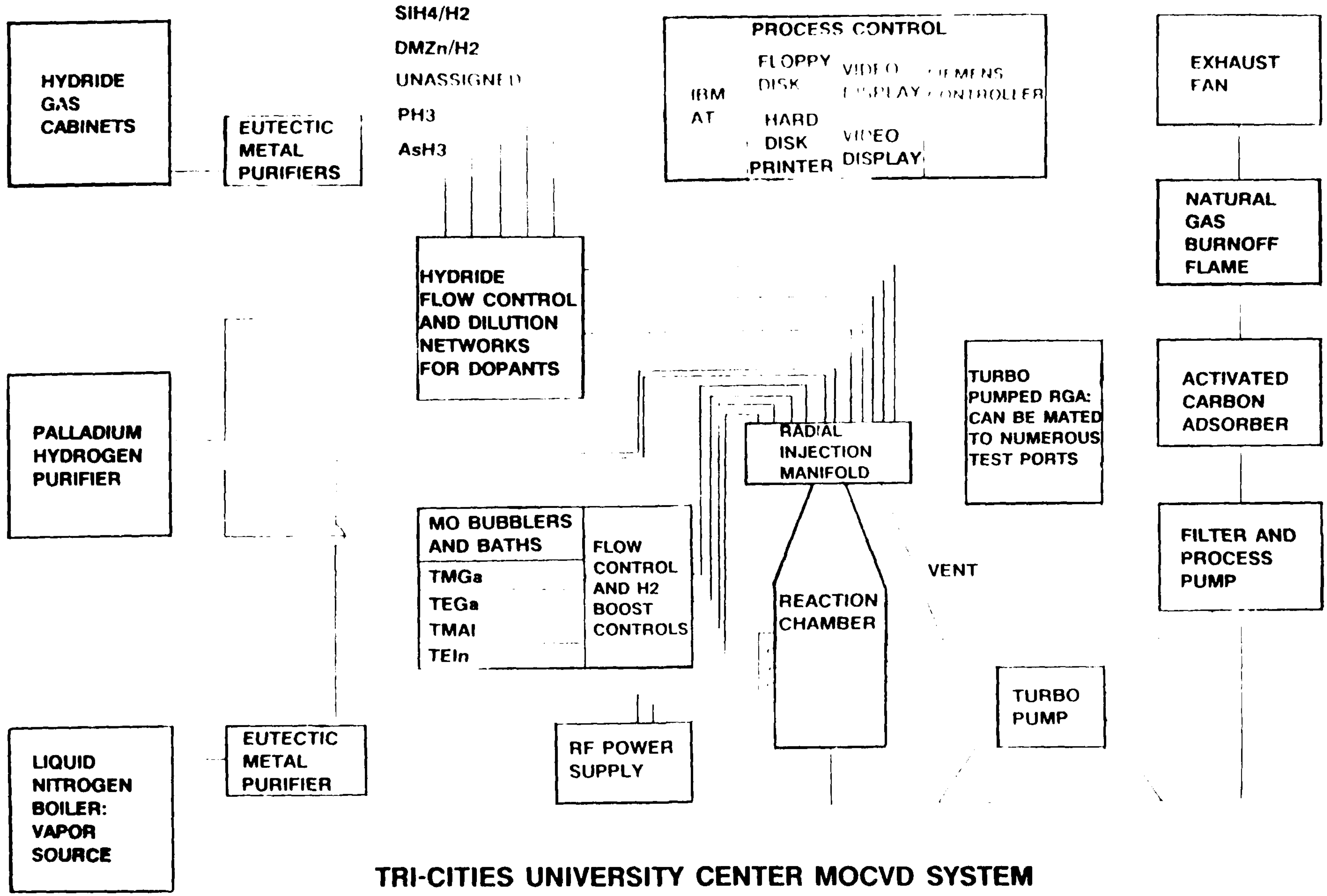


Figure 2. Electronic Materials Laboratory Arrangement. Photolithography Laboratory Not Shown.



TRI-CITIES UNIVERSITY CENTER MOCVD SYSTEM

SPIRE 500XT MOCVD REACTOR

Figure 3: Schematic Component Diagram for TUC MOCVD Reactor.