BENIGN PRECURSORS FOR SEMICONDUCTOR PROCESSING

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Semiconductor manufacture not only requires efficient processes that produce reliable products, but also should use benign precursors as input materials to generate a product and by-products that are environmentally safe. Current device processing, by its nature, is demanding and depends on difficult-to-handle materials. As part of a long-term strategy to develop safer, more efficient processes that also have zero environmental impact, a technique is reported here for III-V processing that obviates the need for transport, storage, and handling of arsine in high-pressure cylinders. This technique, called *in situ* generation, provides an immediate solution for a potential safety problem in current semiconductor processing, the "sudden release hazard" of cylinders of compressed toxic gases. Other promising solutions for economically viable processing that meet safety and environmental compliances exploit chemically inert reagents in plasma reactors or use nontoxic precursors. Applications are described for the fabrication of a broad range of devices requiring dry etching and chemical vapor deposition processing steps.

Introduction

Volatile precursors are used widely in semiconductor technology for fabricating a broad range of devices including diode lasers, light-emitting diodes (LEDs) and long-wavelength detectors. Manufacturing even more advanced high-performance products such as metal-semiconductor field-effect transistors (MESFETs), optical waveguide fiber amplifiers, high-electron-mobility transistors (HEMTs), and integrated electrooptic devices also requires gaseous chemicals as key starting materials for fabricating the thin-film structures on which these devices depend. In all cases, production of the device material

Acronyms and Abbreviations in This Paper

Al	aluminum
APD	avalanche photodiode
As	arsenic
C	carbon
CVD	chemical vapor deposition
FET	field-effect transistor
Ga	gallium
H	hydrogen
HBT	heterojunction bipolar transistor
HEMT	high-electron-mobility transistor
In	indium
LED	light-emitting diode
MBE	molecular-beam epitaxy
MOCVD	metal-organic CVD
MESFET	metal semiconductor FET
P	phosphorus
PACVD	plasma-assisted CVD
PIN	positive-intrinsic-negative
ppb	parts per billion
QWIP	quantum-well infrared photodetector
RF	radio frequency
SEED	self-electrooptic-effect device
Si	silicon
UHV	ultrahigh vacuum
VPE	vapor phase epitaxy

involves converting gaseous precursors into solid layered structures by an appropriate gas-phase technique based on chemical vapor deposition (CVD), metal-organic CVD (MOCVD), vapor-phase epitaxy (VPE), or molecular-beam epitaxy (MBE). Table I summarizes characteristic material structures, corresponding fabrication techniques, and the most frequently used precursors for these devices. In addition to precursors for thermally induced thin-film deposition of device structures, reagents are required for plasma deposition and etching. Other volatile chemicals are used as carrier gases, vapor-transport agents, and dopants, and for atmospheric control during semiconductor processing. Thus, gaseous precursors are used in most of the integrated processing steps of device manufacture.[1] For these processing steps, a wide variety of well established reagents exist. In Table II, various examples are grouped according to similarity in chemical reactivity.

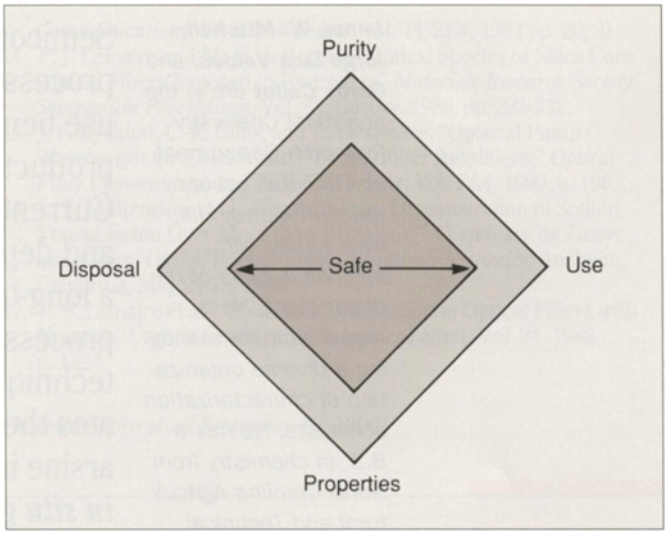

Figure 1. Factors controlling applications of device processing reagents. The diagram indicates that the purity, use, properties, and disposal of a reagent must fall within a range of safety.

Inevitably, the usefulness of any of these specific reagents will be dictated by the prevailing constraints imposed by attempts to properly balance the parameters given in Figure 1. Traditionally, the precursor reagent is selected primarily to ensure that it imparts the required properties to the device being fabricated. Compliance of the reagent with stringent specifications of purity is often a key factor as well. Obviously, in the future, these issues will continue to be important in dictating the selection of the precursor. However, applications for precursors will be increasingly constrained by considerations of safety and needs for preservation of the environment. Whether

Table I. Arsenic-Based Semiconductor Devices

Device	Structure	Processes	Precursors
Avalanche photodiode (APD)	InGaAsP/InGaAs	VPE	In, Ga, $AsCl_3$, PCl_3
Diode lasers	InGaAsP/InP	MOCVD, VPE	In, Ga, As, AsH_3, PH_3, $In(CH_3)_3$
High-electron-mobility transistor (HEMT)	AlGaAs/GaAs	MBE, MOCVD	Al, Ga, As, AsH_3, $Ga(CH_3)_3$
Heterojunction bipolar transistor (HBT)	InGaAs/InP	MBE	As, Ga, In, AsH_3, PH_3
	AlGaAs/GaAs	MOCVD	$Ga(CH_3)_3$, $In(CH_3)_3$, $Al(CH_3)_3$
Light-emitting diode (LED)	InGaAsP/InP	VPE	In, Ga, AsH_3, PH_3
Metal-semiconductor FET	InGaAs/InP	VPE	In, Ga, AsH_3, PH_3
Positive-intrinsic-negative (PIN) photodetector	p-InGaAs/n-InGaAs/ n-InP	VPE	In, Ga, $AsCl_3$, PCl_3
Quantum-well infrared photodetector (QWIP)	AlGaAs/GaAs	MBE	Al, Ga, As, AsH_3, PCl_3
Self-electrooptic-effect device (SEED)	AlGaAs/GaAs	MBE	Al, Ga, As

the reagent can be used safely in the workplace and then its residues and by-products properly disposed of, without harmful environmental effects, are becoming major issues.

Although the increasing importance of safety issues and concerns for preserving the environment could possibly place the continued use of many essential device processing reagents (Groups 1 and 3 of Table II) in jeopardy, a "sword of Damocles" has not materialized for device processing technology. Instead of impeding progress in device processing, these trends of the future are stimulating the development of alternative precursors and processes with greatly improved margins of safety.

This paper discusses the improved procedures for storing and handling existing hazardous reagents and the significant progress occurring in the development of precursors of low toxicity. Advances are cited also for the engineering design of reagent-generating systems and device processing techniques that are tailored to maximize safety while minimizing effects on the environment.

Safe Use of Conventional Reagents

When there are no other suitable options, the semiconductor industry uses potentially hazardous chemicals for device manufacture. Highly hazardous reagents for vapor-phase growth of thin-film structures composed of Si, Al, As, Ga, In, or P are required for the fabrication of devices with tailored properties. For example, silane and its chlorosubstituted derivatives, which are pyrophoric, are still the only precursors for CVD growth of epitaxial films of silicon. A broader range of volatile reagent choices exist for the synthesis of compound device structures containing Al, As, Ga, and P. However, the most prevalently used precursors, trimethylaluminum, arsine, trimethylgallium, and phosphine, are either highly pyrophoric or highly toxic, or both. Because there are few benign sources for the synthesis of semiconductor materials, the device industry proactively promotes the safe use of hazardous reagents in manufacture. The industry practices the highest standards for their safe use in the workplace and has compiled an exemplary record of safety over four decades.[2]

Stringent AT&T guidelines now specify procedures for handling, use, and storage of toxic gases in the workplace. From the plethora of potentially lethal gases, arsine is selected as the representative example discussed throughout this paper. Containment in a compressed gas cylinder of no more than 100 grams in a

Table II. Essential Precursor Reagents for Device Fabrication

Name	Formula	Applications
Group 1. Highly reactive toxic gases		
Ammonia	NH_3	CVD
Arsine	AsH_3	Dopant, epitaxy
Boron trichloride	BCl_3	Ion implanting
Boron trifluoride	BF_3	Ion implanting
Chlorine	Cl_2	Plasma etchant
Dichlorosilane	Cl_2SiH_2	Epitaxy, CVD
Fluorine	F_2	Plasma etchant
Germane	GeH_4	CVD, epitaxy
Hydrogen chloride	HCl	Etching, epitaxy
Hydrogen selenide	H_2Se	CVD
Hydrogen telluride	H_2Te	CVD
Phosphine	PH_3	Dopant, CVD
Silane	SiH_4	CVD, epitaxy
Group 2. Moisture-sensitive inorganic halides		
Antimony pentachloride	$SbCl_5$	Dopant
Arsenic trichloride	$AsCl_3$	CVD, VPE
Germanium tetrachloride	$GeCl_4$	MOCVD, CVD
Indium trichloride	$InCl_3$	CVD, VPE
Phosphorus oxychloride	$POCl_3$	Dopant, MOCVD
Phosphorus tribromide	PBr_3	Dopant
Phosphorus trichloride	PCl_3	Dopant
Silicon tetrachloride	$SiCl_4$	CVD, MOCVD
Silicon tetrafluoride	SiF_4	MOCVD, CVD
Tantalum pentachloride	$TaCl_5$	CVD
Titanium tetrachloride	$TiCl_4$	CVD
Tungsten hexafluoride	WF_6	CVD
Group 3. Interhalogen and heteroatomic gases		
Bromine trifluoride	BrF_3	Plasmaless etchant
Chlorine trifluoride	ClF_3	Plasmaless etchant
Iodine pentafluoride	IF_5	Plasmaless etchant
Nitrous oxide	N_2O	CVD
Nitrogen trifluoride	NF_3	Plasma etchant
Oxygen difluoride	OF_2	Plasma etchant

Table II (Continued)

Name	Formula	Applications
Group 4. Metalloalkyls		
Tetraethylorthosilicate	$Si(OCH_2CH_3)_4$	CVD
Trimethylaluminum	$Al(CH_3)_3$, liquid	CVD
Trimethylgallium	$Ga(CH_3)_3$, liquid	CVD, VPE, MBE
Trimethylindium	$In(CH_3)_3$, crystal	MOCVD, VPE
Trimethytin	$Sn(CH_3)_3$, crystal	CVD
Trimethylarsenic	$As(CH_3)_3$, liquid	CVD, VPE, MBE
Group 5. Stable halohydrocarbons		
Carbon tetrachloride	CCl_4	Plasma etchant
Carbon tetrafluoride	CF_4	Plasma etchant
Chloroform	$CHCl_3$	Plasma etchant
Dichlorodifluoromethane	CCl_2F_2	Plasma etchant
Difluoromethane	CH_2F_2	Plasma etchant
Freon* compounds (*registered trademark of du Pont)	$C_2Cl_xF_{6-x}$, CCl_xF_{4-x}	Plasma etchant
Fluoroform	CHF_3	Plasma etchant
Hexafluoroethane	CF_3CF_3	Plasma etchant
Sulfur hexafluoride	SF_6	Plasma etchant
Trifluorobromomethane	$CBrF_3$	Plasma etchant
Group 6. Inert and diatomic gases		
Argon	Ar	Atmospheric control
Helium	He	Atmospheric control
Hydrogen	H_2	Reducing atmosphere
Nitrogen	N_2	Carrier, purging
Oxygen	O_2	Plasma etching, oxidation, CVD

standard laboratory is permitted. This cylinder must be housed in an approved exhausted toxic gas cabinet, fitted with a sprinkler head, a suitable exhaust scrubber system or absorber unit, and a parts-per-billion (ppb) level arsine sensor. Arsine from the cylinder is transported to the point of use in double-walled, stainless-steel, welded tubing. Additional safety requirements specify a remote alarm that signals the failure of the exhaust system servicing the arsine cabinet. The status of the laboratory in which the arsine is stored must be monitored continuously by sensors and either displayed remotely or visibly without need for entering the laboratory.

As a final step in the safety assurance process, the user of a potentially hazardous reagent must submit details of the system in which the reagent is to be used and his/her entire technical procedure to several process hazard reviews. These reviews involve discussions with safety experts and colleagues who are experienced

Table III. Toxicity Data for Substituted Arsine

Name	Formula	LC_{50}, ppm	Rating
Arsine	H_3As	42	Highly toxic
Dimethylarsine	$HAs(CH_3)_2$	164	Moderately toxic
Trimethylarsine	$As(CH_3)_3$	>14,027	Practically nontoxic
Tert-butylarsine	$H_2As(Bu-t)$	90	Highly toxic

users of hazardous precursor agents that are similar to those undergoing scrutiny for approval.

The ultimate concern for safety associated with the use of a toxic gas is the "sudden release hazard" associated with a catastrophic failure of the compressed-gas cylinder or its associated valve. Where large amounts of arsine are required for manufacture, planning for this unlikely event includes storage of arsine in remote outdoor or rooftop facilities. These facilities can require expenditures of up to $1 million to accommodate containment and storage and to provide transport lines to the point of use in the laboratory. Despite these expenses, the U.S. electronics industry has developed optimized procedures for handling toxic gases and has installed state-of-the-art facilities for containment, storage, recovery, and disposal of toxic reagents.

Alternatives to Compressed Gas Cylinders

A possible solution to the hazard associated with the sudden release of arsine stored in compressed cylinders is containment of the gas at atmospheric pressure. Investigators at the Naval Research Laboratory have demonstrated atmospheric-pressure storage of AsH_3 in zeolites at 23°C.[3] Desorption of the adsorbed reagent (about 25 percent by weight) below 190°C was demonstrated along with the growth of GaAs and AlGaAs materials by vapor-phase epitaxy. This approach eliminates the hazard associated with compressed gas cylinders. However, it does not circumvent the limitations imposed by safety guidelines that restrict storage of arsine to 100 grams or less. While the restriction of the storage of the toxic gas to 100 grams at atmospheric pressure enhances safety in the workplace, device manufacture can be affected negatively. Device yields and reliability are susceptible to fluctuations of reagent quality associated with frequent changes of low-capacity systems. In fact, frequent changes of the arsine source may actually decrease safety by increasing the opportunity for human error.

We have devised other alternatives to arsine in compressed gas cylinders. In principle, the preferred solution is the replacement of arsine by completely nontoxic reagents. Progress in developing nontoxic, volatile alternatives to arsine has occurred. As shown in Table III, lethally toxic arsine is made increasingly less toxic by alkylation. The fully substituted reagent, $As(CH_3)_3$, is practically nontoxic according to contemporary chemical standards. In addition, as the substitution increases, the reagent becomes less volatile. $As(CH_3)_3$ is a liquid at room temperature. This fully alkylated methyl analog of arsine, trimethylarsine, has been investigated in detail.[4,5] Although this reagent is useful in pyrolytic CVD deposition of GaAs at 400°C or more, the resulting film contains considerable levels of carbon incorporation (more than 10^{16} atoms / cm^3). Codeposition of C into the GaAs layer is minimized by using the partially alkylated reagent, tertiarybutylarsine, $[(t-Bu)AsH_2]$.[6] Since this material is only somewhat less toxic than arsine, it still requires precautions in use. Because it is a volatile liquid and not a gas at room temperature, $(t-Bu)AsH_2$ can be handled more safely than cylinders of compressed arsine gas. A commercial source of a high-purity product has been identified and used by device engineers to fabricate MESFET devices that meet current-voltage and other performance specifications.[6]

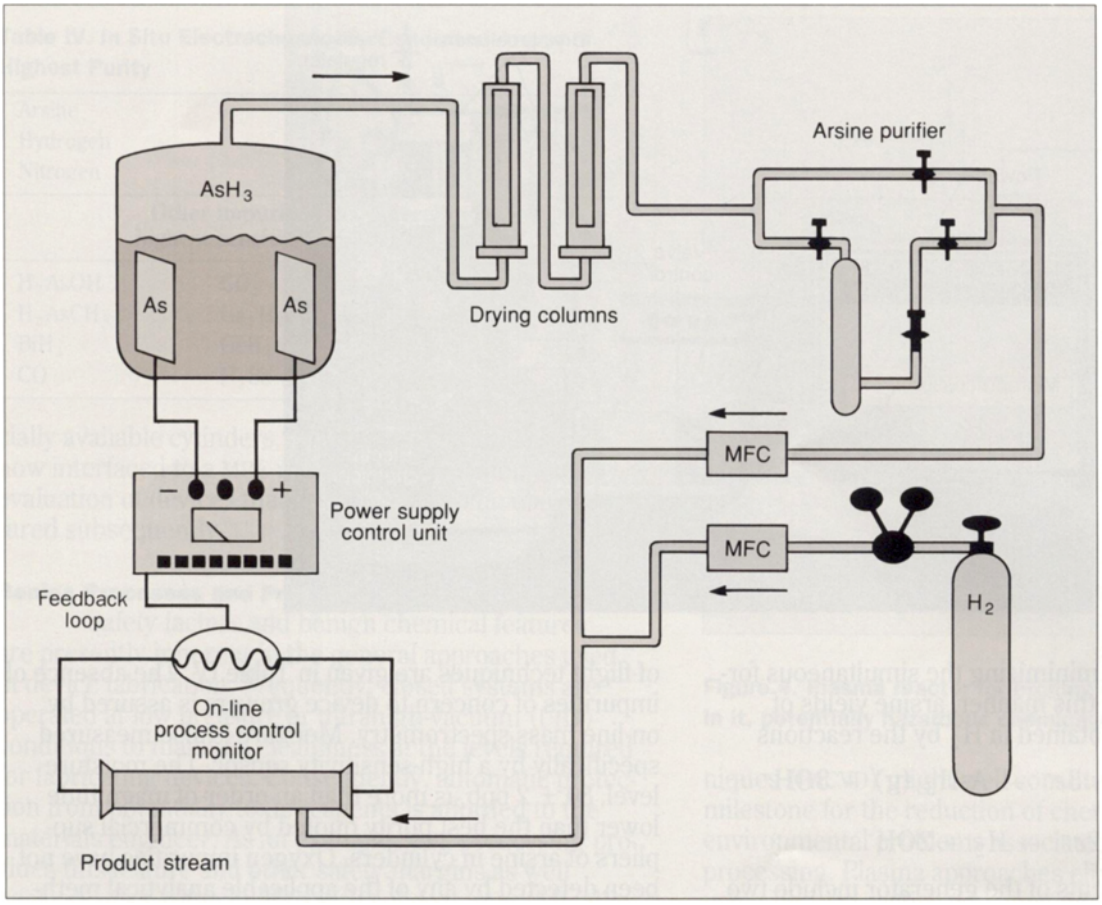

Figure 2. Components of *in situ* electrochemical arsine generator. The system avoids the need to store quantities of arsine.

To eliminate arsine storage, a technique to generate it on demand in desired quantities and deliver it in real time to the reactor has been devised.[7] The technique is based on electrochemical *in situ* synthesis of the reagent at an arsenic metal cathode in a suitable electrolytic cell containing 1.0N potassium hydroxide. Figure 2 is a schematic of the prototype pilot-scale unit. This unit has a total generating capacity of 10 lb of arsine delivered over a wide range of concentrations (2 to 85 percent in H_2), at selectable pressures up to 60 psig, and at variable flow rates up to 1.0 L/min. These reagent fluxes meet all requirements for CVD, VPE and MBE, and satisfy most conditions for MOCVD.

A photograph of the first practical *in situ* generator of a precursor for semiconductor device fabrication is shown in Figure 3. Within the electrochemical cell, a unique packed-bed cathode compartment permits up to 10 lb of pure arsenic metal to be reduced to arsine. The electrode compartment is designed for uniform current distribution, thus assuring controlled consumption of the

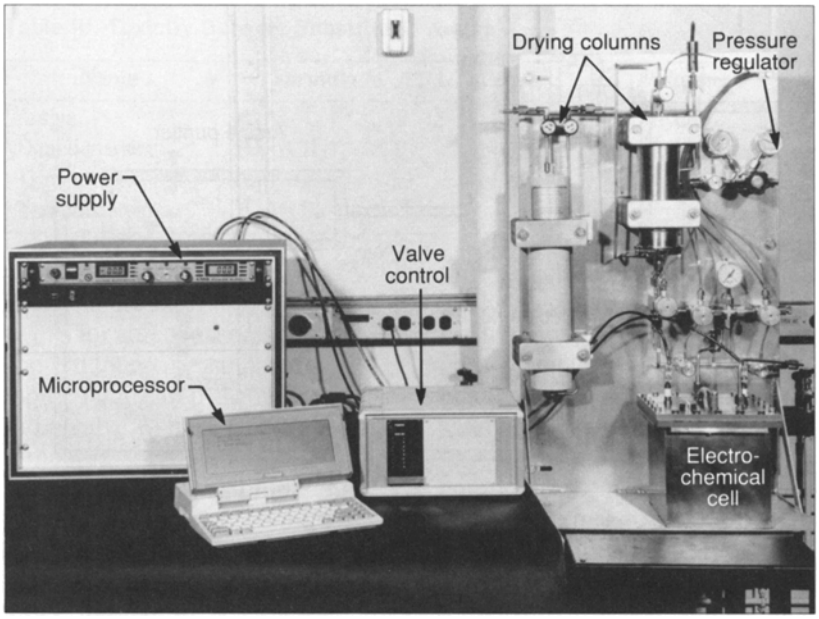

Figure 3. Microprocessor-controlled *in situ* electrochemical generator. The generator is commercially available under license from AT&T.

cathode material while minimizing the simultaneous formation of hydrogen. In this manner, arsine yields of nearly 85 percent are obtained in H_2 by the reactions

$$As\ (s) + 3H_2O + 3e^- \rightarrow AsH_3\ (g) + 3OH^-$$

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^-$$

Other components of the generator include two drying columns in tandem and automated pressure and flow regulation controls. The entire system is controlled by a microprocessor and a direct-current power supply. This system is designed to be housed completely within a standard toxic-gas cabinet. AT&T has licensed Electron Transfer Technologies, Inc., Princeton, New Jersey, to supply the system commercially.

The high quality of *in situ* generated arsine is established by detailed chemical characterizations. Arsine and hydrogen levels measured by on-line acoustic time-of-flight techniques are given in Table IV. The absence of impurities of concern to device growers is assured by on-line mass spectrometry. Moisture is also measured specifically by a high-sensitivity sensor. The moisture level, 80 ± 2 ppb, is more than an order of magnitude lower than the best purity quoted by commercial suppliers of arsine in cylinders. Oxygen impurities have not been detected by any of the applicable analytical methods. During periods when the generator is in idle mode, nitrogen may slowly effuse from the pores of the zeolites used in the drying column. Occasionally, up to 33 ppm of nitrogen has been measured. Fortunately, this inert impurity is innocuous for most applications of arsine.

After verification of the purity of the arsine product, the generator was interfaced to a hydride VPE reactor. InGaAs/InP MESFET devices have been fabricated and shown to equal or exceed the best performance of identical devices fabricated with arsine from commer-

Table IV. In Situ Electrochemically Generated Arsine of Highest Purity

Arsine			83.49%
Hydrogen			16.50%
Nitrogen			~33 ppm
Other impurities not detectable by highest sensitivity mass spectrometry			
H_2AsOH	CO_2	H_2O	PH_3
H_2AsCH_3	Ga_2H_6	H_2Te	SbH_3
BiH_3	GeH_4	O_2	SiH_4
CO	H_2Se	PbH_4	SnH_4

cially available cylinders.[8] The prototype generator is now interfaced to a MBE reactor for fabrication and evaluation of devices that are expected to be manufactured subsequently.

Benign Processes and Precursors

Safety factors and benign chemical features are presently inherent in the general approaches used in device fabrication. Frequently, closed systems are operated at low pressure or ultrahigh-vacuum (UHV) conditions to maintain cleanliness at the levels required for fabricating devices. Consequently, automatic protection from potentially toxic reagents is afforded to the materials engineer. As an example, MBE processing provides this feature and other safety margins as well because of its compatibility with solid sources. Metallic sources of As, Al, and Ga are essentially benign in comparison to the respective hydride or halide derivatives. Thus solid-source MBE greatly reduces and simplifies handling and environmental problems. Precautions, however, are still required during reloading of new sources. As verified by a recent finding, once the MBE system is opened, ppb levels of arsine gas can be produced from reactions of evaporated residues of As with moisture on the walls of the reactor.[9]

The introduction of plasma-assisted CVD techniques (PACVD) might well constitute the most important milestone for the reduction of chemical hazards and environmental problems associated with semiconductor processing. Plasma approaches eliminate the need for using highly reactive and unstable gases (Table II, Group 3) and wet chemical etchants such as HF buffered formulations. These reagents are replaced by considerably more stable halohydrocarbon (Table II, Group 5) precursors, which are converted into highly reactive intermediates within the plasma. As indicated schematically in Figure 4, chemically but not environmentally benign CF_4 is used for the plasma etching of silicon.[10]

In a radio-frequency (RF) plasma, ordinarily inert, non-toxic CF_4 is converted to fluorocarbon radicals and F atoms. The latter chemically etches unmasked

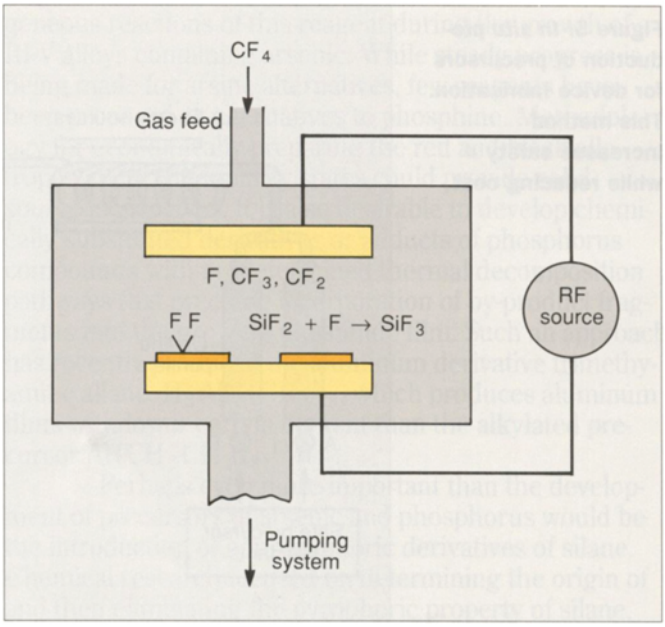

Figure 4. Plasma reactor for etching of silicon with CF_4. In it, potentially hazardous chemicals exist only briefly.

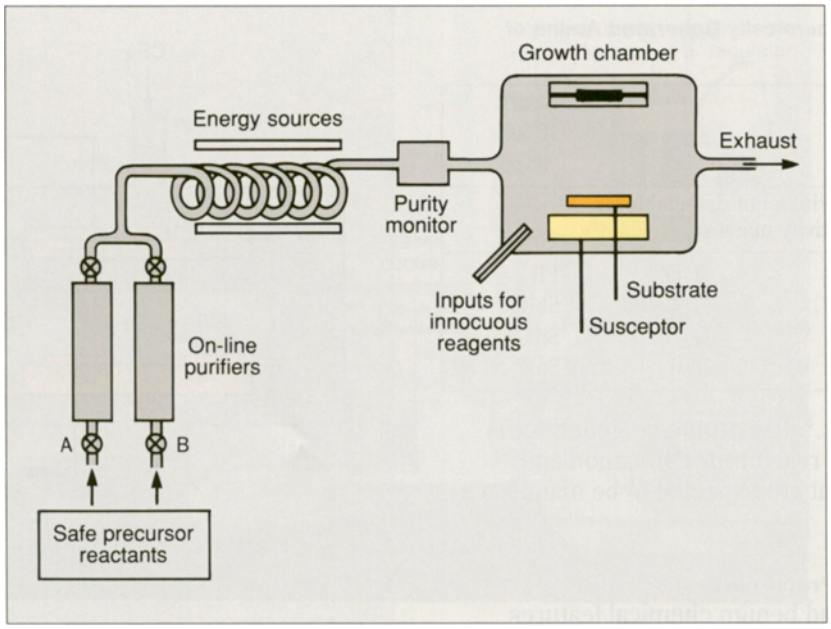

Figure 5. *In situ* production of precursors for device fabrication. This method increases safety while reducing cost.

areas of the silicon substrate. This use of plasmas as a chemical reactor for the transformation of otherwise inert precursors into reactive intermediates is a very promising approach to chemically and perhaps also environmentally benign processing. With this approach, potentially hazardous chemicals are generated *in situ* and exist only as intermediates with relatively short lifetimes inside of the plasma reactor system.

In a similar way, plasma-induced chemistry is positively affecting deposition of thin-film materials. PACVD is now used to deposit silica by reactions of tetraethylorthosilicate with oxygen.[11] This volatile silicon-containing liquid precursor is not pyrophoric and is easily converted into environmentally benign silica particles and ethanol. These properties greatly contrast with those of silane and its chlorinated derivatives. Plasma-induced chemical reactions also permit the deposition of high-purity films and powders. The preparation of halogen-, oxygen-, and hydrogen-free silicon nitride (Si_3N_4) has been demonstrated.[12] The reaction

$$3SiCl_4\ (g) + 2N_2\ (g) \xrightarrow[\text{plasma}]{\text{2450 MHz}} Si_3N_4\ (s) + 6Cl_2\ (g)$$

is exploited in the plasma reactor. Without the plasma, the pyrophoric reagents SiH_4 or SiH_2Cl_2 would be required to produce silicon nitride, which would be contaminated with hydrogen. Through judicious choices of alternative precursors, plasma-induced chemistry holds significant promise for safe processing and fabrication of devices.

Future Directions

The development of completely satisfactory, nontoxic, and environmentally benign alternatives for critically important precursor reagents for Si, As, P, In, Al, and Ga is not likely to be manifested for quite some time into the future. However, the next best alternative of

generating existing toxic precursors *in situ* is already on the horizon.[7] In addition, research shows promise of developing methods for tailoring the properties of benign reagents.

***In Situ* Reagent Generation.** The *in situ* generation of precursor reagents like arsine and phosphine, integrated with on-line purification and concentration monitoring constitutes the approach of the future for assuring consistent quality and quantity of reagents needed for device manufacture. A schematic diagram of this integrated approach is shown in Figure 5. As shown schematically, available low-toxicity reactants (preferably gases) are purified on-line and then induced to react by the application of an appropriate noncontaminating source of energy (electrons, photons, RF or microwave fields, etc.) to produce the desired product, which may ordinarily be a highly toxic reagent. The concentration of the *in situ* generated precursor is then measured and controlled for introduction into the reactor for device fabrication. This approach optimizes safety while reducing the costs of manufacturing by obviating the need for remote storage facilities. The possibility of leveling out or improving device yields by preventing fluctuations in reagent purity from cylinder to cylinder is inherent in the *in situ* generation procedure. Presently, *in situ* arsine is being tested for stringent device requirements. Fundamental studies of other homogeneous and heterogeneous chemical reactions are continuing to pave the way for new approaches for the *in situ* generation of other strategic device reagents such as phosphine.

Toxicology and Chemistry of Precursors. Investigations to pinpoint the chemical structure and toxicity property relationships of semiconductor precursors is warranted to tailor specifically the development of benign reagents for the future. As already observed for arsine, increased alkylation decreases toxicity until $As(CH_3)_3$ is nontoxic. Solving the accompanying problem of carbon incorporation into the depositing film as a result of the use of $As(CH_3)_3$ may well depend on detailed investigations of the physical chemistry of the homo- and heterogeneous reactions of this reagent during the growth of III-V alloys containing arsenic. While steady progress is being made for arsine alternatives, few reagents have been proposed as alternatives to phosphine. Methodology for economically preparing the red and black allotropes in ultrahigh-purity states could provide solid source alternatives. It is also desirable to develop chemically substituted derivatives or adducts of phosphorus compounds with predetermined thermal decomposition pathways that preclude incorporation of by-product fragments into the growing phosphide film. Such an approach has recently produced the aluminum derivative trimethyamine allane, $H_3AlN(CH_3)_3$, which produces aluminum films with lower carbon content than the alkylated precursor $Al(CH_2CH_3)_3$.[13]

Perhaps even more important than the development of precursors of arsenic and phosphorus would be the introduction of nonpyrophoric derivatives of silane. Chemical research focused on determining the origin of and then eliminating the pyrophoric property of silane, while retaining appreciable volatility and ease of conversion to epitaxial silicon, is worthwhile. Industrial and academic research efforts within the United States must also be expanded significantly to develop the new, low-cost, chemically benign precursors and environmentally innocuous processes that are required for semiconductor processing in the next century.

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Biographies (continued)

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