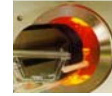


3.1 Introduction

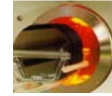
In the previous chapter, it has been analyzed the most appropriate conditions and geometries that could provide the optimum working conditions, not only on the modal properties of integrated optics devices, but also on attenuation or even in its measurement. It is, without any doubt, extremely important to know the processes involved during the fabrication of these devices, since the limit on the dimensions of any optical device is given by technological aspects. Basically, it will be worthless to design a 4μm thick core if technology could not provide it.

The most important requirements to any layer suitable to be used in integrated optics application are: 1. **Transparent.** By no ways can be designed an integrated optical device working at wavelengths where the material is not transparent. 2. **Linear.** In order to avoid birefringence effects (crystalline and polycrystalline materials are generally undesired, although there are exceptions, as could be $LiNbO_3$ or some III-V and II-VI compounds) 3. **Homogeneous.** A slight variation of this magnitude could represent a variation on the refractive index. 4. **Able to be polished** together with the substrate. As it will be seen, the light injection on the optical devices is done by end-fire coupling, which requires the cross-section of the input waveguide to be polished. Hence, if the light guiding material is too soft (as could be, for example, a chalcogenide glass over a silicon substrate) it cannot be appropriately polished, since the process requirements for obtaining a mirror-quality at the chalcogenide glass are not so for silicon. As a rule, materials involved on the integrated optics devices fabrication should have comparable hardness. 5. **Low impurity level.** It is known that impurities generally acts as scatter or absorption centers, causing an increase of the losses. We will see, however, that in some materials, the impurity contents is the responsible of having similar materials with different refractive index value. 6. **Chemically stable.** Since one of the main applications of the devices designed in the previous chapter was in the field of sensors, it is extremely important that the transducer itself does not suffer from aging. 7. **Adherence**. Layers are expected to have good adherence both at the surface and between them so as to allow obtaining stiff structures. Moreover, their mechanical properties, specially their thermal expansion coefficient, have to be as close as possible

in order to avoid cracking during the fabrication process. 8. **Uniform.** Although this is not so critical as compared to the previous requirements, a variation of the core dimensions would signify a modification of all the guiding properties of the structure. If this variation is small, however, it would only mean an increase of the total losses that, although still is undesirable, it could be afforded.

In table 3.1, a brief review from the literature concerning the several materials employed on the fabrication of both ARROW and TIR waveguides is given. The ways of obtaining these materials are completely different, ranging from Molecular Beam Epitaxy (MBE) to Low Pressure Chemical Vapor Deposition (LPCVD). The reason why selecting an specific material instead of another is simply given by its availability. The clearest example is given in this work: it is known that silicon is absorbent at visible range frequencies, then, it could have been chosen several other compounds transparent in the visible range for obtaining the same devices. However, the main advantage of silicon is the compatibility to standard microelectronics technology. Electronical, optoelectronical and optical circuits can be implemented on one substrate. Moreover, the high experience of the Centre Nacional de Microelectrònica (CNM-IMB-CSIC) on the fabrication and characterization of silicon and silicon compounds, has motivated the use of silicon as the basis of our research. Moreover, and concerning the possible future perspectives of this work, it will be relatively simple to design a photodetector on the silicon substrate, reducing fabrication costs and increasing the device reliability.

During all this chapter, we will focus on the technology requirements that allows obtaining integrated optical devices, that is, layer growth and deposition, photolithography and etching. Concrete application of the systems analyzed to the materials and geometries described in the previous chapter will be given.



	Substrate	d_c	d_1	d_2	λ (µm)
TIR					
[1]	Si	As_2S_3			1.3
[2]	Si	SiN			1.3
[3]	Si	$Si_{1-x}Ti_xO_2$			1.3
ARROW-A					
[4]	InP (n=3.16)	InP (n=3.16)	InGaAsP (n=3.553)	InP (n=3.16)	1.55
[5]	Si	SiO_2 (n=1.46)	TiO_2 (n=2.3)	SiO_2 (n=1.46)	0.633
[6]	Si	Si	GeSi	Si	1.33
[7]	Si	SiO_2 (n=1.46)	TiO_2 (n=2.3)	SiO_2 (n=1.46)	0.633
	Ge	NA45 (n=1.54)	Si_3N_4 (n=2.0)	NA45 (n=1.54)	
	GaAs	C7059 (n=1.54)	ZnO (n=1.98)	C7059	
	InP		Al_2O_3 (n=1.65)	(n=1.54)	
			ZrO_2 (n=1.92)		
			Ta_2O_5		
ARROW-B					
[6]	Si	Si	SiO_2	Si	1.33
[7]	Si	NA45 (n=1.54)	SiO_2 (n=1.46)	NA45 (n=1.54)	0.633
	Ge	C7059 (n=1.54)	CaF_2(n=1.43)	C7059	
	GaAs	TiO_2 (n=2.3)	Fluored glasses	(n=1.54)	
	InP	Si_3N_4 (n=2.0)		TiO_2 (n=2.3)	
		ZnO (n=1.98)		Si_3N_4 (n=2.0)	
		Al_2O_3 (n=1.65)		ZnO (n=1.98)	
		ZrO_2 (n=1.92)		Al_2O_3 (n=1.65)	
		As_2S_3 (n=1.87)		ZrO_2 (n=1.92)	
				As_2S_3 (n=1.87)	

Table 3.1: Different materials employed on the fabrication of TIR, ARROW-A and ARROW-B waveguides.

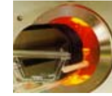
3.2 Layers for Integrated Optics Applications

There exist several system configurations from which it is possible to obtain material layers. The fact that growth and deposition are separately discussed arises from the role that the substrate plays during the process required to obtain the film. If it plays an active part, that is, if vapor species chemically reacts with the substrate, it is called layer growth and includes the thermal oxidation and nitridation. On the contrary, if silicon is a mere substrate and does not interact with the vapor species, it is labeled as layer deposition and can de divided into three subcategories, depending on the primary mechanism that causes the layer to grow. **1. Evaporative methods**. Processes that involves the evaporation of a solid material source at very low pressures and the subsequently deposition of the layer, due to condensation, on the substrate surface. **2. Gas-phase chemical processes**, in which chemical compounds in vapor phase react near or at the substrate surface, forming a solid film on it. **3. Liquid-phase chemical techniques**, including spinning, immersion and spraying of a liquid on the substrate which, after being dried, forms a solid film. As a rule, The material properties, as could be density, impurity contents, mechanical stress and uniformity, depend on the deposition system. The same material can be obtained by several techniques and each one will provide the layer different properties. Thus, parameters such as reproducibility, uniformity, quality, substrate adherence and even cost should be considered in order to choose the optimal deposition system.

Although it would be extremely interesting to review all the possible configurations within the three previously mentioned deposition methods, we would restrict ourselves to the study of these processes that has been used during the fabrication of integrated optics devices.

3.2.1 Evaporative Methods

Different kinds of thin films can be deposited by Thermal Evaporation, Sputtering or Molecular Beam Epitaxy (MBE). All three are examples of Physical Vapor Deposition (PVD). PVD reactors mostly uses a solid as a material source. If the thermal energy is supplied to the evaporated material by ways of a resistance, RF, laser



or electron beam, the process is included in the thermal evaporation category. On the contrary, if the source material is transferred to a gas by target ionic bombardment, the process falls into the sputtering category. Since the latter has been the unique technique employed on the integrated optics devices fabrication (concretely for Al deposition), only this process will be studied. A more detailed explanation of PVD processes can be found in **[8]**.

Sputtering

Sputtering is the preferred PVD technique due to a wider choice of materials to work with, better step coverage and better adhesion to the substrate. Several sputtering configurations are available. The simplest one can be seen in table 3.2. Plasma composed of a noble gas (e.g., argon) is generated between the target and the wafers. Then, a high potential is applied across this setup. The highly ionized atoms in the plasma accelerate towards the target material, physically knocking off atoms from the target, which are then deposited onto the substrate. Since argon plasma is used, some argon is incorporated into the deposited film. In this process, the purity of the deposited material is mostly determined by the purity of the target. The wafers are mounted above the target, and batch deposition is possible using various substrate holder configurations.

As most of the atoms removed from the target are neutral, being in an excited state with high momentum, the sputtering may be regarded as a transfer process of atom linear momentum rather than heat transfer. So, it is a low temperature deposition system and neither the source nor the substrates are generally heated during the process.

During bombardment, other energetic particles such as secondary electrons, secondary ions/photons and X-rays are created at the target and could affect the growing film and/or influence its properties through defect generation caused by heat, radiation or chemical reactions.

Sputtering			
		Temp. (ºC)	Pressure (Pa)
		Room Temp.	0.1-10
Reactor properties	**Advantages**	**Disadvantages**	**Materials**
Cold Wall. Improve uniformity using rotatory reactors. Possibility of continuous deposition.	Alloy composition tightly controlled. Low changes in source material. Good uniformity over large areas. Good adhesion. Film properties controlled by bias and pressure.	Possibility of incorporating impurities. Substrate heating due to secondary particles. Ionic bombardment damage.	Almost unlimited. Specially used for metals and alloys deposition.

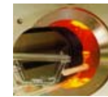
Table 3.2: Standard configuration and general properties of a sputtering system.

3.2.2 Gas-Phase Chemical Processes

Methods of layer formation using only chemical precursors in gas phase include thermal growth and vapor deposition processes. As it will be discussed, the former provides high purity oxide films with a high quality Si/SiO_2 interface, but it is a very limited technique. On the contrary, the main features of deposition techniques are its versatility for synthesizing both simple and complex materials with controlled properties.

3.2.2.1 Thermal Growth

Thermal growth is a chemical process in which the substrate provides part of the necessary elements to form a new layer. The most significant example is the growth of silicon dioxide (SiO_2) over silicon substrates. This process is the key point on the planar silicon technology: patterning a silicon oxide layer allows that, during the doping



process, impurities are only diffused in the desired areas. Moreover, due to its excellent insulator properties, it is also used for superficial passivation and for shielding devices.

Thermal oxidation is obtained when silicon substrate is heated at high temperatures (700ºC-1250ºC) in a gas stream. The equipment used can be schematically seen in fig. 3.1. It consists on a resistance-heated furnace containing an open quartz tube and a gas flow regulator system. The substrates are placed vertically and closely stacked in a quartz boat. Temperature can be controlled up to ±0.5ºC, in order to assure extremely uniform layers.

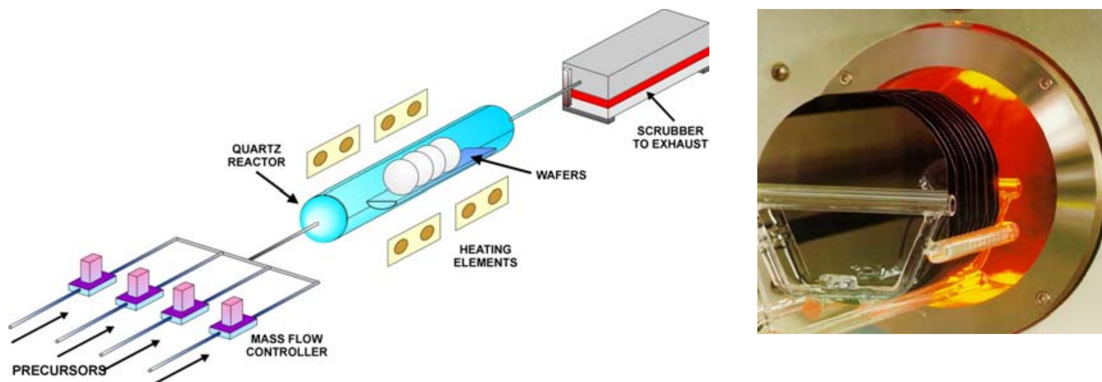
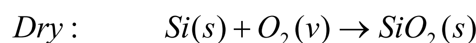
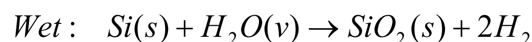


Fig. 3.1: Scheme of the standard thermal growth system and a detailed picture of the silicon wafers entering into the quartz reactor.

Silicon oxide films are labeled *dry oxides* or *wet oxides* depending if the precursor gas stream contains oxygen or water (generated by boiling deionized water or burning hydrogen in an oxygen atmosphere). The basic reactions that provide both oxides are given by



$$Dry: \quad Si(s) + O_2(v) \rightarrow SiO_2(s)$$



$$Wet: \quad Si(s) + H_2O(v) \rightarrow SiO_2(s) + 2H_2$$

When a silicon substrate is under an oxidant atmosphere, a thin layer fastly grows at a speed that is limited by the reaction ratio between the oxidant and the silicon. As the silicon oxide layer is getting thicker, the growing speed is limited by the necessity of diffusion of an specie through the layer. Using isotope ^{18}O, it has been possible to demonstrate that are the oxidant species those that diffuse through the layer until they reach the Si-SiO_2 interface. Hence, during the oxidation process, silicon is partially consumed. If a silicon oxide layer of thickness t is grown, the Si/SiO_2 interface

is placed at 0.45t of its original position. The interface is not at 0.5t since silicon and silicon dioxide have different densities and molecular weights that gives rise to a volume expansion.

Comparing both processes, it has been shown **[9]** that wet growth is faster than dry growth, since the diffusivity and solubility of water is much higher than those of O_2. As an example, for growing a 1µm thick silicon dioxide layer at 1100ºC, it would be necessary 2.3 hours in water vapor and 40 hours in oxygen. Since the basic ARROW-A structure has a 2µm-thick second cladding, wet growth arises as the optimum process for obtaining so thick layers.

Concerning their optical properties, thermal oxides present a refractive index value of 1.46 @ 633nm. It can be slightly varied by the addition of dopants during the growth process. However, the near invariability of the refractive index, together with its growth conditions (high temperature), forces the use of a second type of process, in which silicon only plays the substrate role onto which layers are deposited.

3.2.2.2 Deposition Processes: Chemical Vapor Deposition (CVD)

An alternative, more flexible process that allows obtaining layers bases its principle of operation on the fact that all precursors will be delivered form an external source, being them possible to be gaseous, liquid or solid. However, the deposition technique from a vapor phase (chemical vapor deposition, or CVD) is by large the preferred technique, since it can be easily transferred to mass production without significant changes on its working parameters. Moreover, it can be used for coating irregular or even internal surfaces and it has a good control on the properties of the final layer, as could be homogeneity, uniformity, stoichiometry and refractive index. The most important issue on these kind of processes is the fact that all residual products from the reaction must be volatile, in order not to contaminate the layer, which is the single reaction product in solid phase.

The formation of a solid layer on top of a substrate with this deposition system occurs due to the chemical reaction of precursors in its gas phase near or even on the substrate surface. As a rule in all the CVD techniques, temperature, pressure, precursor flow rates, reactor geometry and operating principle determine the deposition rate and

the layer properties (including adherence to the substrate and/or underneath layers). It is extremely important, as previously mentioned, that all the reaction products to be volatile at the deposition conditions so as to assure film purity. Properly speaking, CVD processes have to proceed by a heterogeneous reaction. If this is not so and homogeneous reaction is produced in the gas phase, it will cause a particle nucleation, which may form powdery deposits, leading to layers with particle contamination instead of the expected clean and uniform layers.

As compared to other deposition techniques, as could be PVD or thermal growth, it has several significant advantages, being the most important of them: **1. Precursors:** Materials deposited by CVD covers a huge variety of compounds with different properties. Inorganic, organic and organometallic precursors are used so as to obtain isolators, semiconductors and conductors. Gas phase reactants are preferred because they can be easily measured and uniformly distributed in the reactor. On the contrary, liquid and solid precursors must be vaporized without decomposition at suitable temperatures and transported, generally using a inert carrier gas, through conveniently heated pipes to the reactor. This process, although effective, hardens the overall processing. **2. Quality:** Deposition conditions are chosen so as to be as close as possible to equilibrium conditions. This fact assures the layers to have the best possible quality. However, deposited silicon oxide layers have lower quality as compared to these obtained by thermal growth. **3. Complementarity:** With CVD technique, it is possible to obtain layers that decompose during evaporation and thence cannot be deposited by PVD (as could be some III-V and II-VI compounds). **4. Vacuum:** It does not require, except LPCVD and PECVD systems, vacuum conditions. **5. Substrate:** As compared to another deposition techniques, there exists a complete flexibility on the substrate to be used. Neither conducting nor ionized materials are required. **6. Temperature**: Normally, materials deposited at low temperatures (i. e., below 600ºC) are amorphous. Higher temperatures tend to lead to polycrystalline phases. Finally, at very high temperatures (900ºC-1250ºC for silicon) the absorbed species have a high mobility, which allows obtaining a high degree of crystallographic perfection in the growth layer. The orientation of these films is according to the substrate crystal