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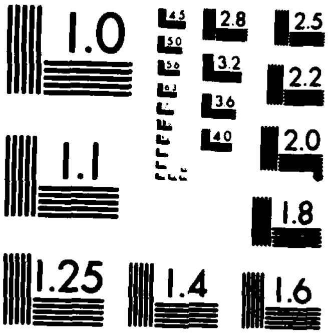

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DATE: April 28, 1987

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ABSTRACT

The construction of a novel type of plasma assisted chemical vapor deposition device for microelectronic applications has been completed. The machine is based on a flowing afterglow system in which the source generation plasma is physically decoupled from the deposition substrate. Because of the high gas throughput the machine can operate not only as a novel plasma assisted CVD region system, but as a physical deposition device as well. The combination of physical deposition capabilities, versatile plasma capabilities, and normal CVD capabilities suggests unusual flexibility in the production of high quality insulating films for indium phosphide based microelectronic structures. The achievement of high quality insulating layers on indium phosphide substrates could lead to the development of indium phosphide based metal-insulator-semiconductor (MIS) devices. The MIS structures might eventually be utilized for very high speed VLSI applications. The devices should be able to achieve operating speeds in the 100 GHZ region and beyond. Initial efforts have thus far proved unsuccessful in obtaining high quality films, although the system still shows great promise for circumventing the limitations of other deposition techniques.

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Chapter

I. Introduction

The problem of obtaining high quality insulating films on indium phosphide substrates is of considerable interest in the development of high frequency MISFET structures. A good deal of work has already been done in this area. Section III of this report provides a brief summary of the associated history and technology. In brief, a novel flowing afterglow deposition technique has been developed at Wayne State University in order to overcome the limitations associated with other approaches. The results and conclusions obtained with this technique are presented in Sections IV and V, respectively.

II. Objectives

A. Primary Goals

The research objectives are divided into several groups. The primary intention is to develop an ability to produce high quality insulating films on indium phosphide substrates. Associated with this intent is a series of smaller steps. First construction of the flowing afterglow system is to be completed. The operating parameters of the flowing afterglow system must then be explored in order to delineate the optimum regions for high quality film deposition. Since substrate cost is a factor, the choice of substrates is intended to progress in an orderly sequence from glass through crystalline silicon to indium phosphide.

B. Secondary Goals

At the same time that the system parameters are being explored, work will be conducted on the development of in situ diagnostic techniques. The primary emphasis of the diagnostic apparatus is intended to center on the deposition process itself. This includes the development of an in situ optical imaging technique that permits rapid analysis of the relatively complex combinations of system parameters. Following the successful growth of high quality oxide films on indium phosphide substrates, an attempt will be made to fabricate an entire MIS structure.

III. DISCUSSION

A. Introduction

The development of high frequency Field Effect Transistors (FET's) suitable for operation in the 100 GHz region and beyond is closely linked to the effort to develop stable, high-quality Metal-Insulator-Semiconductor (MIS) device structures. Although GaAs and InP both possess the high electron mobilities and saturation velocities necessary for high frequency operation, GaAs has proved unsuitable for MISFET structures primarily because of its inability to achieve low surface state densities in contact with grown or deposited insulating films.[1-3] GaAs FET's have consequently been restricted to the slower Metal-Semiconductor variety (MESFET's), while development efforts have continued on InP-based high frequency MISFET structures. Apart from the surface state density effect, the ultimate frequency limit is inherently higher in InP than in GaAs due to the higher possible saturated drift velocity limit.[1]

The problem of producing stable, high-quality dielectric interfaces on InP substrates has led to the development of a new and evolving materials technology. There has been a steady progression in oxide deposition techniques ranging from chemical vapor deposition[4-8] through plasma enhanced chemical vapor deposition[9,10] to indirect plasma enhanced chemical vapor deposition.[11] Although relatively low interface densities have been achieved, the problem of device stability still requires resolution and is critically dependent on the oxide preparation technique.[3]

A funded program is underway at Wayne State which is specifically geared towards the development of ultra-high-quality insulating films on indium phosphide substrates. The program is based on the development of a new evolutionary step in materials technology termed "Flowing Afterglow Chemical Vapor Deposition" (FACVD). This technique has the potential for circumventing the limitations of previous approaches. The FACVD process is a complicated mix of physical deposition, chemical vapor deposition, and plasma deposition. It is not simply a modified commercially available device. Each item for the machine has been and is being separately developed in a detailed and complicated construction process. The work has been supported for the past year by NSF and, in part, by the Rome Air Development Command. In that brief time the basic machine has been constructed and a plasma has been obtained. R. Arrathoon has just been awarded a $160,000 DOD equipment grant and another $31,000 equipment grant from the Provost's fund.

Architectural and construction projects are now under way at the expense of the College of Engineering to insure that the equipment will be safely housed and vented.

II. B. Present Status of InP-Dielectric Interfaces

The basic building block for MOS devices in LSI and VLSI circuits is the Si MISFET (metal-insulator-semiconductor field effect transistor). The insulator is an oxide layer which is relatively easy to fabricate by thermal oxidation or pyrolitic chemical vapor deposition (CVD) techniques. Pyrolitic CVD and thermal oxidation are relatively high temperature processes which are generally unsuitable for forming high-quality oxides on III-V compounds such as gallium arsenide or indium phosphide. Lorenzo[12] and others[13,14] have shown that thermally grown oxides on GaAs are poor in quality: they are neither stoichiometric nor spatially homogenous. One reason is that column V elements have a high vapor pressure with respect to column III elements. At temperatures above 350 °C the stochiometry of InP, for example, will degrade due to a relatively large loss of phosphorus; moreover, it is generally not desirable to process carefully doped VLSI circuitry at elevated temperatures. For very precisely controlled conditions, pyrolytic CVD with silane-oxygen mixtures can take place in the range 325-450 °C. Initial attempts to form oxide films on InP substrates employed this technique.[4-8] Later attempts to lower surface state densities and improve device stability centered on plasma deposition methods.

Low energy plasmas or gas discharges provide a mechanism for the formation of oxides at low atomic temperatures. The technique was first employed by Miles et al.[15] in 1963, and subsequently applied by Ligenza,[16] Swann et al.,[17] and others[18-21] to promote the low temperature oxidation of silicon. These early plasma systems yielded Si-SiO_2 interface state densities in the range 10^{11} to 10^{12} states/cm^2-ev. Post deposition annealing lowered the densities by roughly an order of magnitude[18,22] to values comparable to those obtained from the best thermally grown silicon oxides.[23,24]

In 1966 Weinrich[25] first applied the method to GaAs. Oxide films were deposited which were amorphous and good insulators; however, the deposition process was extremely slow. Attempts to speed up the deposition process resulted in the formation of polycrystalline gallium oxides. The insulation properties of polycrystalline materials in general are inferior to those of materials in the amorphous state. Fortunately, in the 1970's a good deal of work on amorphous silicon films (a-Si) in glow discharge systems[26] led to a considerable body of information on glow discharge amorphous film formation techniques. Drawing on this body of knowledge, in 1978 Sugano and coworkers[27] succeeded in avoiding the formation of polycrystalline gallium oxides and produced a moderately good amorphous film simply by adjusting the plasma parameters. In common with other glow discharge deposition techniques, however, reproducibility and film stability were erratic. Efforts continued[28-30] and some improvements were achieved, but surface state densities remained high,[1] resulting in the pinning of the Fermi level near the middle of the bandgap. By contrast, for InP interfaces the Fermi level can be displaced across the entire bandgap permitting the fabrication of depletion and enhancement mode transistors.[2]

The development of high-quality dielectric films on InP substrates has evolved through a number of stages. Pyrolytic CVD efforts have already been discussed. The next stage involved PECVD, which resulted in improved surface state densities and device stability.[9,10] In 1982 Meiners[11] published a paper demonstrating that an indirect plasma approach produced superior interface characteristics; nonetheless, interfacial trapping effects have remained a major problem with respect to long term device stability. All indications are that the magnitude of these effects is critically dependent on the details of the dielectric growth process.[3] Details of past techniques and the transition to the next evolutionary phase, the proposed FACVD approach, will be presented in Sections III. C and D.

III. C. Flowing Afterglow and Plasma Diagnostic Review

The flowing afterglow technique was originally developed in the early 1960's by Schmeltekopf et al.[31,32] for use in atomic and molecular collision studies involving reactions of atmospheric interest. The method provides a copious source of "cool" He^+ $(^2S_{1/2})$ singly ionized ground-state ions as well as "cool" He (2^3S) metastable atoms. By appropriate current adjustments, the ratio of these two species can be varied continuously. In the afterglow no other ionic or excited atomic species remain, and the electronic, ionic, and atomic velocities have slowed to the point where thermal equilibrium is established at or near room temperature. The process provides some special advantages over pulsed afterglow methods[33-36] for the study of relative collision cross sections. These advantages have been outlined previously in some detail,[37] and have led to the extensive use of flowing afterglows for the study of various laser related collision processes.[38-40] Among the processes which have been studied are:

1) Charge Transfer

$$He^+ + M \rightarrow He + (M^+)^* + \Delta E \qquad (1)$$

2) Resonant Transfer

$$He\ (2^3S) + Ne \rightarrow He + Ne\ (2s_2) + \Delta E \qquad (2)$$

3) Penning Ionization

$$He\ (2^3S) + M \rightarrow He + (M^+)^* + e + \Delta E \qquad (3)$$

4) Hybrid Penning Ionization

$$He^+ + M \rightarrow He + M^{++} + e + \Delta E \qquad (4)$$

5) Two Electron Penning Ionization

$$He\ (2^3S) + M \rightarrow He + M^{++} + 2e + \Delta E \quad . \qquad (5)$$

The particular advantages of flowing afterglow techniques with respect to pulsed afterglow methods may best be appreciated by briefly examining the details of the flowing afterglow apparatus. A typical system is illustrated in Fig. 1. The method involves the near sonic flow of a buffer gas down a long, wide bore, flow tube. Operating pressures from 100 microns to 10 torr are normally accessible. The buffer gas is strongly excited by means of an

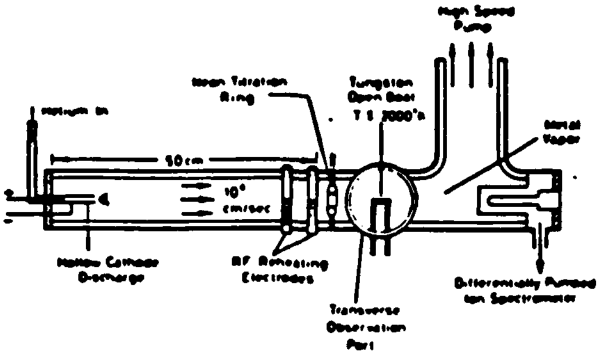


Fig. 1. Flowing Afterglow Apparatus (Ref. 37).

electrical discharge immediately upon entering one end of the flow tube. A hot plasma is formed with electron temperatures and number densities characteristic of a glow discharge.[41-45] The hot plasma is sucked away from the excitation region down the discharge tube. As the plasma travels down the tube, ambipolar diffusion and recombination result in a steady loss of ions until the plasma dissipates and eventually returns to a completely neutral state. At the source end of the discharge chamber there exists a hot plasma with a high electron temperature, a high electron-ion density, and a relatively long Debye length (a relatively unscreened plasma). At the other end of the discharge chamber, the plasma has cooled to room temperature and the electron and ion temperatures are identical. In this region the Debye length has shortened to the point where there is a high degree of screening. The various collision processes involved in the thermalization process are well known,[41-45] and the main ion loss mechanism in this type of system is ambipolar diffusion. Viewed transversely, the decaying plasma looks somewhat like a large diameter oxy-acetylene jet, with color and intensity dependent on the buffer gas and excitation conditions. The jet-like behavior of the plasma is due to the high velocity of the gas flow, since at velocities of order Mach 0.3 gases behave as incompressible fluids.[46] In effect, the flowing afterglow technique transforms the temporal distribution of the pulsed static afterglow into an equivalent spatial distribution. For the pulsed system, a given point in time can only be sampled periodically, whereas in the flowing system an equivalent temporal point may be examined continuously. The introduction of a substance into the flowstream at a particular point in space corresponds to the introduction of that substance at only one temporal point in the afterglow. The point of introduction of the injected substance is termed the reaction region, and materials with evaporation temperatures as high as 2000 $^{\circ}$K may be evaporated in the reaction region.[37] By contrast, the physical introduction of a foreign substance into a pulsed afterglow necessarily results in its presence for all time. Materials can not be injected arbitrarily into a pulsed afterglow without drastically altering the characteristics of the plasma. Further, the flowing afterglow method permits the use of various discriminant techniques such as current adjustment, rf reheating, and gas titration[37] to separate metastable collision effects from those of ions.

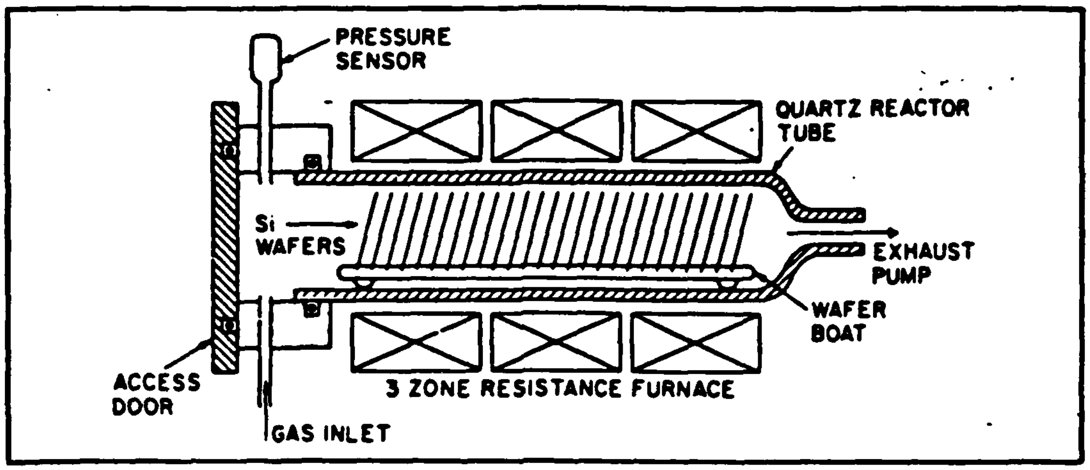
The plasma characteristics of flowing afterglows have been studied extensively using floating double probes[47,48] and cylindrical Langmuir probes[49,50] Optical spectroscopic measurements in conjunction with probe and microwave cut-off experiments have established that DC driven hollow cathode discharge systems[37-40] are capable of achieving afterglow electron number densities in the 10^{10} to 10^{13} cm^{-3} range and afterglow metastable densities approaching 10^{11} cm^{-3}. This was found to be a considerable improvement over earlier systems which were excited with microwave sources.[31,32] In practice the point of introduction of the foreign substance in equivalent time can be changed rather easily by throttling down the flow rate. This produces variable velocities in the flow stream.

Since the Principal Investigator is intimately familiar with flowing afterglows, it may be presumed that he will not need to spend an excessive amount of time on plasma and spectroscopic preliminaries before concentrating on the main problem of developing the flowing afterglow chemical vapor deposition (FACVD) apparatus.

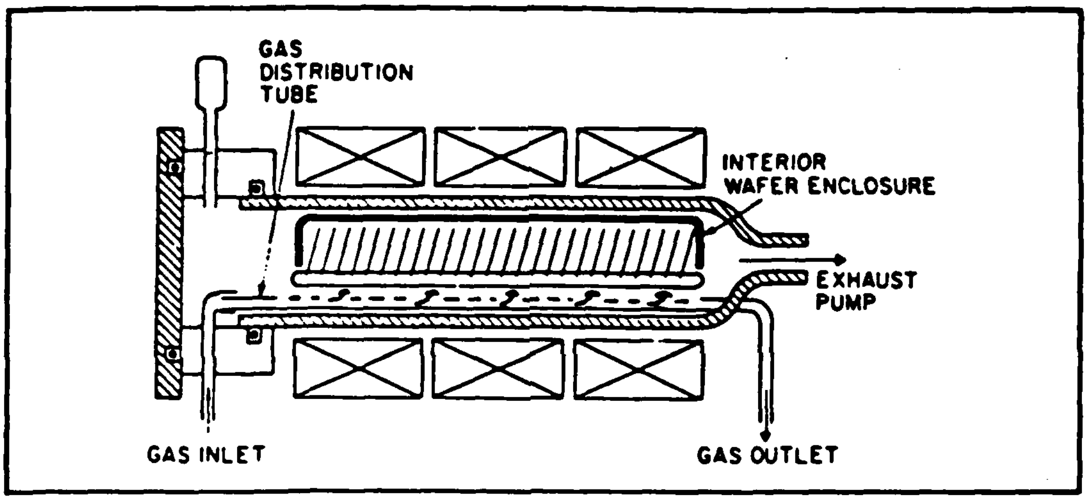
III. D. Proposed FACVD Deposition Process

The FACVD process permits a complicated mix of physical deposition, chemical vapor deposition (CVD), and plasma deposition methods to be used simultaneously in a single system. FACVD should not be regarded as simply a hybrid approach. It contains a number of unique features which have never been applied to material growth problems. In order to better describe the advantages of the FACVD system it is worthwhile to review briefly the limitations of conventional chemical vapor deposition (CVD) systems, glow discharge deposition systems, and plasma enhanced CVD systems (PECVD).

Chemical vapor deposition is a process which forms the backbone of the modern microelectronic fabrication industry. CVD relies on chemical processes as opposed to those deposition processes relying on physical methods, i.e. sputtering, evaporation, etc. CVD is such a broad technique that it is used to form amorphous materials, polycrystalline materials, crystalline materials, alloys, conductors, superconductors, and magnetic materials. As far as the substrate is concerned, most CVD reactions may be categorized as surface controlled or volume controlled and are respectively classified as heterogeneous or homogeneous.[51] When the deposition reaction takes place at the substrate surface, as is normally the case, it is termed heterogeneous. Homogeneous reactions usually occur only in the gas phase. In general, CVD reactions include pyrolysis, reduction, oxidation, hydrolysis, and chemical transport. One example of a pyrolysis reaction is the decomposition of silane employed in silicon epitaxy. Pyrolysis and oxidation have also been employed to grow amorphous SiO_2 films on InP substrates.[4-8] For the epitaxial preparation of III-V compound semiconductors, almost all of the known processes may usually be placed into one of three categories.[51] The first two categories are the hydride and chloride methods, while the third is the organometallic method. The organometallic (MOCVD) approach will be discussed in some detail in the next section. In the chloride method group V trichlorides interact with a group III metal or a III-V compound. The method has produced very low impurity concentrations, but is difficult to control because the trichloride is introduced by heating a liquid. This results in an exponential increase in vapor pressure with temperature, a process which is difficult to regulate. In the hydride method, group V hydrides interact with group III monochlorides. This approach has the advantage that all the input reactants are gaseous and can be carefully regulated. The fine control of individual constituents is important in the growth of compositionally adjusted or graded ternary compounds such as $Ga_{1-x}In_xAs$. Both the chloride and hydride methods are termed vapor phase epitaxy (VPE) and require equilibration of the carrier gas system with a number of heated zones.[52] Fig. 2 indicates some typical low pressure CVD (LPCVD) systems schematically.[51] Note the similarity to the simplest form of the proposed FACVD machine illustrated in Fig. 3. With no plasma, appropriate heaters, and a throttled flow, the FACVD



End-feed LPCVD reactor.



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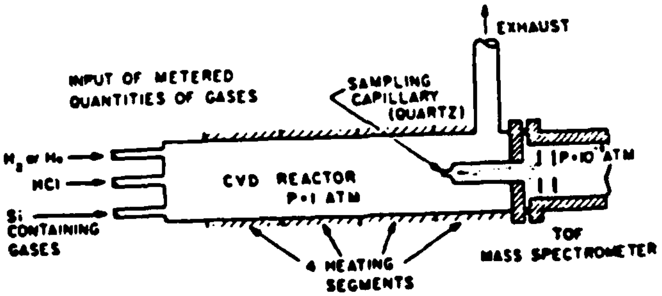


Fig. 2. Examples of LPCVD systems (Ref. 51).

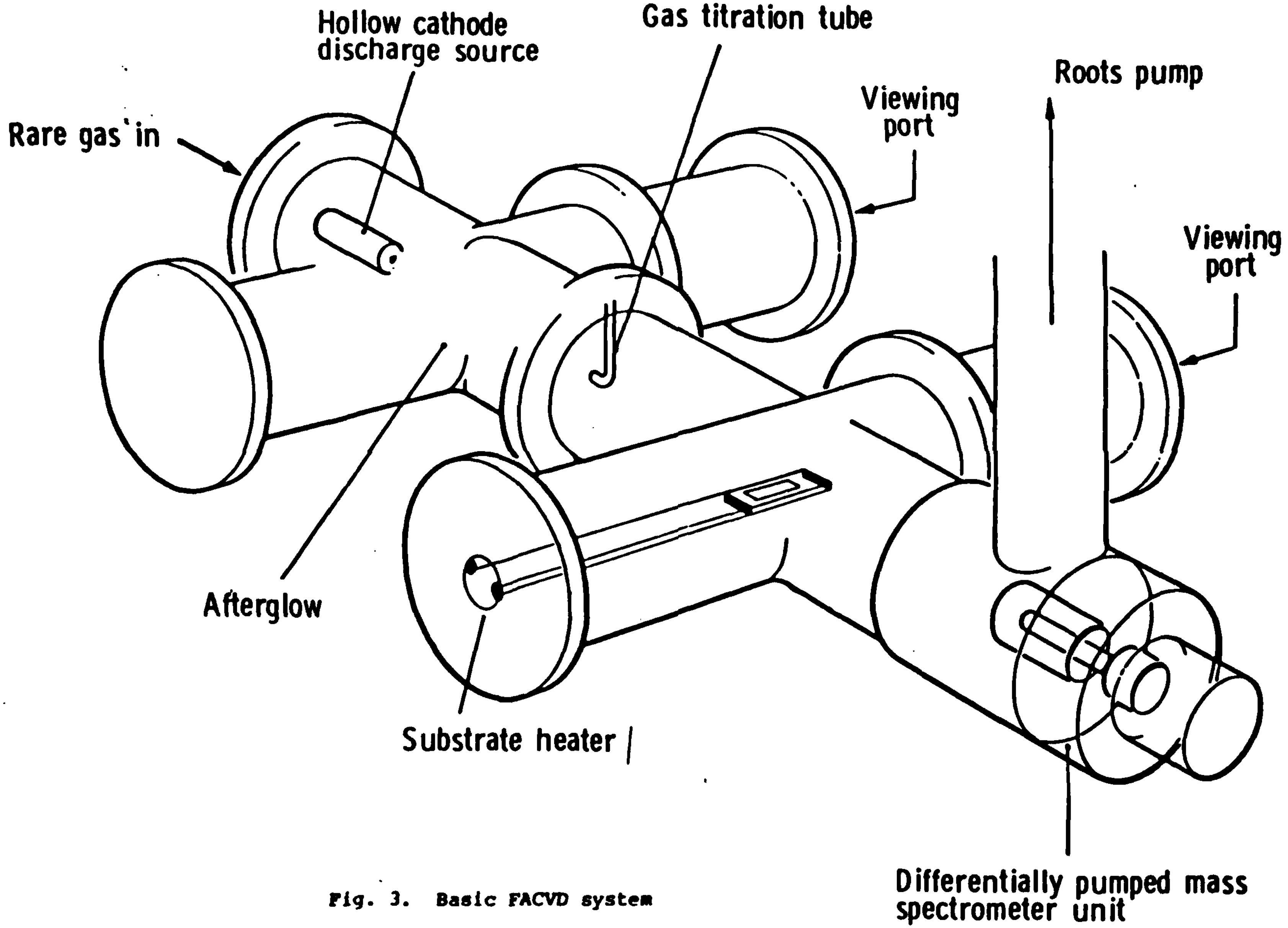


Fig. 3. Basic FACVD system

machine is capable of pure vapor phase epitaxy. The FACVD apparatus is also very much like the flowing afterglow system of Fig. 1. The major difference is the replacement of the reaction assembly with a substrate heater. Before proceeding with a detailed description of the FACVD process, it is useful to briefly continue our review of deposition techniques with a discussion of glow discharge deposition systems.

Conventional glow discharge deposition systems[15,26] are "locked in" to a narrow range of plasma parameters basically delineated by the requirement that the electron-ion production rate equal the electron-ion loss rate. The production and loss rates are closely linked to the physical geometry of each particular system, and there is usually a rather limited range of geometries and pressures which will sustain a stable plasma. Worse yet, the deposition surface and the source plasma are coupled together so that the character of the plasma changes as the film thickness builds up. As indicated in Fig. 4, the factors entering into the plasma include the nature of the discharge gas, excitation power, excitation frequency, gas flow rate, discharge geometry, and pumping speed.[53] The factors entering into the plasma-surface interaction include surface geometry, surface potential, surface temperature, and surface characteristics. As mentioned previously, both the plasma and the surface are coupled so that all of the factors are linked together. Fig. 5 illustrates the complexity of the spectrum obtained from a simple carbon tetrachloride discharge with silicon dioxide electrodes.[53]

The glow discharge systems just described really form a subset of plasma assisted CVD reactions. The presence of a plasma normally exposes the substrate to such a harsh environment, consisting of hot electrons, ions, and various excited atomic and molecular species, that surface bombardment

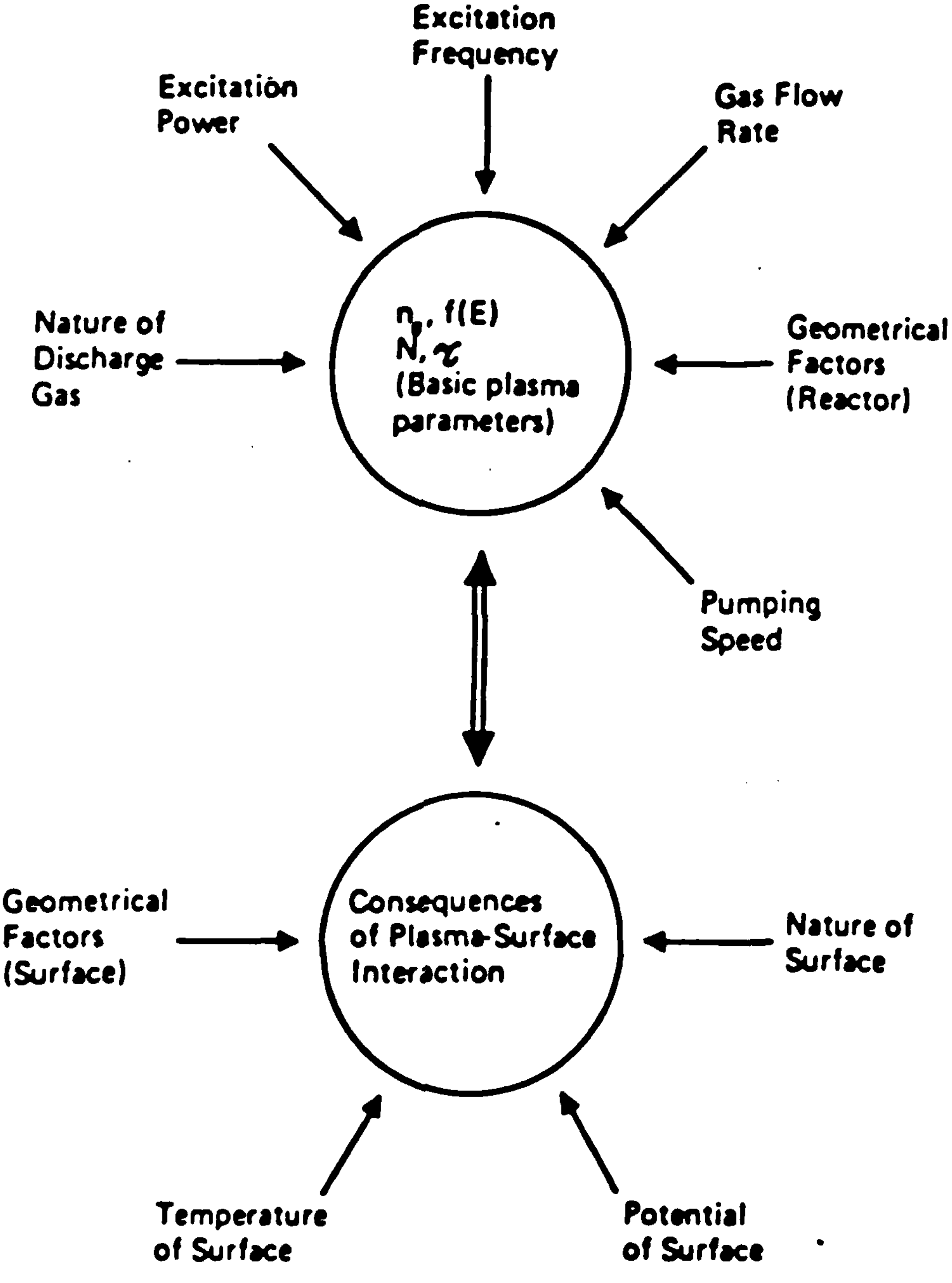


Fig. 4. Plasma-surface coupling parameters (Ref. 53).

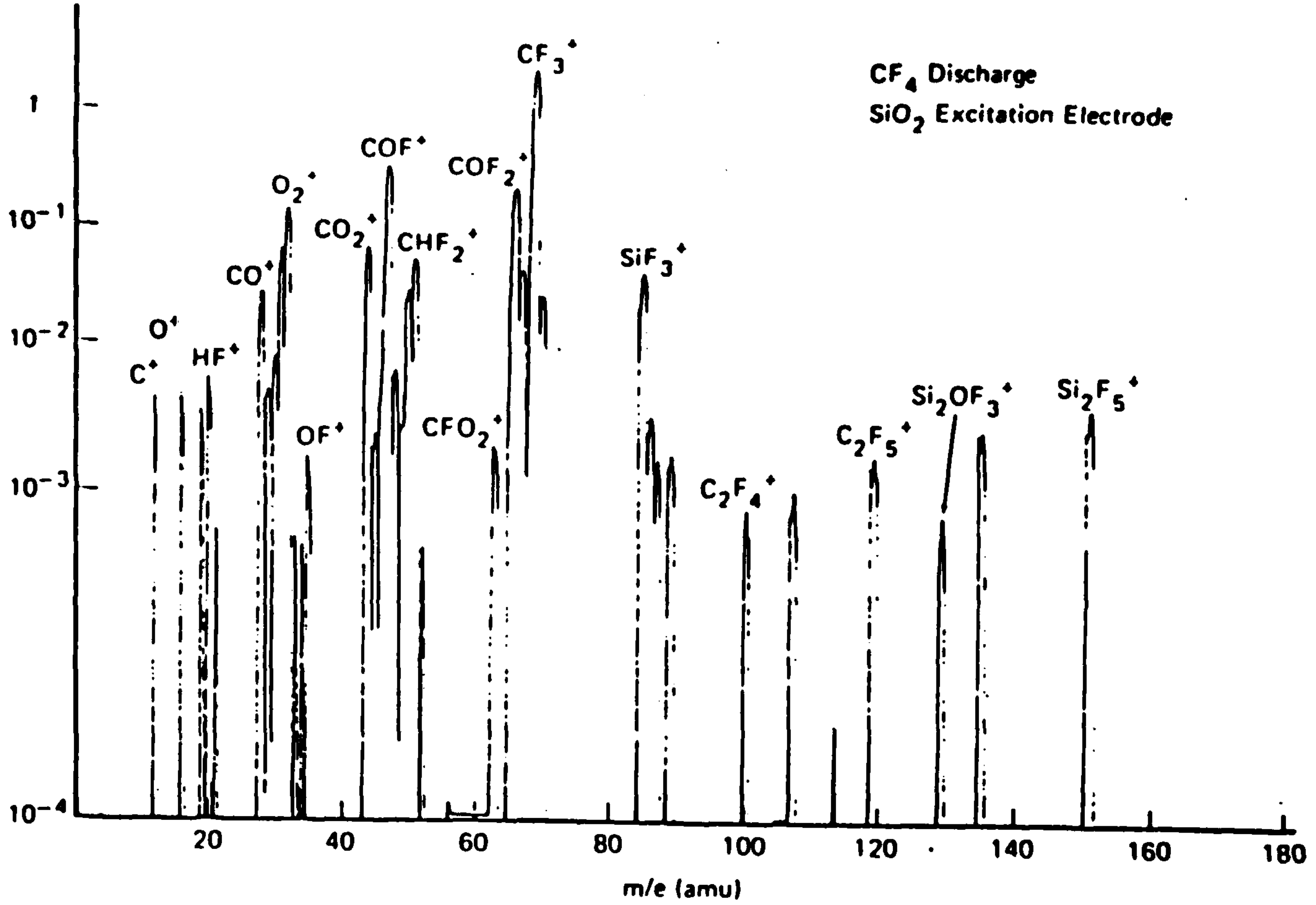


Fig. 5. Mass spectrum of CF_4 discharge with SiO_2 electrodes (Ref. 53).

occurs[54,55] and epitaxial growth becomes impossible; however, plasma assisted epitaxial deposition has been achieved recently by Hariu et al.[56] who employed a combination of physical deposition (evaporation) and plasma excitation. The process was not a CVD reaction and took place at the relatively low pressure of 10 microns, a pressure normally inaccessible to CVD. This type of hybrid plasma-physical deposition process is also possible with the proposed FACVD approach, as is a unique form of "cool" plasma epitaxial growth relying on thermalized metastables and ions in the late afterglow. There is great potential for FACVD epitaxial growth, which will be discussed in connection with MOCVD techniques in the next section.

For the preparation of amorphous films, plasma assisted low pressure CVD systems (LPCVD) are capable of operating at temperatures low enough for oxide film deposition on III-V compounds without stochiometric disruption; however, they suffer from the same limitations as their glow discharge counterparts. These systems are also termed plasma enhanced CVD or PECVD. In the proposed FACVD approach, a deposition substrate is introduced into the jet region with the idea of forming an exceptionally high quality oxide layer. The visual analogy may be likened to placing a penny in the flames of a horizontally positioned bunsen burner. If the buffer gas is helium and a dielectric film is to be formed on an InP substrate, two reactant gases need to be introduced into the reaction region. The reactant gases in this case could be silane and nitrous oxide or silane and oxygen. They would be introduced into the downstream flow region. The combination would react in the vicinity of the substrate to produce films of amorphous silicon dioxide on the substrate. Similarly silane and ammonia could be mixed to form films of silicon nitride. In the case of an oxide layer it is desirable to produce a purely amorphous structure and to avoid pollycrystalline or crystalline growth. The choice of structure is determined by controlling the reaction chemistry and the deposition temperature. The oxidation process would proceed in the following manner. Upon contact with the metastables and ions in the thermalized plasma, the reactant gases would immediately be excited and ionized. They would then be able to react chemically in the vicinity of the substrate to produce the desired films. The whole process would be carried out far in the afterglow where the He (2^3S) metastables, the electrons, and the He$^+$ ground state ions all have atomic velocities close to room temperature. This type of low temperature deposition is quite likely to preserve the delicate stoichiometry of the III-V substrate. It is known that high energy electron-ion bombardment can induce surface damage[54] which can cause the formation of polycrystalline oxide films of poor insulating ability. Similarly, hot plasmas are known to produce trapping centers[55] which limit oxide stability. It is therefore quite possible that superior insulating films for III-V compounds may be produced with the cool plasma of the FACVD approach. Indeed evidence already exists from the work of Meiners[11] that indirect plasma deposition of dielectric films on InP produces lower surface state densities than does PECVD. Later work by Lile et al.[3] indicates decreased interfacial trapping and improved MIS device stability with this deposition technique. The indirect deposition approach permits only atomic oxygen to reach the deposition region. It is an afterglow approach, but a very limited one with respect to FACVD. Ignoring some of the hybrid advantages of FACVD (which will be discussed shortly), FACVD provides much finer control of afterglow constituents than does indirect plasma deposition. It also provides a vastly enhanced range of afterglow densities. As discussed in the previous section, the various afterglow species of the FACVD process may be selectively descriminated against. This permits great versatility in examining the effects of these species on film quality. The ability to scan through some three orders of magnitude in afterglow density is also likely to prove significant. Finally, the FACVD system is inherently a "cleaner" system and should produce films with significantly lower levels of impurities. The cleanliness issue will be discussed in detail later in this section.

A number of other unique advantages of the FACVD method should now be considered. For example, there may be some optimum point between a hot plasma and a cool plasma which produces very high quality dielectric films. The position of the substrate, together with the point of introduction of the reactant gases, can be used to produce a very broad range of electron temperatures and number densities in the reaction region. This would not be possible with a conventional PECVD machine, but is possible with the FACVD approach because the electron-ion production rate is now controlled independently, of the loss rate. All the electrical excitation takes place upstream of the reaction region, and the production rate is now decoupled from the reaction process. In practice, the point of introduction and therefore the electron temperatures and number densities may be continuously varied by changing the pumping speed and thus altering the length and character of the plasma jet. Typical electron temperatures here might be adjustable from 300°K to $100{,}000^{\circ}$K, while electron number densities might range from 10^{10} to 10^{13} electrons/cm^3. By contrast, in normal quasi-static glow discharge deposition systems, the range of available electron temperatures and number densities is inherently quite narrow. Typical temperature ranges here might be $50{,}000$-$100{,}000^{\circ}$K, while typical number densities might be only 10^{10} to 10^{11} electrons/cm^3. These restrictions are caused by the fact that a normal glow discharge must have a certain minimum electron-ion production rate to balance losses, and that the production rate is severely restricted by geometric and physical factors. Because plasma parameters are known to be crucial to the formation of high quality oxide layers, the flowing afterglow system with its much greater range of plasma parameters should be capable of producing oxide films with superior characteristics.

There is another advantage of FACVD over conventional glow discharge deposition. When a substrate or reactant gas is introduced into a normal glow discharge deposition system, the foreign body or bodies alter the character of the discharge system -- the system acts as a coupled whole. As films build up on the substrate, the character of the plasma therefore changes continuously. In the flowing afterglow system, the source plasma is generated upstream, independently of the reaction region -- the system is essentially decoupled. Therefore the results are likely to be more repeatable and relatively free of discharge contamination products.

Another unusual feature of the flowing afterglow system is the exceptional cleanliness of the system. The gas flow is very rapid so that there is very little time for impurity outgassing from the walls or atmospheric leakage to mix with any given volume of plasma. Further, because of the laminar flow patterns in the tube, gas leaks can not easily penetrate into the central region where the substrate is located. The fact that the gas behaves like an incompressible fluid[46] also inhibits impurity contamination. The improved cleanliness is likely to to produce dielectric films with low defect densities.

Still another advantage is the flexibility of the system in the sense that it will have the ability to introduce a large variety of substances into the buffer gas stream without affecting the source plasma. As shown in Fig. 6, this includes the ability to sublimate solids in a tungsten boat directly in front of the deposition substrate. The buffer gas is flowing so rapidly that the sublimation product is immediately swept into the substrate area. If the reactions are chosen to be endothermic and are thermodynamically permissible, deposition will occur preferentially in the vicinity of the heated substrate. For example, various substances such as phosphorus, arsenic, selenium, tellurium, germanium, silicon, aluminum, gallium, tin, lead and copper may be deposited simultaneously with gases introduced (titrated) into the system. FACVD is so versatile that it may even have the capabilty to produce amorphous solar cells and then coat them with indium tin oxide! The method just described is essentially a curious hybrid combination of physical evaporation, chemical vapor deposition, and plasma assisted interaction. The system is also capable of pure physical deposition (like an evaporator) or of combined physical deposition and plasma excitation, as in the apparatus of Hariu et al.[56] discussed earlier in this section. For FACVD operation in the combined physical deposition and plasma mode, the presence of an optional high velocity flow stream permits the operating pressure to be extended from Hariu's rather restrictive 10 micron region well into the torr range. The effects on epitaxial growth are unknown at this point, but the ability of the FACVD system to operate in a physical deposition mode means that new reactions not accessible to plasma enhanced low pressure CVD systems may be examined. For example, it may be possible to sublimate silicon directly into the flow stream and eliminate the use of silane altogether. In this situation, oxide

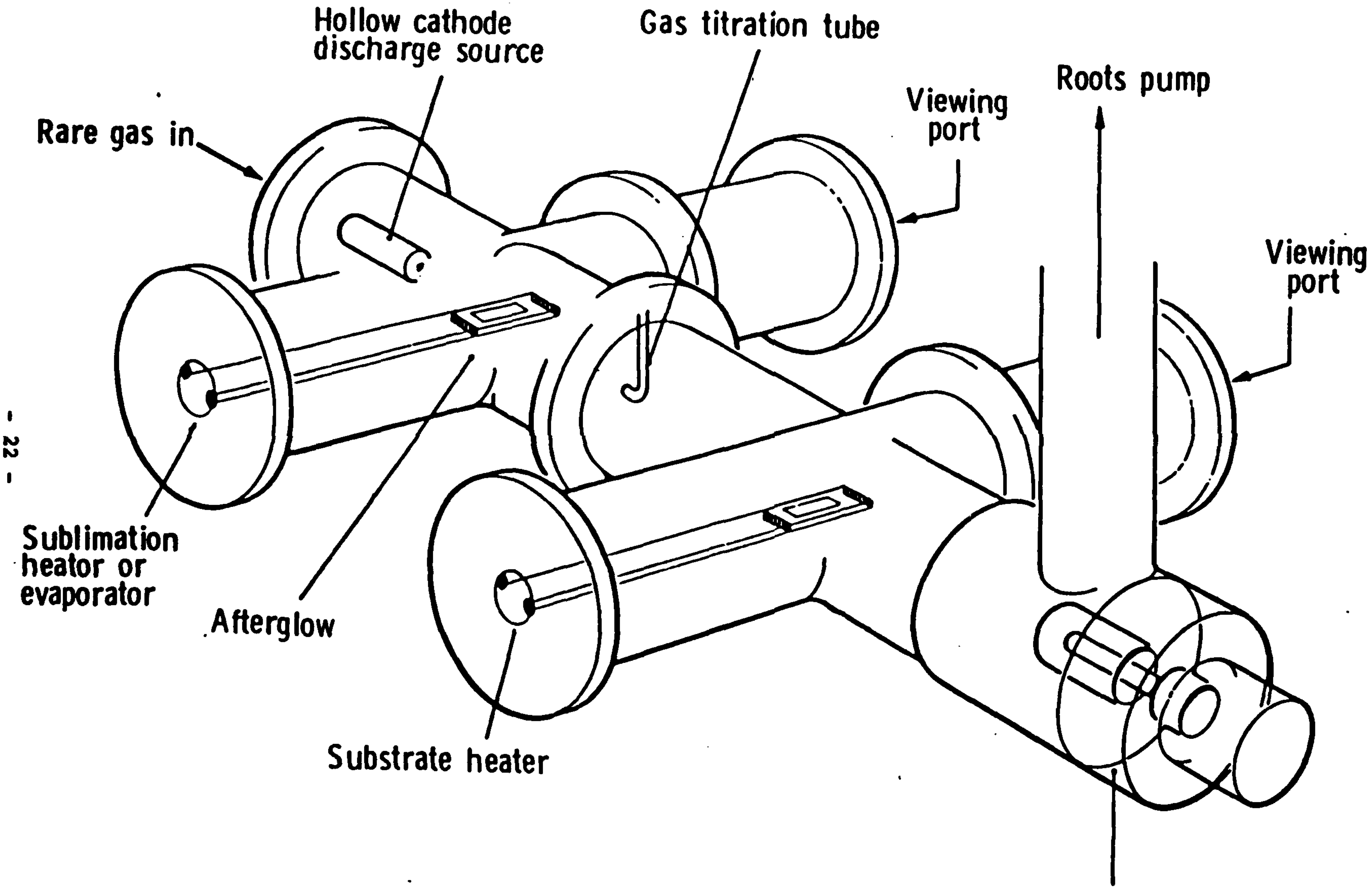


Fig. 6. FACVD apparatus with physical deposition capability

films with very unusual characteristics may be produced.

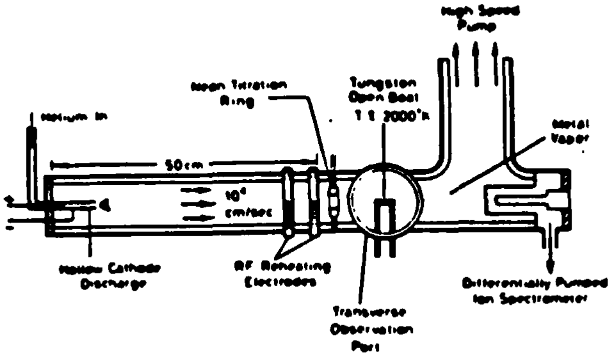
III. E. Comparison of FACVD with MOCVD

Organometallic pyrolysis (OMP), commonly referred to as metalorganic CVD or MOCVD, is a relatively recent addition to the CVD family.[57-61] In this approach diethyl or dimethyl compounds of metals interact with group VI hydrides. MOCVD provides even more precise control of the input constituents than is possible with the hydride technique discussed earlier. This additional control permits graded superlattice structures of varying bandgap materials to be grown. Grading is necessary in the heteroepitaxial growth of ternary and quaternary compounds in order to minimize lattice mismatches. Whereas normal CVD requires temperature control over several heated zones, MOCVD requires control over only one heated zone. The equilibration time is therefore considerably shorter and much sharper compositional changes (greater control) can be achieved.

A typical MOCVD system[59] is illustrated in Fig. 7. It is clear that the basic components are very similar to the simplest form of the FACVD machine illustrated in Fig. 1. MOCVD operation can be simulated with a stripped FACVD machine simply by throttling down the flow rate and turning off the FACVD plasma. An exact simulation requires the insertion of a baffle in the vicinity of the substrate heater to achieve very precise thermal control; nonetheless, the basic FACVD machine clearly can be adapted to reproduce existing MOCVD operation. Perhaps more exciting is the possiblity that some of the unique features of the FACVD process will permit operation in regimes previously inaccessible to MOCVD.

III. F. Status of Construction Project

As shown in Fig. 8, a good portion of the basic FACVD machine has already



Flowing Afterglow Apparatus

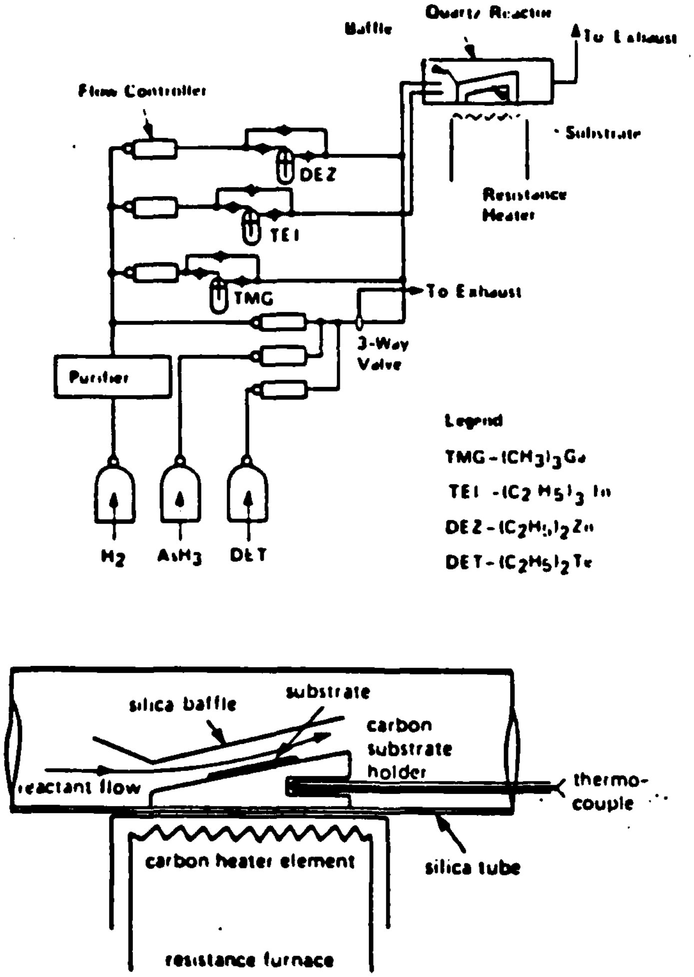


Fig. 7. Typical MOCVD system with close up of reaction zone (Ref. 59)

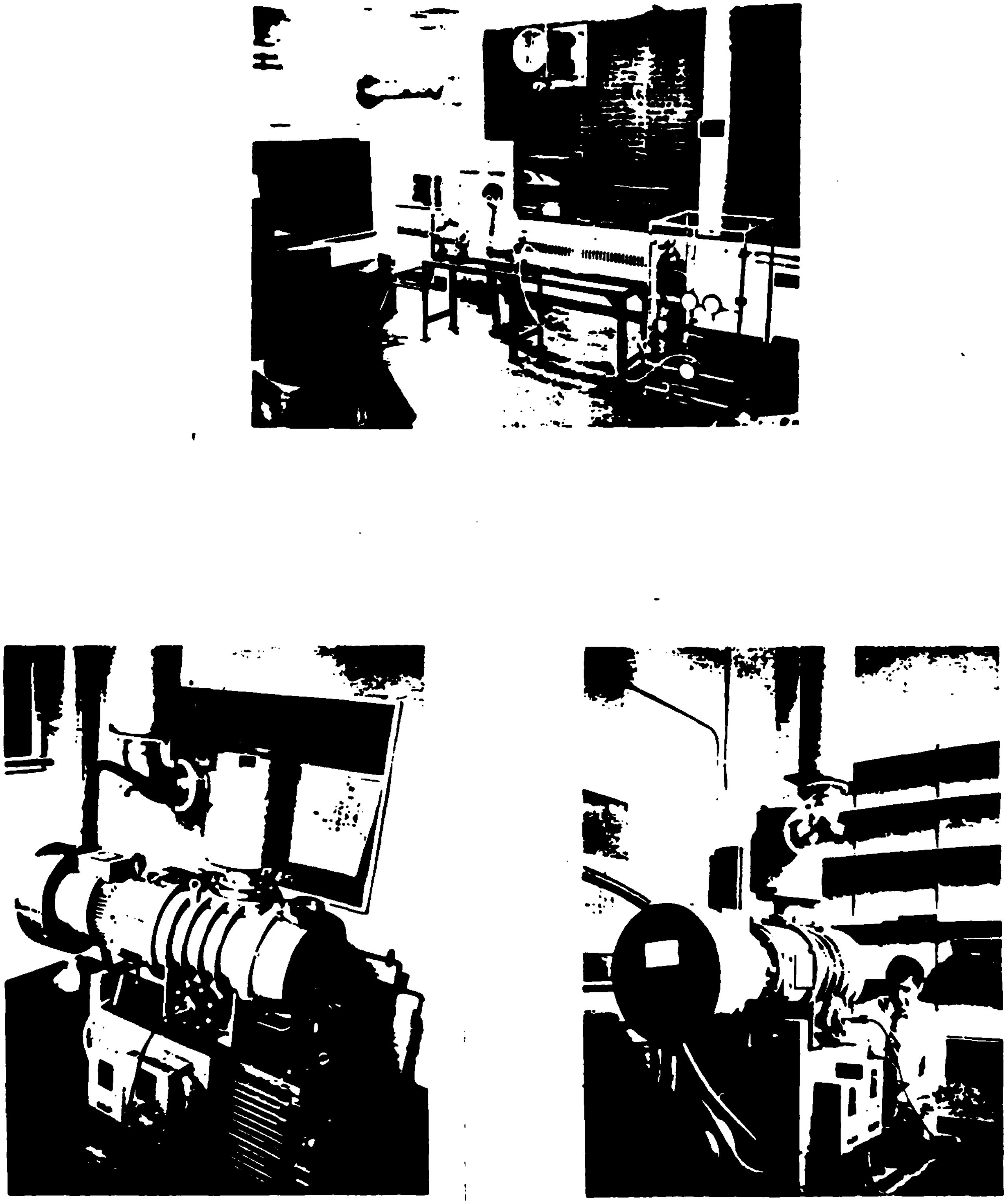


Fig. 8. FACVD system under construction at Wayne State

been completed. The pumping system alone stands over five feet in height and has a rated throughput of 1300 cfm in the pressure range 30 microns to 6 torr. It consists of a Roots blower pump backed by a large rotary pump. Installation of the pumps was a major construction project and involved pipefitters, plumbers, carpenters, and electricians. The noise and vibration generated by these pumps required them to be installed in a room adjacent to the experimental room, necessitating a pumping line about 15 feet in length. This was fabricated from 1/16" stainless steel vacuum ducting of 8" internal diameter, cut and welded into a number of vertical and horizontal sections. The dimensions of the pump line are such that it does not significantly reduce the pumping speed at normal pressures of operation; however, for some experimental conditions it is desirable to reduce the pump speed. This is accomplished by placing a gate valve directly between the end of the pump line and the afterglow apparatus. The gate valve was especially designed and fabricated to provide a variable conductance, but not a vacuum-tight seal. It consists of a plate driven across the pipe by a calibrated rack and pinion drive. An external 10 turn potentiometer ensures that the conductance can be reset to reproduce any particular flow conditions.

The flow tube consists of modules of thick wall Pyrex tubing with an internal diameter of 10 cm. The advantage of this form of construction is that it provides flexibility in the layouts possible for the flow assembly when the various Pyrex crosses and additional straight sections are inserted. In addition the modular approach is easier to clean and maintain. A metal 'T' piece was designed and fabricated to support the gate valve assembly and Pyrex tubing. Vacuum seals are made with Teflon 'O' rings. Previous experience with these systems indicates that the heat balance is such that Teflon is adequate even for reaction temperatures approaching 2000°K.

At the inlet to the flow tube a nozzle and hollow cathode assembly were especially designed and fabricated to provide efficient discharge excitation and a jet like flow. Despite the fact that the Oxford group[37-40] demonstrated that DC hollow cathode excitation was superior to microwave excitation in these systems, microwave excitation may eventually prove more attractive for FACVD work. First, recent improvements in microwave excitation sources[62-64] suggest that their use should be reconsidered. Secondly, sputtering effects in the hollow cathode discharge could possibly cause downstream contamination in the reaction region. In the initial work, these potential refinements were ignored and a plasma was routinely maintained with the hollow cathode excitation source. The use of microwave excitation should permit all internal electrodes to be eliminated and provide substantial improvements in system impurity levels.

All of the parts, together with the gate valve, are clamped to a single 4 x 2 inch steel channel-section girder 10 feet in length. The clamps can be moved to any position along the bench and their height and angle adjusted to allow for small inaccuracies in alignment between the flow tube axis and the bench. The channel is supported by a heavy angle iron framework, which also provided a stepped height adjustment.

Gases are supplied to the discharge with a gas control assembly. Cleanliness of the gas supplied to the discharge is of the utmost importance in preserving high afterglow ion densities since impurities are known to have adverse quenching effects.[37-40] Helium of 99.995% purity is typically employed. Further purification of the gases is achieved by passage through a molecular sieve. The flow rates are such that the machine consumes about $200 worth of Helium every five hours.

IV. Experimental Results

Upon completion of the basic flowing afterglow system and the substrate heater assembly, a series of experiments were conducted to determine the optimum deposition region. An in situ optical imaging system was set up to monitor film deposition. The computerized imaging system was capable of processing and displaying frames representing temporal sequences of film growth. The imaging system supplied a gross disgnostic capability that permitted a rapid assessment of surface film properties. This included the ability to recognize pinholes and evaluate deposition uniformity as a function of time.

Since substrate cost was of considerable importance, the choice of substrates was intended to progress in an orderly sequence from glass through crystalline silicon to indium phosphide. The initial experiments were conducted using glass microscope slides as substrates. The operating region for plasma deposition was considerably expanded over the initial specifications. The available range included pressures from 0.2 torr to 200 torr. Substrate temperatures were continually variable from 100° C to 250° C.

The plasma density is dependent on a complex combination of parameters consisting of flow rate, buffer gas composition, titration gas composition, cathode current, and substrate position. Previous experience with similar systems indicated that an ion density on the order of 10^{14} per cm^3 should be attainable. Experiments were conducted over the entire range of operating parameters. With helium as a buffer gas and silane as a titration component, appreciable deposition was obtained only at the upper end of the pressure and ion density range. After exhaustive tests, no combination of parameters in any other region could be found that permitted noticeable film deposition within a 30 minute time frame. In fact, appreciable deposition only ocurred at pressures in excess of 100 torr and cathode currents close to melt down conditions. In this region, the entire one inch tungsten hollow cathode assembly glowed white hot with an intensity that required the use of dark glasses for personnel in the immediate area of the discharge.

The deposition quality was rather poor. Because the pressure was so high, Mach striations, indicating propagation at or beyond the speed of sound, became apparent. The axial uniformity varied so substantially that uniform film thicknesses across the entire substrate assembly were unobtainable. A decrease in the operating pressure immediately resulted in improved film uniformity; however, this improvement was accompanied by a nonlinear increase in deposition time. The cathode was incapable of generating high enough plasma densities at the lower pressures to achieve appreciable silane decomposition rates. The high pressure region also resulted in the incorporation of a substantial number of pinholes in the film structure. These pinholes disappeared entirely at lower pressures. Since the deposition quality was observed to be inadequate for the growth of MIS structures, no experiments were conducted with crystalline substrates .

V. Conclusions

As anticipated in the experiments conducted before the silane gas handling system was fabricated, the system was indeed able to produce thin film structures. The design did not anticipate the fact that relatively high ion densities and operating pressures would be required to produce a reasonable rate of silane dissociation. On the other hand, the experimental evidence consistently indicated that lower operating pressures were better suited to the growth of high quality films. In the low pressure regime, the system was unable to produce a sufficient ion density to achieve a reasonable deposition rate.

In order to achieve the necessary ion density in the low pressure regime, a major redesign of the system is required. One approach might be to build a preionization assembly so that the plasma could be generated in a two stage process. Adding a third stage of microwave pumping is also a possibility. Another modification might include the use of optical pumping to help provide the required dissociation rate. Perhaps the best solution would be to increase the pumping rate so that the plasma temperature did not drop substantially in the flow region between the discharge assembly and the deposition region. Unfortunately, this particular solution suffers from the disadvantage that a substantial increase in capital investment is required in order to purchase a pumping system with the necessary capacity. Indeed, all of the suggested modifications are beyond the budget limitations of this project. For this reason no further work has been undertaken on system improvements.

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