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APPENDIX

EXPERIMENTAL PROCEDURES*

The details of the system operation and maintainance are given in this section. These include the cleaning procedures for the various parts and the assembling of the latter, the start-up procedures and the mechanical operations such as valve open/close sequences. Some of these detailed operations may at first appear as black-art tedium but are actually based on sound scientific relationales. For example, the growth and/or air lock chamber after exposure to air would have to be evacuated and refilled with H_2 seven times.

Liquid Phase Epitaxy System.

1. Parts Cleaning Procedures.

a) Brass or Stainless Steel.

1) Clean with detergent and tap water to remove oil which occurred during machining.
2) Immerse in acetone to remove dirty water.
3) Immerse in methanol to remove acetone.
4) Immerse in deionized H_2O tank, and rinse with running deionized H_2O to remove meOH.
5) Dry the internal parts with N_2 (0.1 micron filter).
6) Dry with external heating (hair dryer).

Note that the DI water we have is the cleanest solvent and hence in use as the final rinse in all cleaning here and below.

* This section is written to serve also as an operating manual for posterity.

b) Quartz Parts.

1) Etch for 1 hour with 50% aqua regia.

1. part HNO_3 conc.

2. parts HCL conc.

3. parts deionized H_2O.

2) Immerse in deionized H_2O.

3) Rinse with deionized H_2O.

4) Dry with N_2 .

5) Dry with extenal heating.

c) Rubber Parts (O-Ring).

1) Cleaned with a solution of KOH 20% at 70 °C.

2) Rinse with deionized H_2O.

3) Dry with N_2 .

2. System Installation. the numbers in ( ) as indicated in Fig. A1.

1) Vertical centering of upper coupling(1), furnace(2) and lower coupling(3).

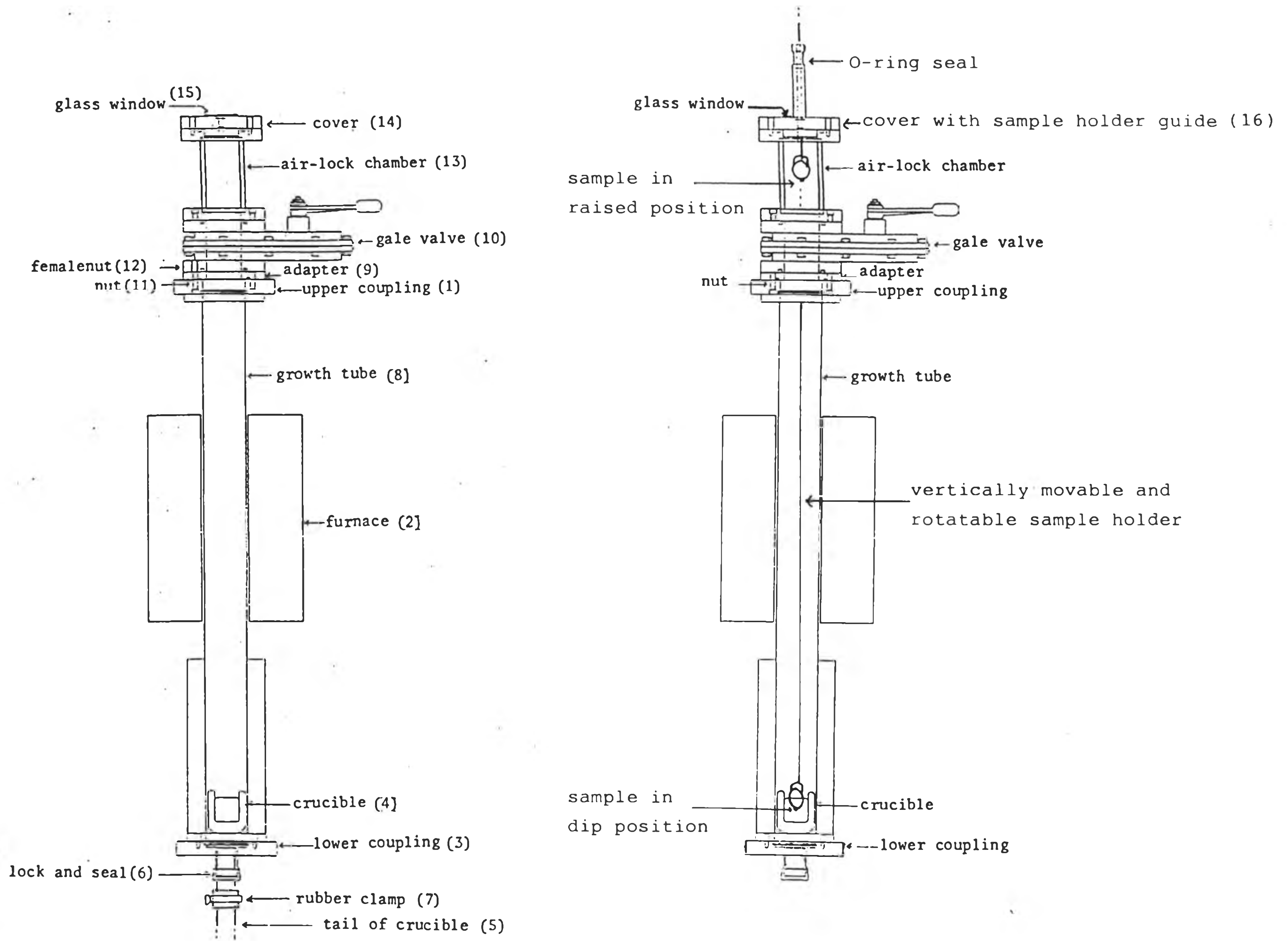

Fig.A1 Schematic of the growth part of VLPE system.

2) Insert crucible(4) through upper coupling, furnace, and insert the tail of crucible(5) up and into lock and seal(6) at crucible tail coupling.
3) Clamp the tail with rubber(7) for safety during subsequent pumping.
4) Insert growth tube(8) through upper coupling, furnace, crucible and carefully put the lower end of tube into lower coupling.
5) Put adaptor(9) the upper coupling.
6) Put the gate valve(10) on the adaptor and put six holes on the adaptor into female nuts(12) in the gate valve.
7) Place the air lock chamber(13) onto the gate valve and put six nuts through six holes on the chamber into female nuts in the gate valve.
8) Closed the chamber with the cover(14) which has the glass window(15) on the center.
9) Evacuate and refill with N_2 to a slight possitive pressure and check for leaks with detergent bubbles.
10) Connect the H_2 gas line from H_2 generator MARK V and purity again with H_2 purifier HP 25 to the system H_2 contains less than 1 ppm of oxygen.
11) Evacuate and refill with H_2 and check leak with detergent bubbles.
12) Evacuate and refill with H_2 overall seven times. If the "vacuum" state of the system is a fraction p of the operating pressure which is approximately one

atmosphere, then the seven cycles represent a reduction of contaminant gases by a factor p to the seventh power.

3. Hydrogen Generator Start Up Procedure.

1) Set the hydrogen pressure control knob to 60 psi.
2) Open the hydrogen output valve.
3) Connect the power cord (220 V from automatic voltage regulator).
4) Turn the POWER switch on.
5) A click will be heard as the solenoid valve opens a the MAXIMUM OUTPUT light will illuminate.
6) After approximately ten minutes, the hydrogen output will begin, and will increase to the flow rate of the cell over a period of time ranging from one-half to several hours.
7) Allow the Mark V to operate in this mode for at least 30, and preferably 60, minutes to throughly purge the system of air and to warm up the cell.
8) Then close the HYDROGEN output valve.
9) The MAXIMUM OUTPUT light will stay on for a few minutes until the pressure build up to the setting of the HYDROGEN PRESSURE control, then it will go out.

4. Hydrogen Purifier HP-25 Start UP Procedure.

We used H_2 from Hydrogen Generator Mark V as the input of Hydrogen Purifier HP-25, H_2 from the generator should be at 30-40 psi.

1) Adjust valves* in sequence indicated

Valve#	Position
vac	closed
H1	closed
10	closed
bleed valve	closed
1 and 4	closed
11	open
UPH	open

* Valve designations as in Fig. A2

2) Turn on vacuum pump power and adjust valves in sequence indicated

vac	open
(evacuate pass valve 11 and UPH)	
10	open slowly

3) Let stand to low pressure and adjust valves in sequence indicated

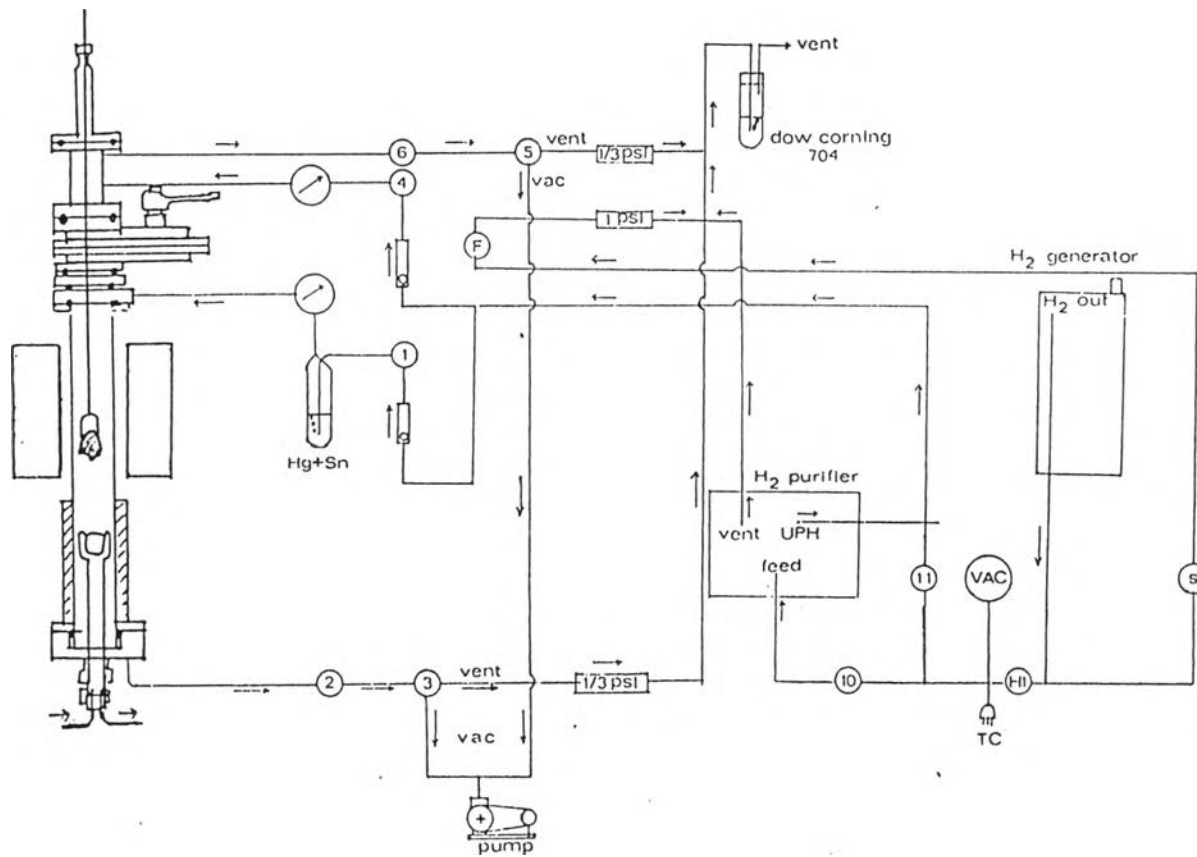

Fig. A2 Schematic diagram of the VLPE system

11	closed
10	clsoed

4) Open H_2 valve from the generator and then open H1 let H_2 go to vacuum pump and followed by closed VAC rapidly. Wait about 1 minute until the MAXIMUM OUTPUT light goes out.

5) Open 10 slowly to let H_2 go into inner cylinder of the palladium cell.

6) Allow the inner cylinder to fill (about 1 minute) and then use 11 to slowly let H_2 into the outer cylinder of the cell. The MAXIMUM OUTPUT light will go on. Wait until the light go out (about 5 minutes). Then perform the next step.

7) Adjust valves in sequence indicated

	Valve#	Position
	10	closed
	H1	closed
Note	11 and UPH	open
	VAC	open
	(evacuate outer cell)	
	10	open slowly
	(evacuate inner cell)	

8) Closed H_2 valve from generator. Let stand to low pressure. And then return to step 3).

9) Perform the step 3) through 7) five times. And the last time will stop at step 5). Then go to step 10) instead of 6)

10) Wait for about 5 minutes in order to let H_2 is filled up fully in the palladium cell. Then closed UPH flow.

11) Open bleed valve as small as the bleed indicator just indicates a small flow.

12) Connect the power cord (110 V from automatic voltage regulator). Turn power on and then set T_{min} ~680 F, T_{max} ~ 750 F.

13) The H_2 supply system is now ready. Vacuum pump and TC pressure gauge power supply are no longer necessary and can be turned off.

5. Loading Procedure and Heat Cleaning the Substrate.

At this point all valves should be in the position indicated.

Valve	Position
gate valve	closed
4	closed
5	closed
6	open
1	open
2	open
3	vent

Remove the blank cover (14) in Fig. A1 of the chamber and mount the sample holder cover (16) with the substrate holder into the chamber carefully. Tighten the six hex screws and then go through the following steps.

1) Turn on the vacuum pump power.
2) Valve # 5 is vac. position.
3) Wait until the pressure about - 31 in Hg.
4) Valve # 5 is closed.
5) Valve # 4 is opened.
6) Wait until the internal pressure comes up to a small positive pressure ($\sim$1/4 in Hg).
7) Turn valve # 5 from closed to vent position.
8) Repeat the step 2 (to step 7) seven times.
9) Open the gate valve.
10) The system is ready for loading the substrate into the growth tube.
11) The substrate is moved down to the middle of furnace position for heat cleaning at 475 $^{\circ}$C for 45 minutes to desorb the passivating oxide layer.

Unwaxing and Solvent Cleaning the Substrate.

The substrates used for all experiments were (111) InSb oriented less than $\pm 0.2^{\circ}$ off axis, 20 mils thickness, 3 cm. diameter, {111}B polished face (the other side lapped). Normally the wafer InSb was waxed from the supplier because it is very brittle. The wafer was unwaxed by putting the plastic tray containing the wafer on the

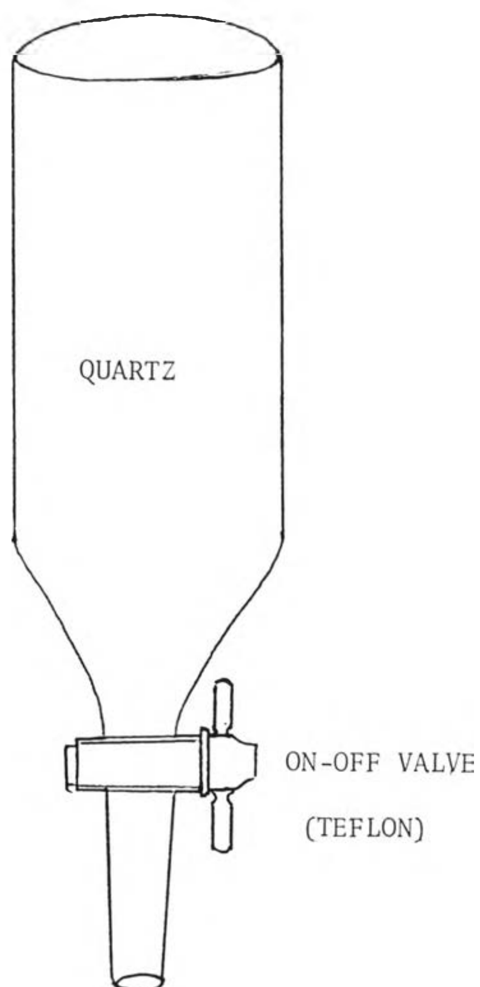

Fig.A3 Separating funnel for using in eleaning the wafer after unwaxing.

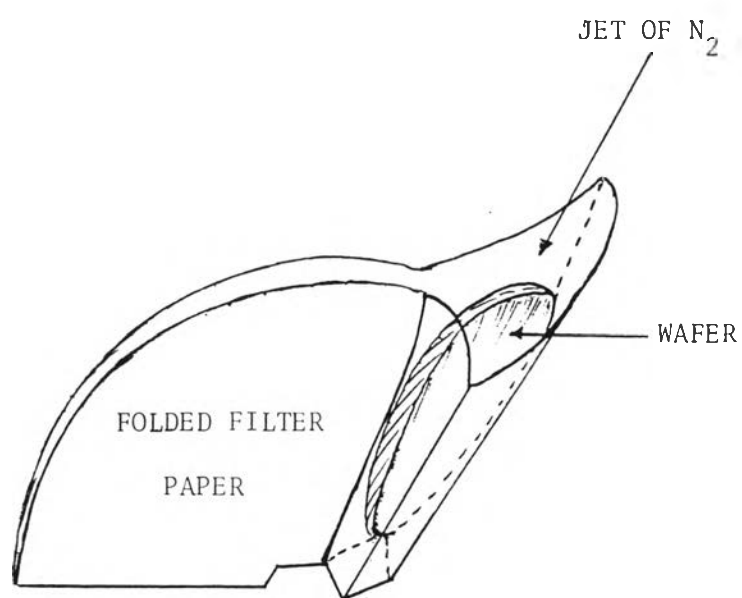

Fig.A4 Folded filter paper is contained with wafer for drying with filtered N_2 jet.

hotplate (65 °C). Observe until the wax was completely melted and the wafer was removed from the tray using a piece of clean filter paper. Put the wafer into the seperating funnel which contains solvent. The seperating funnel has a teflon on-off valve at the bottom for letting the solvent out, have shown in Fig. A3. Open the valve to let out the solvent very carefully. Do not let the upper level of the solvent decrease lower than the wafer; dry residue will result which cannot be removed. And refill with the solvent again. Repeat successively with TCE for 10 times, acetone 6 times, and MeOH 4 times, then with deionized H_2O (18 Megohm-cm resistivity of Millipore product) 10 times. Finally flushed with deionized H_2O for ten minutes. Tilt the seperating funnel until the wafer slides into the filter paper which is folded in the shape shown in the Fig. A4. And dry with the jet of N_2 (0.1 μ filter). The wafer is ready for chemical etching. For etching procedure see Chapter II of text.

BIOGRAPHY

Mr. Vittaya Amornkitbamrung was born on June 22, 1955 in Nongkhai. He received the B.Sc. and M.Sc. in Physics from Chulalongkorn University in 1977 and 1981 respectively. During studying for a Ph.D. degree, he received the Rachadapiseksompoj Research Fund from Chulalongkorn University in 1987. He is currently working on LPE for Physics Department, Faculty of Science, Khon Kaen University.