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► **To cite this version:**

Richard Walling, Rodney Moss. Safety and environmental aspects of CVD. Journal de Physique III, 1992, 2 (8), pp.1399-1411. 10.1051/jp3:1992104 . jpa-00248811

HAL Id: jpa-00248811

<https://hal.science/jpa-00248811>

Submitted on 4 Feb 2008

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Classification
Physics Abstracts
82.65

SAFETY AND ENVIRONMENTAL ASPECTS OF CVD

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(Received 1st February, accepted 15 April 1992)

Abstract: The use of toxic materials in many CVD processes means that careful attention to safety procedures in handling and using these materials is essential. This paper addresses the safety aspects of the CVD process as a whole and then discusses two particular areas in more detail. The possibility of using less hazardous materials in processes is considered and illustrated by recent developments in MOCVD. Techniques for scrubbing waste gases from CVD effluent is also reviewed and some of the key issues discussed.

1. Introduction

Chemical vapour deposition (CVD) processes frequently use a wide range of toxic and/or flammable materials. Recent technical developments have resulted in an increase and variety of the toxic sources and the waste products as newer materials are investigated. Furthermore, the applications of CVD are widening and many newer processes are now moving into production. This increase in toxic material usage poses greater safety challenges and risks which need to be recognised, quantified and addressed. These trends in technology need also to be seen in the light of changing regulatory positions in many countries, where tighter controls on toxic material, its storage, handling, use and disposal are being introduced. There is also now a growing increase in public awareness of safety issues, partly due to prominent reporting of major chemical problems, spillages or toxic gas leaks and also a general concern over possible environmental damage caused by the use of chemicals.

Early concerns over the handling of toxic materials related primarily to the safety of the operator. Safety procedures relating to the prevention of ingestion, inhalation and skin contact have been developed and in the field of CVD processing many good standard practices are being used. The safety procedures adopted for dealing with toxic gases and fumes have often relied on dilution in a forced-air extraction enclosure, so that the concentration of toxic gas in the exhaust under normal operating conditions is reduced to a level below the maximum exposure limit (MEL). However, simply to rely on dilution of toxic fumes is no longer considered to be an acceptable practice and means for removing toxic gases before venting to an extraction duct is now the preferred method. The method used for removing toxic fumes results in the formation of liquid and/or solid toxic waste products which themselves raise issues regarding final safe disposal. Clearly the techniques employed as part of the CVD process for removal of toxic waste will impact significantly upon this.

It is not possible to cover in detail all aspects of safety relating to CVD processes, so in this paper we will present a general review of CVD from a safety viewpoint and then highlight two particular areas

where recent developments have resulted in safety improvements. One is the use of alternative source materials which obviates the need for high pressure, highly toxic gases and the second area is to look at the various approaches to treating waste gases. Finally a few general principles will be drawn for assessing procedures and seeking to arrive at an optimum solution for any given situation. In view of the wide range of materials and CVD processes being used, we will take a general overview and then illustrate with a few specific examples from our own work in MOCVD.

2. CVD Systems and Safety Aspects

In any CVD process the primary objective is a technical one, in order to obtain deposited material of the right quality and with the sufficient control to make it suitable for the application required. Hence one is looking for an efficient process which will reliably produce the required product. In addition to the technical objective of the process, one is concerned to provide a process which is safe in operation, both for those using it and other people in the vicinity. In achieving such safe operation consideration also needs to be given to any adverse effect on the environment.

Safety procedures can be costly to implement and merely to add on safety devices or procedures without seriously considering their effectiveness, may not actually enhance safety. It is essential to look at the precise safety issues involved and address them for a given process and situation and then to make a judgement of what is a cost effective way of achieving good safety standards. It is easy to presume that safety issues relate just to the CVD equipment itself: the toxic sources used and the waste products from the process. However, to cover all the aspects of safety of CVD, it is essential to look at not just the process but also the materials, delivery, storage and how waste products are treated and disposed. In figure 1 there is a schematic diagram of the total CVD process. It has been subdivided into its components and the safety aspects relating to each part are given.

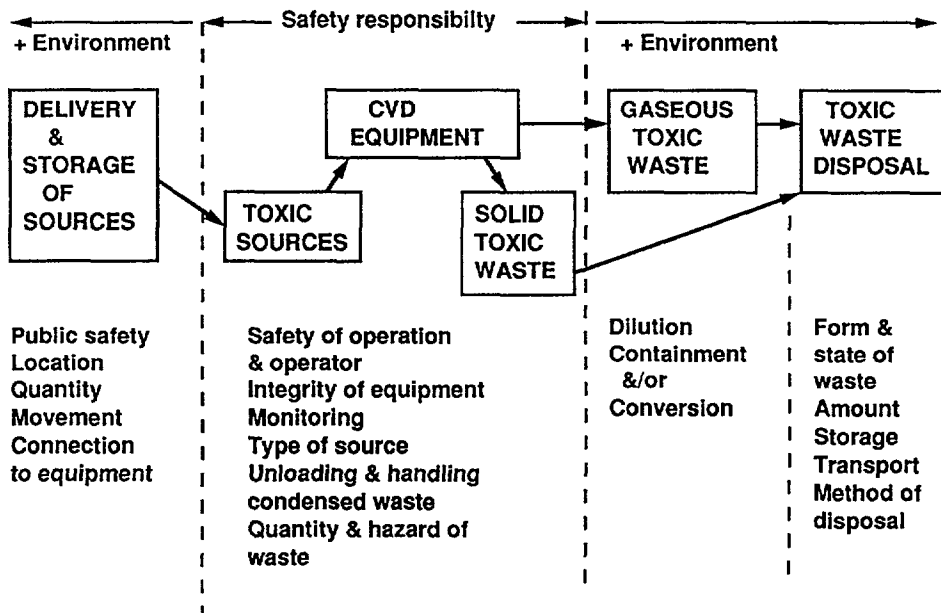


Figure 1. CVD Process.

Central to the safety assessment of the whole process is the actual CVD equipment, for it is the point of use for the toxic materials and is also the process/operator interface. The location of the equipment is important when considering the access and mode of operation for handling the materials involved. The key issues here are the chemistry of the process and the design and quality of the manufactured equipment itself, along with a detailed analysis of the consequences of process or system failure. For many processes, monitoring of the environment adjacent to the equipment is required to take account of fault situations where leaks of toxic material may occur. Forced ventilation of the air around the equipment is also desirable to provide protection for the operator when installing toxic materials or unloading product at the end of a process. In addressing the safety issues of this area, the types of hazard relating to the source materials and the nature and quantity of the waste products are paramount. In the case of gaseous toxic waste, a decision has to be made as to whether dilution is adequate or whether containment and/or conversion to a less hazardous and more stable product is required. Here the important issues are the nature of the waste product, its concentration, volume, state and also any storage on site needed until transport for waste disposal can be arranged.

Considering now the source input to a CVD system. In order to maintain a smooth operation especially in a production situation, sufficient source material must be maintained on site. Location of the storage site and its impact on the surrounding community and environment in the case of an emergency situation need to be considered particularly in the case of high pressure toxic gases. Where large amounts are being used, the quantity that can be stored is frequently limited by legislation. Transport of the toxic materials from the store to the point of use and the safe installation onto equipment are all issues which need to be considered and have been the subject of review documents in the past [1].

Safety responsibility of CVD processes covers all aspects, from the arrival of toxic materials on site to their final safe disposal, whether on or off site. Two areas have been the subject of development in recent months and these will now be discussed. The first is the use of less hazardous alternative source materials and the second, techniques for scrubbing toxic waste gases. In this paper we use the MOCVD of group III-V materials to illustrate the issues involved. In figure 2 there is a schematic diagram of an MOCVD system. In MOCVD of III-V materials, the sources are traditionally group III

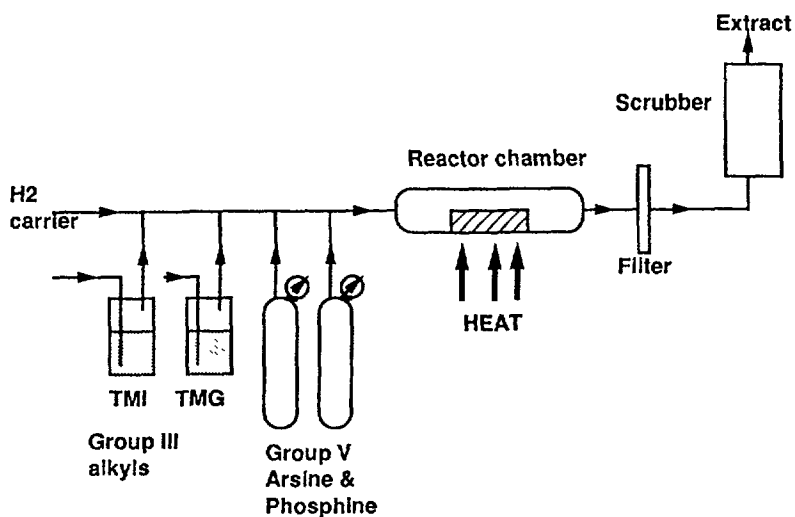


Figure 2. MOCVD of III / V material.

metal alkyls and group V hydrides (arsine and phosphine). These are metered in vapour or gaseous form into the reaction chamber where thermal decomposition takes place to produce the desired semiconductor layer on a single crystal substrate, eg. gallium arsenide or indium phosphide. Particulate solid waste products formed can be filtered out and unreacted arsine and phosphine pass through a scrubber before the carrier gas is vented into the extract system.

3. Safer Source Materials

The principal concern in MOCVD and in many other CVD systems is the use of the highly toxic group V hydrides. These are contained as high pressure gases in cylinders and have very low MEL values. Small leaks of these gases can present considerable safety hazards. Clearly the release of the contents of a full arsine or phosphine cylinder would have major impact, not only on the immediate location but also the surrounding environment. In residential areas this could well result in a major incident. In addressing the question of 'How can we improve the safety of the process?', one immediate issue is apparent, 'Do we have to use high pressure, highly toxic gases?'. Because the element arsenic is part of the final product, the hazards relating to it can never be finally eliminated. However, there are alternatives to the use of arsine and phosphine, eg. in-situ generation of the gases; units for producing arsine have been tried and tested [2]. This still utilises the highly toxic gas, but one has removed the problems of large quantities being stored under high pressure. An alternative approach is to use the controlled desorption from a saturated molecular sieve [3]. Here temperature is used to release the absorbed arsine from the sieve. This also removes the need for a high pressure cylinder of arsine but has the disadvantage that a limited amount of gas can be stored and this may not be suitable for a production environment. Furthermore, it is necessary to have an adequate method of recharging the molecular sieve when expended; this merely moves the use of the high pressure arsine from one point to another.

A third approach which has been showing considerable promise, is the use of organo-arsenic and organo-phosphorous compounds. For a vapour deposition system one requires a source material with reasonable vapour pressure in order to provide an adequate input to the CVD system. A number of different materials have been investigated and some are listed in Table 1. It can be seen that replacing hydrogen atoms on the hydrides with an alkyl group not only reduces the innate toxicity of the material, but also converts it from a gas to a liquid. Trimethyl arsine (TMAs) is considerably less toxic than arsine, however, its thermal decomposition characteristics are such that carbon may be incorporated into the layers. For many applications this is not acceptable, although it might be suitable for some processes. In the case of the other organo-arsines and phosphines listed, the LC50 value, although higher, is not substantially improved over arsine or phosphine. However, the improvements in safety due to the reduced risks of dispersion in the event of a leak, mean that there is a major safety advantage in using them. Furthermore, there are toxic gas monitors which are able to detect these materials as effectively as arsine and phosphine themselves. Besides those listed in the table there are other materials which have been investigated but full data is not available for them yet. We have investigated the use of TMAs, tertiary butyl arsine (TBAs) and tertiary butyl phosphine (TBP) in the growth of indium phosphide and compounds of gallium, indium, arsenic and phosphorus (GaInAsP) materials.

All three of these alternative source materials have been tested and found to produce good quality specular layers. In the growth of GaInAs using TMAs, background doping levels of carbon in the range $5-10 \times 10^{16}/\text{cm}^{-3}$ were measured using secondary ion mass spectrometry (SIMS). In the quaternary materials GaInAsP, carbon incorporation becomes less for higher phosphorus content compounds and for $1.3\mu\text{m}$ wavelength quaternary material the carbon incorporation was less than $4 \times 10^{16}/\text{cm}^{-3}$. For some devices, carbon incorporation makes TMAs an unsuitable source material, but for other devices where additional doping has to be introduced anyway, eg. in laser structures, then the presence of the carbon may not be such a problem. In table 2 we show the results of $1.3\mu\text{m}$ BH lasers using TMAs and phosphine. The laser structure is given in reference [4] where the active region of the laser was grown using TMAs instead of arsine. The results obtained for the devices are typical of that which can be obtained using the hydride.

Table 1 Replacements for arsine and phosphine

		LC50, ppm	
Arsine	AsH ₃	42	gas
Dimethylarsine	(CH ₃) ₂ AsH	164	liquid
Trimethylarsine (TMAs)	(CH ₃) ₃ As	>14000	liquid
Tertiarybutylarsine (TBAs)	(CH ₃) ₃ CAsH ₂	90	liquid
Phosphine	PH ₃	55	gas
Tertiarybutylphosphine (TBP)	(CH ₃) ₃ CPH ₂	>1100	liquid

Table 2 BH laser results using TMAs

1.3 μm BH laser:

421 devices tested

I_{th} (best) .. 8mA

I_{th} (mean) .. 13.2mA

Output powers >10mW @ 100mA

A more extensive investigation of the use of TBAs and TBP has been reported [5]. A summary of the results obtained for BH lasers is given in table 3. It can be seen that in most respects the performance of the devices made using the organo-arsine and phosphine precursors are equivalent to their hydride equivalents. It was discovered that the batch of TBP used for these particular experiments being an early batch from the suppliers, was not as pure as is currently available. Mircea et al have shown that excellent 1.3 μ m lasers can be produced using TBP [6].

Table 3 BH laser results using TBAs & TBP [5]

Comparison of InGaAsP ($\lambda = 1.55\mu\text{m}$) lasers:

	TBAs, TBP grown	AsH ₃ , PH ₃ grown
Threshold current, mA	20	10-16
Efficiency, mW/mA	0.1	0.18
Output @ 100mA, mW	6	17

Comparison of InGaAs ($\lambda = 1.67\mu\text{m}$) lasers:

	TBAs grown	AsH ₃ grown
Threshold current, mA	12-17	15-17
Efficiency, mW/mA	0.1	0.08-0.1
Output @ 100mA, mW	7-8	6-7

The references quoted are but a few of those using TBAs and TBP for the growth of device materials and currently many laboratories are replacing arsine and phosphine in MOCVD with these precursors. Continued investigation of alternatives is currently being pursued. The motivation being not only the safety aspects but also improved process characteristics through operation at lower temperatures and enabling better composition control. The whole field of chemical beam epitaxy (CBE) is offering new impetus for identifying new source materials since arsine and phosphine have to be cracked at high temperature before they can be used in that process. The thermal decomposition characteristics of these alternative source materials using organic precursors is giving an additional degree of freedom in controlling the process

These materials offer improvements in safety due to the reduced risks of widespread dispersion compared with high pressure toxic gases. However, it must be stressed that current data on their toxicity is very limited and one cannot assume that they are necessarily significantly less toxic to humans than their corresponding hydrides. Great care must be taken in their use in CVD systems.

4. Treatment of CVD Exhaust Gases

In most CVD processes, 100% utilisation of the source materials is never achieved, also, even when one is using some of the alternatives to the group V hydrides, reaction within the system can produce toxic gaseous products. Filtration of solids and particulates from the gas stream can be fairly readily

achieved adjacent to the deposition chamber. It is the treatment of residual toxic gases that we now address. There are a number of commercially available systems for this work. We will describe the principles of four of the more commonly used ones, and identify some of the issues which need to be considered. The principal methods are:

- * Incineration
- * Liquid scrubbing
- * Activated carbon absorption with and without oxidation
- * Dry reactive columns

Some of these techniques have been used in combination with each other to give a sequential treatment of gas effluent.

4.1 Incineration

In figure 3 a schematic of an incineration process is shown. Gases from the CVD system are passed through a pipe into a flame which burns continuously, in order to oxidise the waste products. In the case of arsine, arsenic oxide as a particulate solid is formed which can then be removed by a filter and the residual gas then passing into an extraction system. The particulate solids have to be removed periodically and are sealed in a disposable bag and then sent for waste disposal. This type of approach is being used in both research and production environments and seems to be a very effective way of dealing with waste. It does however require very precise procedures for handling the filter and its highly toxic dust. Such procedures can be devised and the system requires careful interlocking to ensure that there is a continuous flame burning. In principle it can be used to treat more than one system at a time since there is no interconnection between the gases outlets from each individual CVD system. Where large volumes of waste gas are to be treated, careful design of the incineration chamber is necessary to ensure total oxidation occurs.

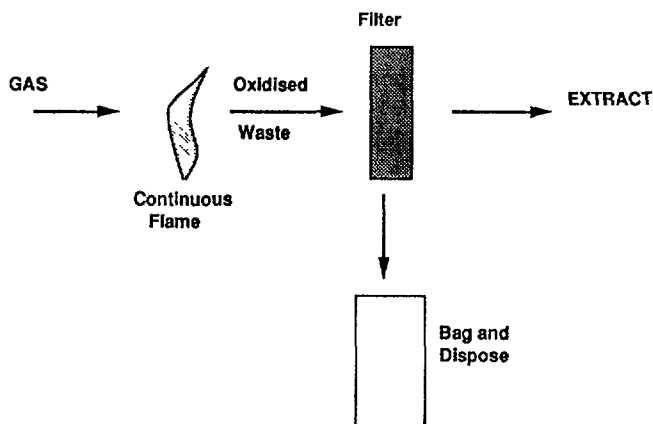


Figure 3. Incineration.

This technique is particularly effective for arsine, however phosphine produces phosphorus pentoxide which is hygroscopic. Since most systems operate with hydrogen as the carrier gas, and in any case one is burning hydrocarbons in the flame, there is always moisture present in the gas system which reacts with P_2O_5 to produce phosphoric acid. This can clog filters, creating back pressure problems and difficulties in removing waste products. Furthermore, phosphoric acid can cause corrosion problems in the system itself.

4.2 Liquid Scrubbing

Figure 4 shows a schematic of a typical liquid scrubber system. The principle of operation is that a reactive solution is contained within a vessel and the toxic gases are mixed with it; they react to form soluble and/or insoluble products which are contained within the solution. Efficient mixing of the gas with the liquid is achieved using spray columns and/or mesh filters to give close interaction of the gas with the reactive liquid. Such systems are able to cope efficiently with both arsine and phosphine and other toxic gases. They can also be used for production systems and there are a number of different chemical treatment fluids in common use. By the use of Venturi inlets it is possible to isolate several inputs from different CVD systems without causing any back diffusion of waste gas from one system to another. These scrubbers require one or more pumps which need regular maintenance and the solutions have to be replenished at intervals. In some cases this can result in large volumes of toxic contaminated liquids. Frequently the liquids themselves are corrosive in nature and therefore handling and maintenance procedures have to be carefully considered.

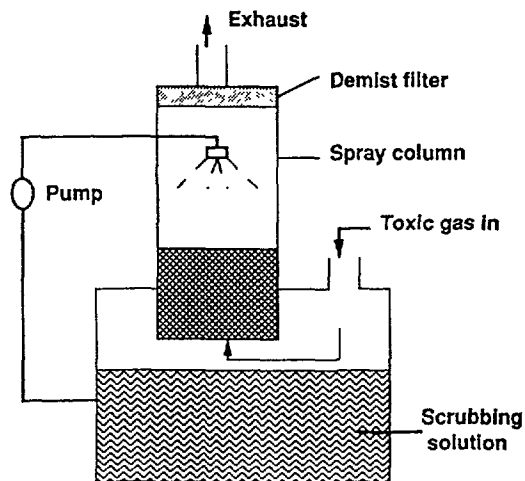


Figure 4. Liquid Scrubber.

4.3 Carbon Absorption

One method commonly used for treatment of waste gases is gas absorption using carbon normally chemically treated carbon with eg. copper and chromium oxides for the treatment of arsine and phosphine. Such carbon units operate both by physisorption and chemisorption of the species. Because of the convenience of being able to use a self-contained canister which

can be connected directly to the output of a CVD system and then disposed of when exhausted, it seems in principle to be a simple and convenient method of treatment. In operation there are a number of design considerations that need to be taken into account:

- * The volume of toxic gas to be removed relative to the volume of carbon absorbant
- * The packing of the carbon granules. We have experienced considerable variations of lifetime for canisters, almost certainly due to the different methods used and the ease with which tracking of the gas flow through the carbon occurs
- * The behaviour of the absorption characteristics with time

In figure 5 the measured output from a small carbon canister on an MOCVD system is shown schematically with time. We have observed that initially approximately 100% absorption of the toxic gas occurs, but after a period of usage, which depends on the volume of gas entering and also the size of the canister, a low level of toxic gas is measured at the outlet of the canister. We observed that this level of output continues even when the toxic gas on the input side is turned off. In other words, the carrier gas passing through the canister appears to entrain a small amount of toxic gas, making it a secondary source of toxic gas. This level does not seem to rise significantly, until the canister becomes totally exhausted when a rapid rise at the output occurs.

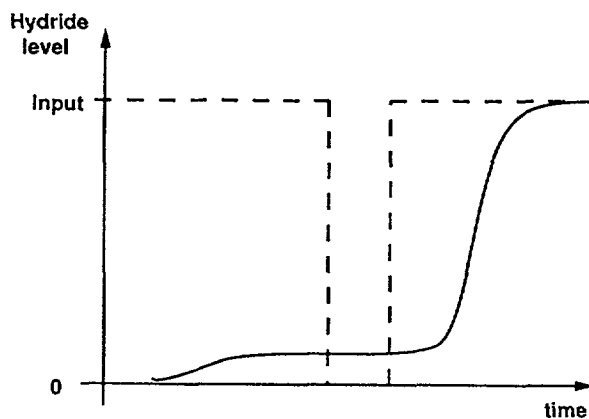


Figure 5. Carbon absorption with time.

The low level output can be removed by inserting a second canister downstream. It appears that initially the chemically treated carbon chemisorbs the toxic hydride, but once that surface has been used up then physisorption occurs, which is less efficient, hence the carrier gas sweeps a small amount off continually. It should be noted that most of these activated carbons are designed to be operated with oxygen present in the gas stream. Many CVD systems operate with hydrogen as a carrier gas and oxygen has to be avoided because it is a contaminant. However, by introducing a low level of oxygen downstream through the carbon, the chemical reactivity can be partly regenerated. Two methods of doing this have been used:

One is to periodically recharge the canister when the system is not being operated; and the other is to introduce a very low level of oxygen continuously into the waste gas stream.

The latter process requires very careful control to ensure that the lower explosion limit for hydrogen:oxygen mixtures is not exceeded and oxygen contamination of the system does not occur. Where phosphine and arsine have been used, we have observed that this oxidation process can generate considerable quantities of heat and therefore the oxygen flow needs careful control. If small canisters are being used, oxygen moving through the carbon canister generates heat, so the toxic gas can be desorbed ahead of the incoming gas front due to the temperature rise. This results in toxic gas being released from the outlet of the canister during the process.

There are now available, larger commercial units employing a carbon absorption approach. By using a larger volume of carbon and increasing the path length of the carbon from the entrance to the exit of the canister, it is possible to minimise the problems mentioned above. In figure 6 there is a schematic diagram of such a unit. The waste gas is introduced at the bottom and passes into the carbon. As the canister is then treated with oxygen to fully oxidise the hydrides, so the temperature rise caused by the reaction can be monitored using thermocouples positioned at regular intervals along the length of the canister. It is also possible to ensure that the carbon can be replaced before final punch through of the gas occurs, since the movement of the temperature front gives an indication of the degree to which the carbon has been exhausted.

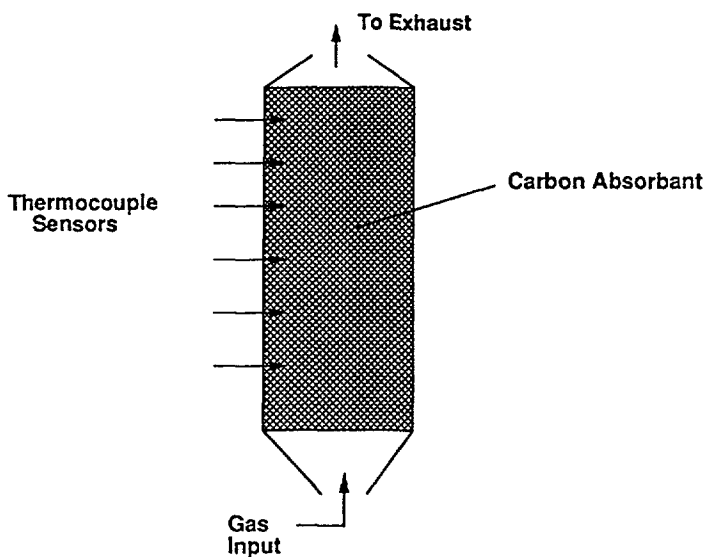


Figure 6. Schematic of large carbon absorption system.

We have used one such system in our laboratories, containing a 25kg charge of carbon and in a period of 10 months it has absorbed approximately 1kg of combined arsine and phosphine. According to the manufacturer, it is expected that the column should be able to cope with at least 2kg of hydride before the charge needs to be replaced.

Such systems are in principle simple in operation, but in cases where the charge is being replaced from a static container, it is necessary to have good safety precautions to deal with any toxic dust that is generated as the carbon granules are removed. Unlike the incineration and liquid scrubbing techniques, it is more difficult to use this type of system for more than one CVD outlet. Where one wishes to use a single carbon scrubbing unit for treating the exhausts from several CVD kits, careful thought must be given to isolation of the lines so that back diffusion or back pressure, from one system to another, does not occur.

4.4 Dry Reactive Columns

As we have mentioned in the previous section, it is not only important to provide an efficient method of toxic removal, but also to try and ensure that the waste products produced by the scrubber system are in a least hazardous form. In figure 7 there is a schematic diagram of a dry reactive column used for removal of hydrides. The precise chemical composition and process is still subject to commercial confidentiality, but the principle is to convert arsine and phosphine to an arsenate or phosphate which is fused into the host matrix. The unit consists of a column containing the active constituents which is positioned inside a tube heater and maintained at a temperature of $\sim 550^{\circ}\text{C}$. The toxic gas enters the bottom and as it passes through the heated column is converted into the arsenate or phosphate. The system can be used until either the chemicals in the column are exhausted or the back pressure build up as the column becomes filled, rises to a point which is unacceptable for the CVD process. On exhaustion the entire cartridge is removed and replaced with a new one without the necessity of emptying and refilling the contents.

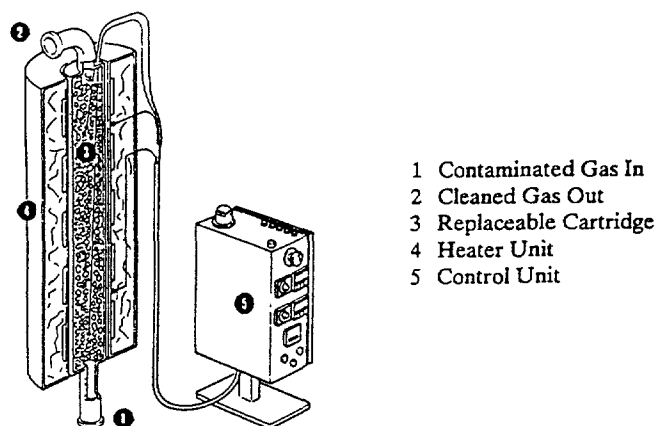


Figure 7. Dry Reactive Column.

We have tested such a unit on an MOCVD system using a standard cartridge which was used for 10 months before being changed for operational reasons. During this time the hydride level was monitored in the exhaust duct just above the outlet from the scrubber. The level remained below 17ppb of arsine, the sensitivity limit of the toxic monitor used. It is difficult to measure the precise volume of gas passed into the unit, but we estimate that about 850l of phosphine and arsine mixture passed through the unit before it was changed.

A second column specially modified for the use of phosphine and arsine has been in use without any back pressure build up and approximately 700l of hydride have been passed through it to this time. Currently we have no information on its suitability in a production environment but we understand that tests are in progress. Whilst this system does have the advantage of convenience and simplicity in use, it requires a continual supply of power to maintain the temperature. The thermal lagging on the heater element is such that in the event of a power failure the cooling rate of the column is unlikely to prevent it remaining operational for the short period during which toxic gas may be purged out of the CVD system.

From this brief survey of systems available for scrubbing toxic gases it becomes apparent that there are a number of options available for any given situation and there is no universal solution. Each system has to be considered according to the nature of the gases being used, the location, the volume, the frequency of use and whether a single, central unit is desirable rather than a number of separate units for each CVD system. In arriving at the appropriate solution one needs to ask what is required of an ideal scrubbing system. The key points are:

- * Effective removal of toxics
- * Conversion of toxics to a stable form
- * Waste in a form that is easy and safe to handle
- * Low risk to the operator when recharging the unit
- * Minimum maintenance and low cost operation
- * Ease of waste disposal

By bearing in mind these issues one can select a system which is most suited for a given application and location.

5. Summary

In this paper we have sought to outline some of the main issues regarding safety in using CVD systems and we have addressed two particular areas where new developments are occurring. In view of the toxic nature of many of the chemicals used in CVD processes, it is essential that one takes a responsible approach not only to the safety of operators, but also to the general use and disposal of waste products, for these have significant environmental impact. The increasing attention and importance of taking environmental issues into consideration provides opportunity to review the way that CVD is approached and carried out in many laboratories. We have sought to indicate the breadth of the issues involved; it is not solely the chemical process that must be considered and therefore we suggest 5 principles for reviewing processes which use toxic materials:

1. Assess the scale of the problem.
What materials are being used? What is the nature of the hazards they present?
What quantities and concentrations are involved?
2. Review the use of alternative materials
Is it essential to use such toxic materials? Can the hazards be reduced by choosing an alternative source?

3. Optimise the efficiency of use of material.
How efficient is the process in terms of its usage of these toxic materials? Can the equipment design be altered to give greater efficiency? Low efficiency means larger quantities of waste to be handled and safely disposed.
4. Consider the form of the waste product
Can the waste products be converted into a less toxic or less hazardous state by some conversion process at the point of use? Can we limit the volume occupied by the hazardous waste?
5. Adopt a comprehensive view of the process
Consider all the safety aspects from the arrival of the source materials on site, right through to the product leaving the site and to the method of waste disposal off site.

Whilst this review has inevitably been limited in the scope and depth to which different issues could be covered, it is our hope that the issues raised will stimulate consideration of all aspects of the processes being used and result in a more systematic and safer approach to the use of CVD.

The work described in this paper represents our best understanding of current practice. British Telecommunications plc. can accept no liability whatsoever for any consequences which might arise from the use of any information contained herein or for any omissions.

6. Acknowledgements

The authors would like to express their appreciation for the useful discussions held with colleagues at BT Laboratories, with other workers in the field and also with commercial suppliers of toxic waste management systems.

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