A Waveguide Material for Integrated Optical Sensors: Silicon Oxynitride

H. Albers, L.T.H. Hilderink, J.H. Berends, K. Wörhoff,
N.F. van Hulst and P.V. Lambeck
MESA Research Institute, University of Twente, P.O.Box 217, 7500 AE
Enschede, The Netherlands
E-mail: H.Albers@el.utwente.nl

Abstract: SiO_xN_y is a flexible material for making planar waveguide structures for sensors. This material can be deposited with different technologies; PECVD and LPCVD are described. Layer properties of films produced with these technologies are presented. High optical transparency, tunable refractive index and high uniformity are the main characteristics. The layer properties of PECVD films can be improved by annealing. Besides optical loss from scattering also the absorption due to hydrogen is reduced. The development of the application of SiO_xN_y in devices is shown. Finally the relation between uniformity and device performance is discussed.

Introduction

Silicon oxynitride (SiO_xN_y) is a flexible material for making planar waveguide structures for sensors. The material is transparent in a wide wavelength range from the UV region up to the near infrared. Therefore it can be applied in the field of integrated optical sensors, which operate mostly in the visible region as well in devices for telecommunication, which operate in bands around 850 nm, 1300 nm and 1550 nm. The thickness of SiO_xN_y films can be varied from several tenths of nanometers up to several tenths of micrometers without a significant change in other properties like structure and texture, and refractive index. Very important for integrated optic devices is the uniformity in thickness and optical properties over large areas. An adequate uniformity can already be reached and further improvement is expected. The most conspicuous flexibility is the tunable refractive index from 1.46 - 2.0. This offers the device designer another degree of freedom to optimize his device. The most important applications of this freedom are:

- Optimizing the extension of the optical field outside the waveguiding layer
- Optimizing the field strength on the interface of the waveguiding layer
- Match the refractive index with that of another material in the device
- Optimizing the index contrast between waveguiding and gladding layers
- Optimizing the dimensions of the waveguide

Proceedings 1996 National Sensor Conference,Delft, The Netherlands, March 20-21, 1996.

Of course the mentioned properties are strongly interconnected. Another degree of freedom is therefore so important. The first two aspects determine how strong a waveguide senses the "outside world". They are very important for sensors, especially chemical sensors. The last two are important for connecting the integrated optics device with other optical components, semiconductor lasers, detectors and fibers.

Another advantage of the flexible refractive index is that within the same technology layers with different refractive indices can be easily combined in a device without compatibility problems that can be expected if different technologies have to be used.

For the shaping of channel waveguides and other structures a well-developed number of wet and dry etching techniques are available. Accurate dimensions and sharp edges can be obtained.

The application of SiO_xN_y in integrated optic devices started in the mid 80's with the use of Si_3N_4 waveguides[1],[2]. Shortly afterwards several other groups started with SiO_xN_y waveguides[3],[4],[5]. In Germany this work was stimulated in the framework of a stimulation program "Mikrosystemtechnik" with a sub-program for integrated optics on silicon[6]. Many of these activities were focused on sensors[3],[7],[8],[9].

Table I. Source gases used in CVD processes

Element	PECVD	LPCVD
Si	SiH_4	SiH_2Cl_2
N	N_2, NH_3	NH_3
O	N_2O, O_2	N_2O, O_2

Deposition Technologies

Silicon oxynitride can be deposited with different technologies. Plasma enhanced chemical vapour deposition (PECVD) and low pressure chemical vapour deposition (LPCVD) are the most extensively used technologies. Both technologies have been used for integrated optics devices. The layer growth is based on a chemical reaction between gaseous precursors that are mostly first absorbed on the substrate surface. An overview of the source gases is given in table I. The reaction mostly takes place only if an energy source is available to overcome an activation barrier. Different types of energy sources used in CVD processes are shown in Table II. We will give a short introduction to both and then we compare them.

Table II. CVD processes

Process	Acronym	Energy
Atmospheric CVD	APCVD	thermal
Low pressure CVD	LPCVD	thermal
Plasma enhanced CVD	PECVD	electrical
Photo CVD	Photo-CVD	UV-light
Laser CVD	LCVD	UV-light IR-light

PECVD

In plasma enhanced CVD (PECVD) the process gasses are feed through a plasma. Molecules are ionized, dissociated and activated. The resulting species are more reactive than the process gasses and can perform a reaction to SiO_xN_y. A PECVD reactor is basically a vacuum vessel with two electrodes that are connected to a RF power supply. There are two general used designs (Fig. 1 and 2).

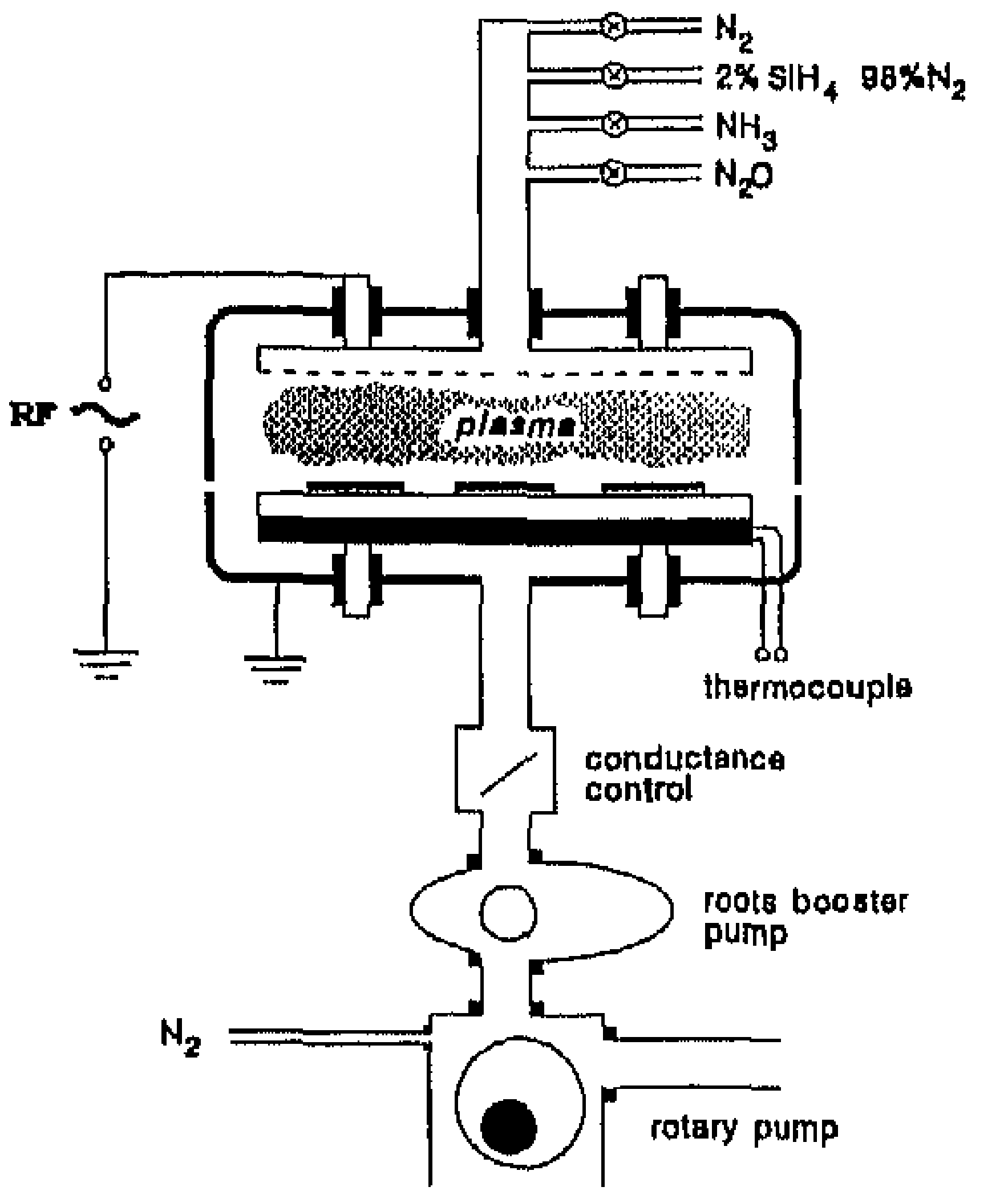


Fig. 1. Parallel plate PECVD reactor

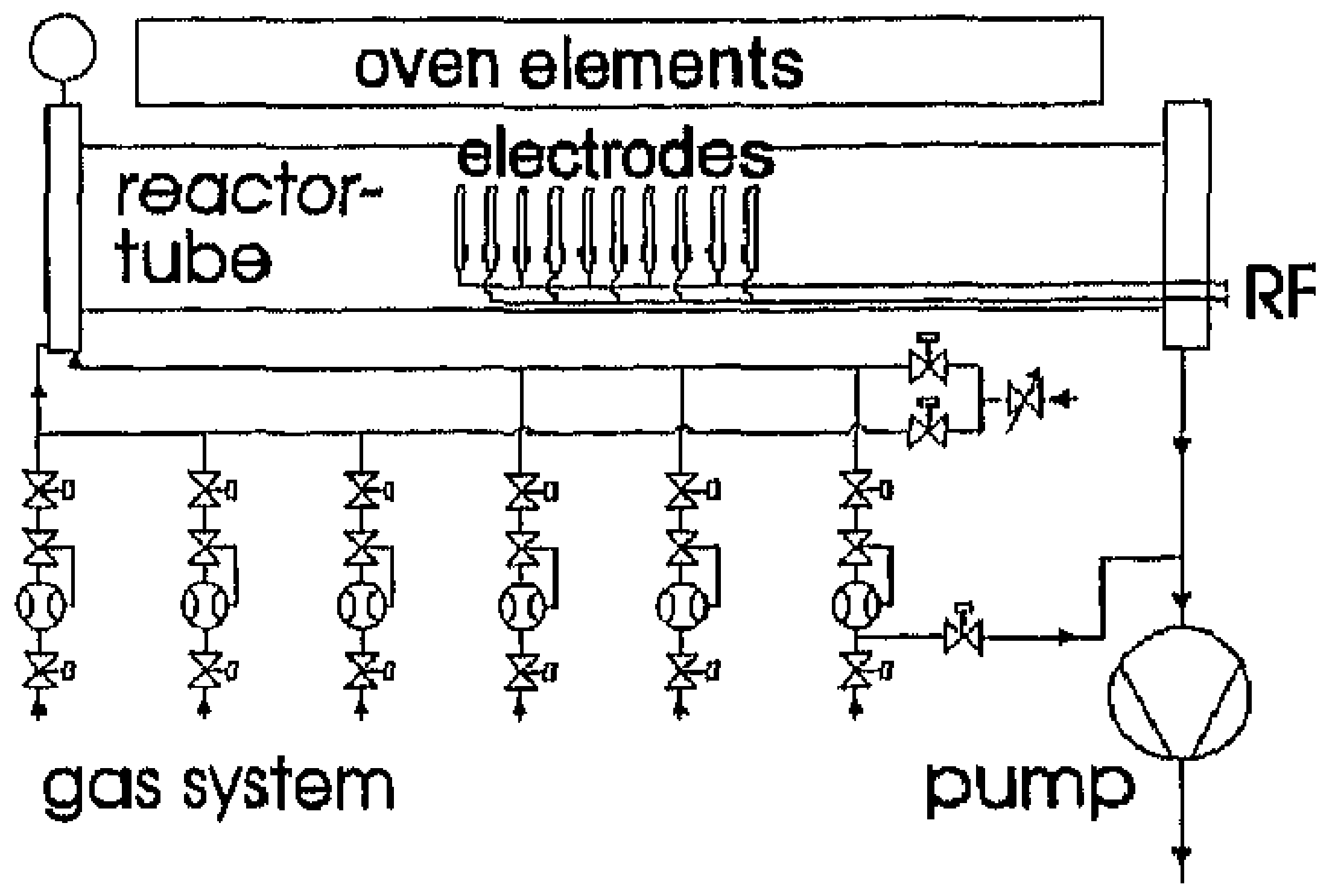


Fig. 2. Hot Wall PECVD reactor.

In both reactors the substrates are heated to 300-380 °C. The parallel plate reactor is operated with a RF frequency of 13.56 MHz or about 200 kHz, while the Hot Wall reactor in only operated in the range 50-200 kHz.

A new type of reactor using a high density plasma source, ICP, ECR or microwave, has been used incidentally. These reactors are developed mainly for reactive ion etching (RIE) but may have also advantages in deposition rate, layer composition and uniformity for deposition.

LPCVD

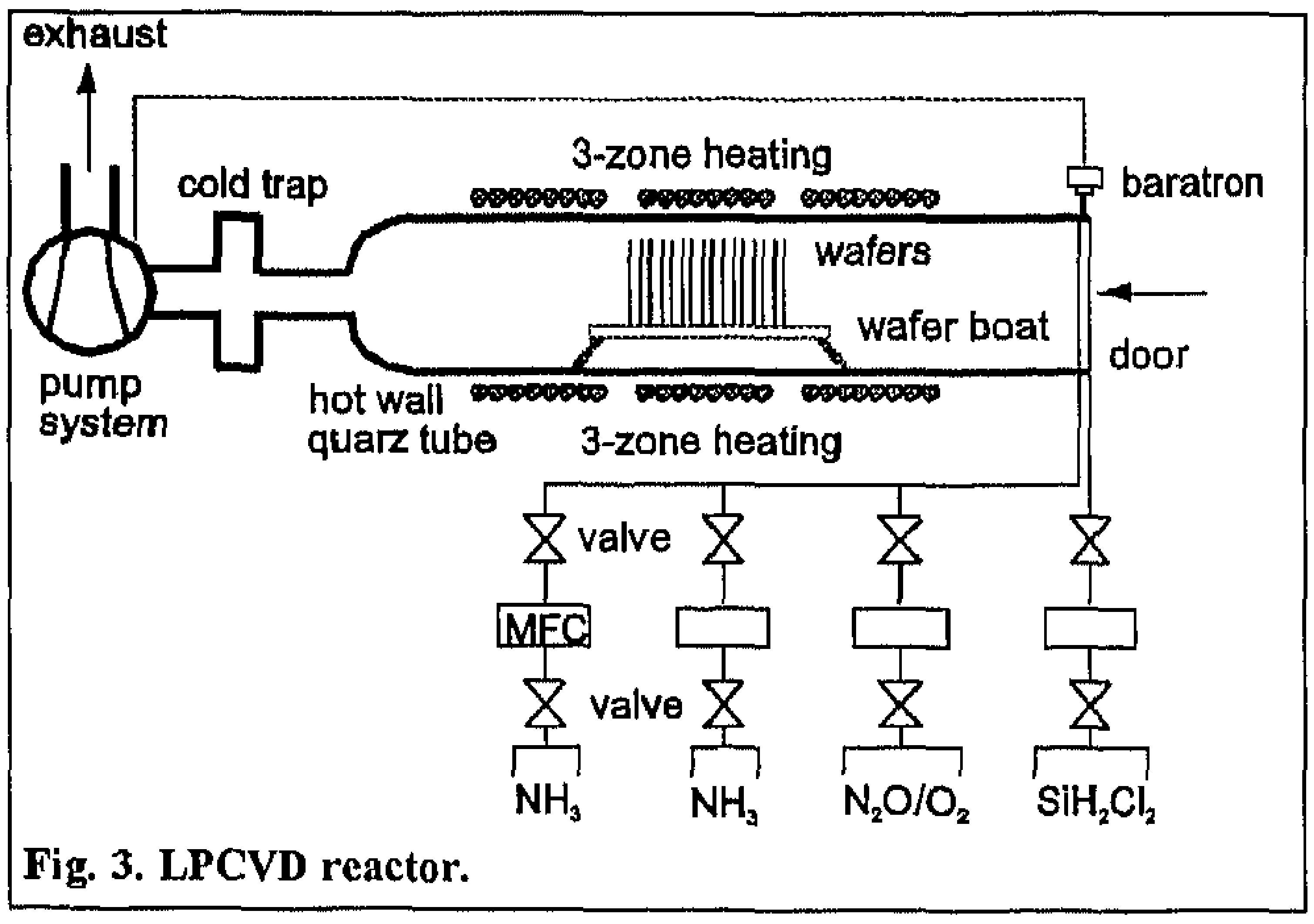


Fig. 3. LPCVD reactor.

In a Low pressure CVD process the energy needed for the reaction is generated by heating the substrate and gas phase to a sufficient high temperature. For SiO_xN_y deposition temperatures between 800 and 980 °C are used. Special tube ovens with a zone of constant temperature have been developed for these processes (Fig 3).

Comparison of PECVD and LPCVD

A parallel plate reactor is typically a single wafer or small batch size reactor, while a PECVD hot wall and a LPCVD reactor can process large batch sizes. There is a large difference in process temperature between PECVD and LPCVD. The higher temperature in the LPCVD process results in in principle higher quality films, but it appears that these films have also a larger tensile stress, which restricts the layer thickness that can be deposited.

Silicon Oxynitride Layer Properties

PECVD

The main optical property is the refractive index. As stated in the introduction, this index can be changed by changing the process, e.g. the N_2O flow (fig.4). This change is caused by a change in composition. For low N_2O concentrations the change in index is large. The process reproducibility is doubtful in this region. In fact the process is useful for refractive indices lower then 1.75.

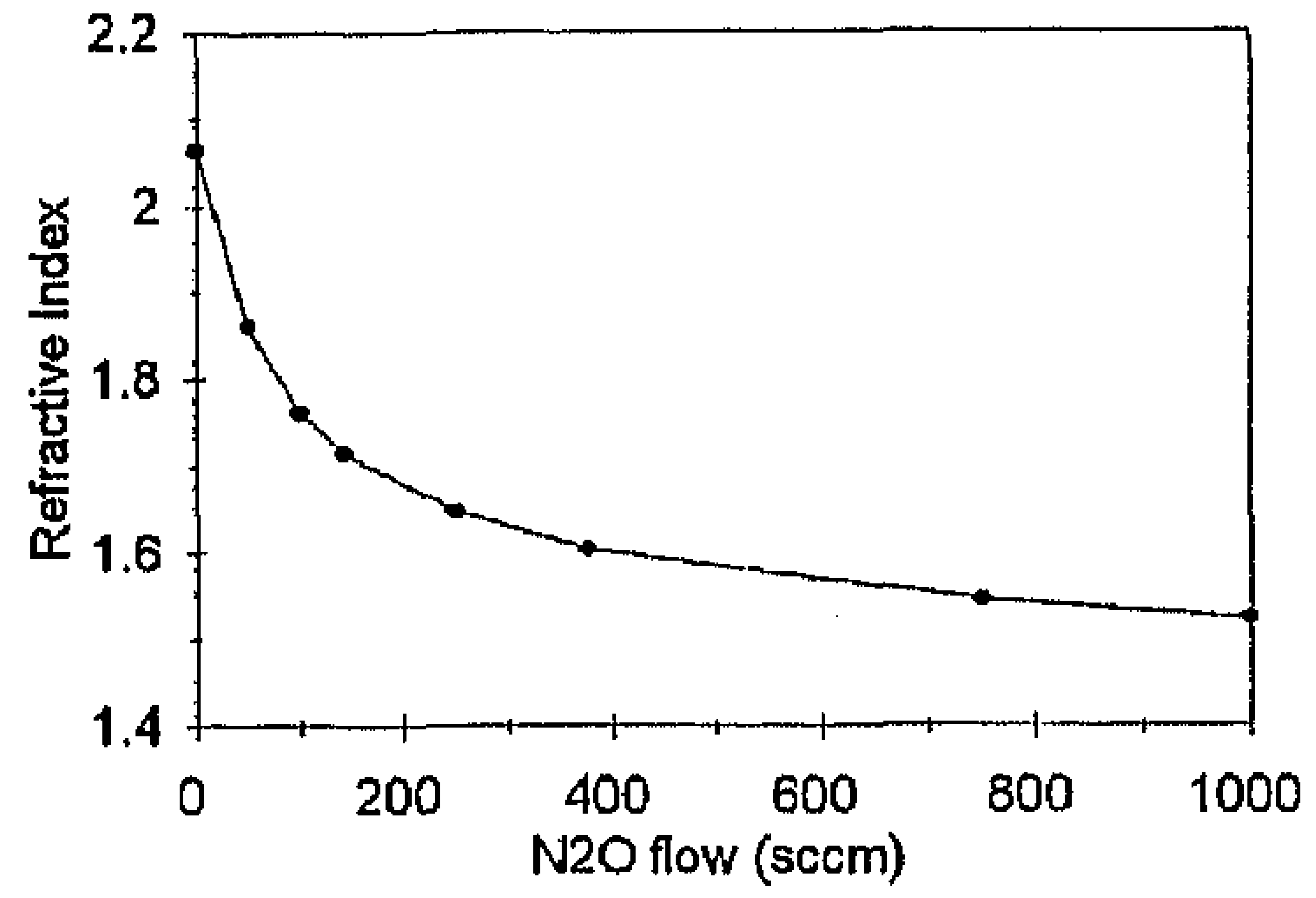


Fig. 4. Refractive index as function of the N_2O flow for a parallel plate reactor.

The growth rate increases over the same range from 20 - 35 nm/min. The optical loss of a fabricated waveguide, as deposited, is about 0.5 - 1 dB/cm at a wavelength of 632.8 nm. This loss is mainly bulk and surface scatter loss. A reduction of the loss to < 0.2 dB/cm is obtained after annealing at temperature between 500 - 700 °C. The uniformity of the PECVD process depends on the reactor type and process conditions. Most important is that the electrodes are very well parallel and the reactor is not too strong contaminated. The best results obtained after well aligning and cleaning are presented in table III. The uniformity in a parallel plate reactor is better because the flow to the surface is more directly controlled. Further improvements specially for the parallel plate reactor are expected by improving the reactor design, deposition and cleaning procedures and process optimization.

Table III. Uniformity of PECVD process for Parallel Plate and Hot Wall reactor. The values are the difference between the maximum and minimum over a square of 50x50 mm on a 3 "wafer.

reactor	refractive index (n)	n - uniformity (Δn)	thickness - uniformity (Δd in %)
Parallel Plate	1.7	0.0005	2
Parallel Plate	1.5	0.0006	1.4
Hot Wall	1.6	0.005	8

LPCVD

Also for this process the refractive index can be varied. Two processes, based on either N_2O or O_2 as an oxygen source, have been used (fig. 5 and 6).

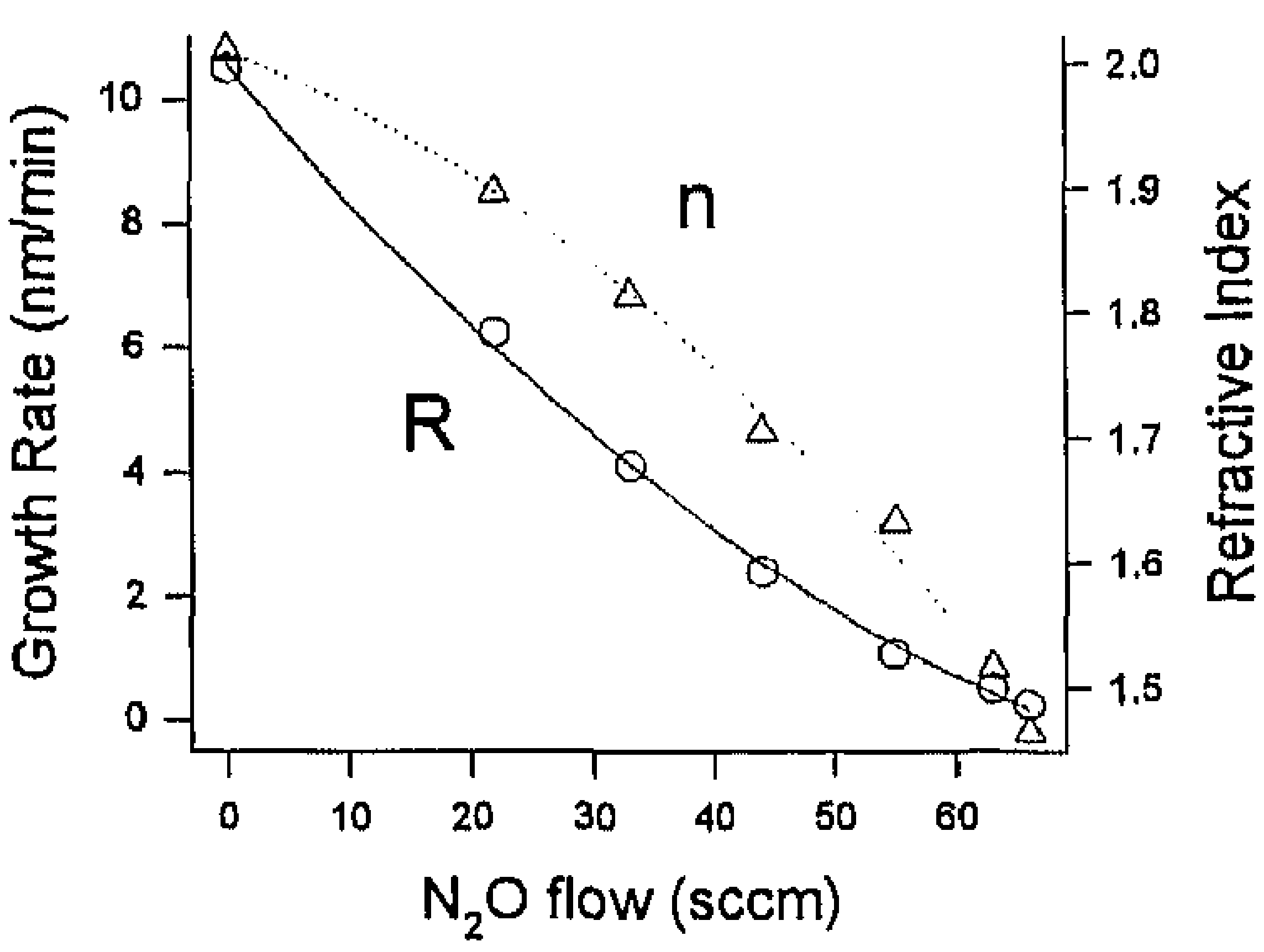


Fig. 5. Refractive index and growth rate as function of the N_2O flow for the LPCVD process.

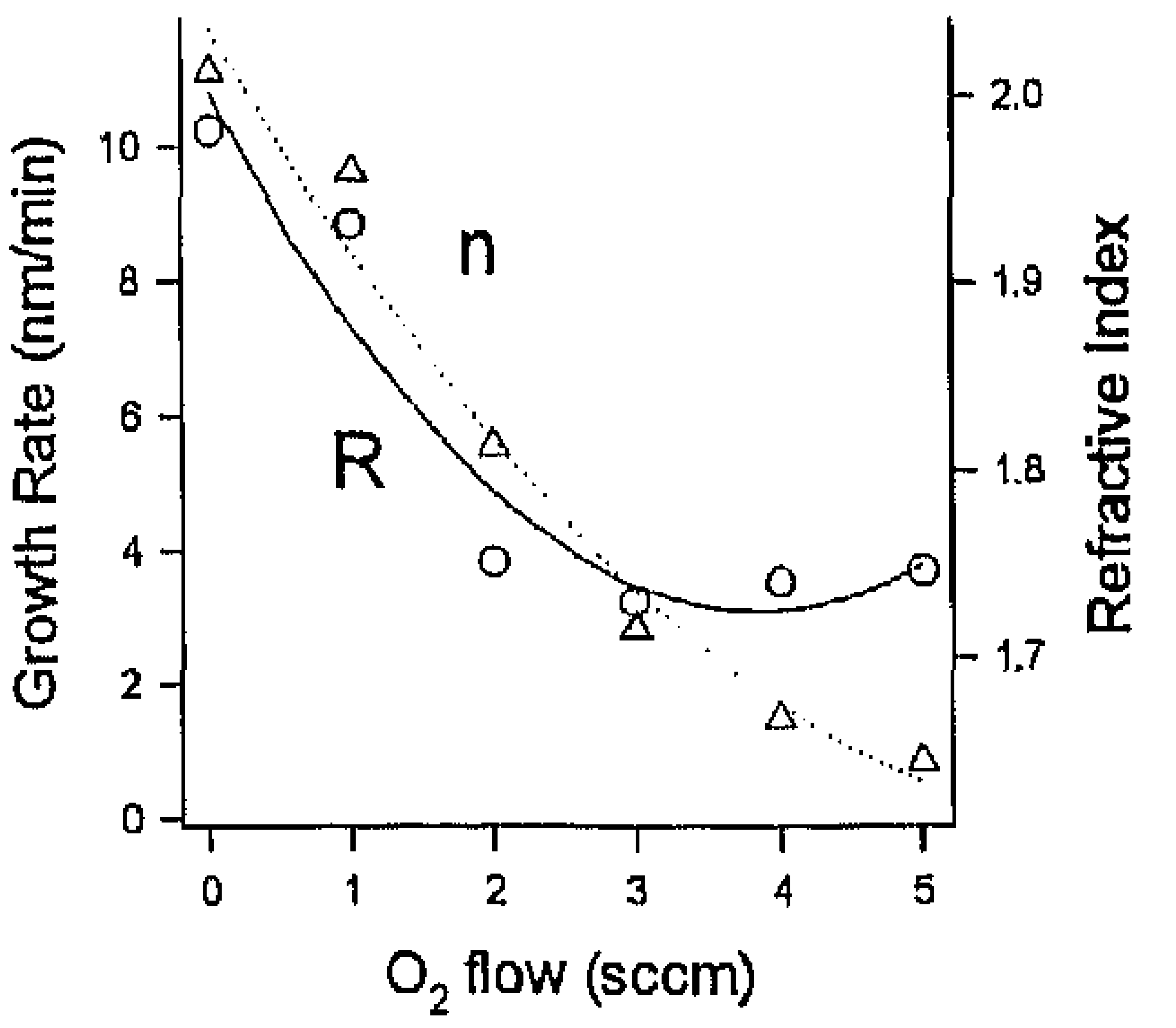


Fig.6. Refractive index and growth rate as function of the O_2 flow for the LPCVD process.

Unlike the PECVD process the deposition rate strongly decreases for higher N_2O or O_2 flows. Films grown with both processes have very low losses (< 0.2 dB/cm) in the visible region.

In the IC industry mainly the nitride process is used. Good uniformity is reached in this process because the growth rate is determined by the rate of a surface reaction. Therefore non-uniformity due to variation in transport to different parts of the wafer is a second order effect. In oxynitride deposition the process is more complicated and position dependent concentration variations occur. Larger non-uniformity then for nitride is found (Table IV). The strongest increase is found in the refractive index variation, while also the thickness uniformity is worse for layers with a refractive index below 1.85.

Table IV. Uniformity of LPCVD process using oxygen. The values are the difference between the maximum and minimum over a square of 50x50 mm or 20x20 mm on a 3 " wafer.

material	refractive index (n)	n - uniformity (Δn)		thickness - uniformity (Δd in %)	
		50x50	20x20	50x50	20x20
Si_3N_4	2.0	<0.0005	<<0.0005	2	0.2
SiO_xN_y	1.82	0.008	0.003	2	0.2
SiO_xN_y	1.7	0.006	0.003	5	0.5

Comparison between PECVD and LPCVD

PECVD and LPCVD appear to be complementary technologies for integrated optics applications. Films with a refractive index below 1.75 can be deposited with PECVD. Better uniformity can be obtained and the process is more suitable for the required higher layer thickness because of the higher deposition rate and lower stress. Films with a higher refractive index then 1.75, especially the nitride, can better be deposited with LPCVD. The better uniformity together with lower scattering losses are the main advantages. Growth rate is not so important because the required thickness is only a few tenths of a micron. The low surface roughness of these thin films is very important to avoid scatter losses.

Hydrogen induced losses

PECVD and LPCVD film contain hydrogen due to the use of hydrogen compound in the deposition process. Reported concentration range from 20-30 at% in PECVD films to 3 at % in LPCVD films. Absorption losses due to the incorporated hydrogen are more important at wavelengths in the near infrared (1300 - 1600 nm), which find more application in telecommunication. The main absorption peaks are at 1380 nm and 1520 nm, both are overtones from vibration absorption's of the Si-OH and N-H bonds. The first occurs only in PECVD SiO_2 while the second is present in all nitrogen containing material. The amount of hydrogen is be determined by infrared spectroscopy that is calibrated with Elastic Recoil Detection (ERD) (fig. 7.)[10]. The absorption loss in the peak maximum (1520 nm) can be as high as 16 dB/cm. With high temperature annealing (1150 °C) the amount of incorporated hydrogen is reduced to 1 - 2 at%. An example of an absorption loss spectrum is given in figure 8. The peak height and width are reduced, so the residual loss at the important telecommunication wavelength of 1550 nm is < 0.2 dB/cm. Unfortunately the film tensile stress increases such that films with a refractive index above 1.6 cracked. The here presented data are for a PECVD process using a 187.5 kHz plasma generator. For the often used frequency of 13.56 MHz the hydrogen is bonded more to silicon, which causes increased stress and cracking.

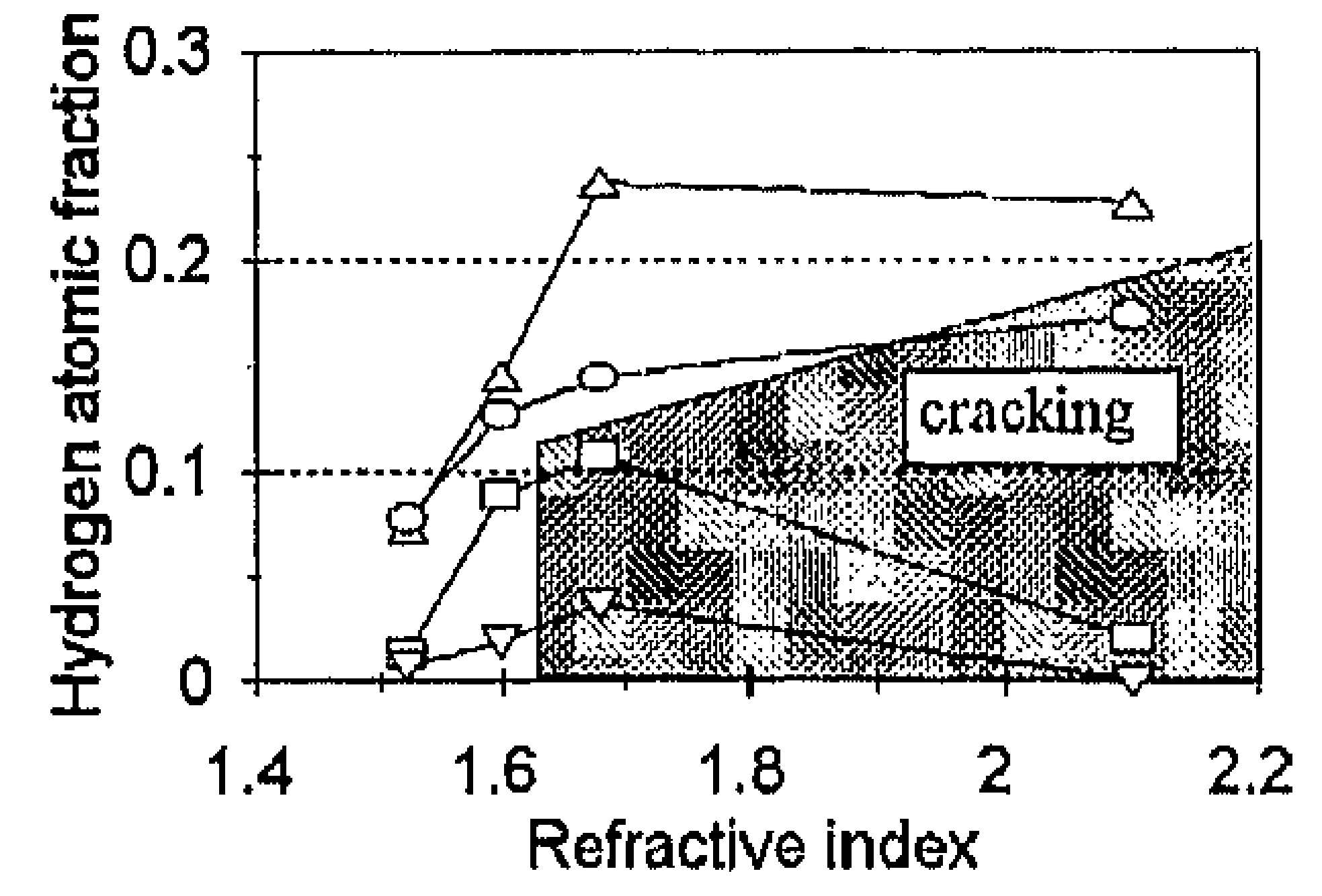


Fig. 7. Hydrogen content of PECVD films as function of the refractive index, as deposited (Δ) and after 700°C (O), 1000°C (□) and 1150°C (∇) annealing. If the point is in the gray area the film cracked upon annealing.

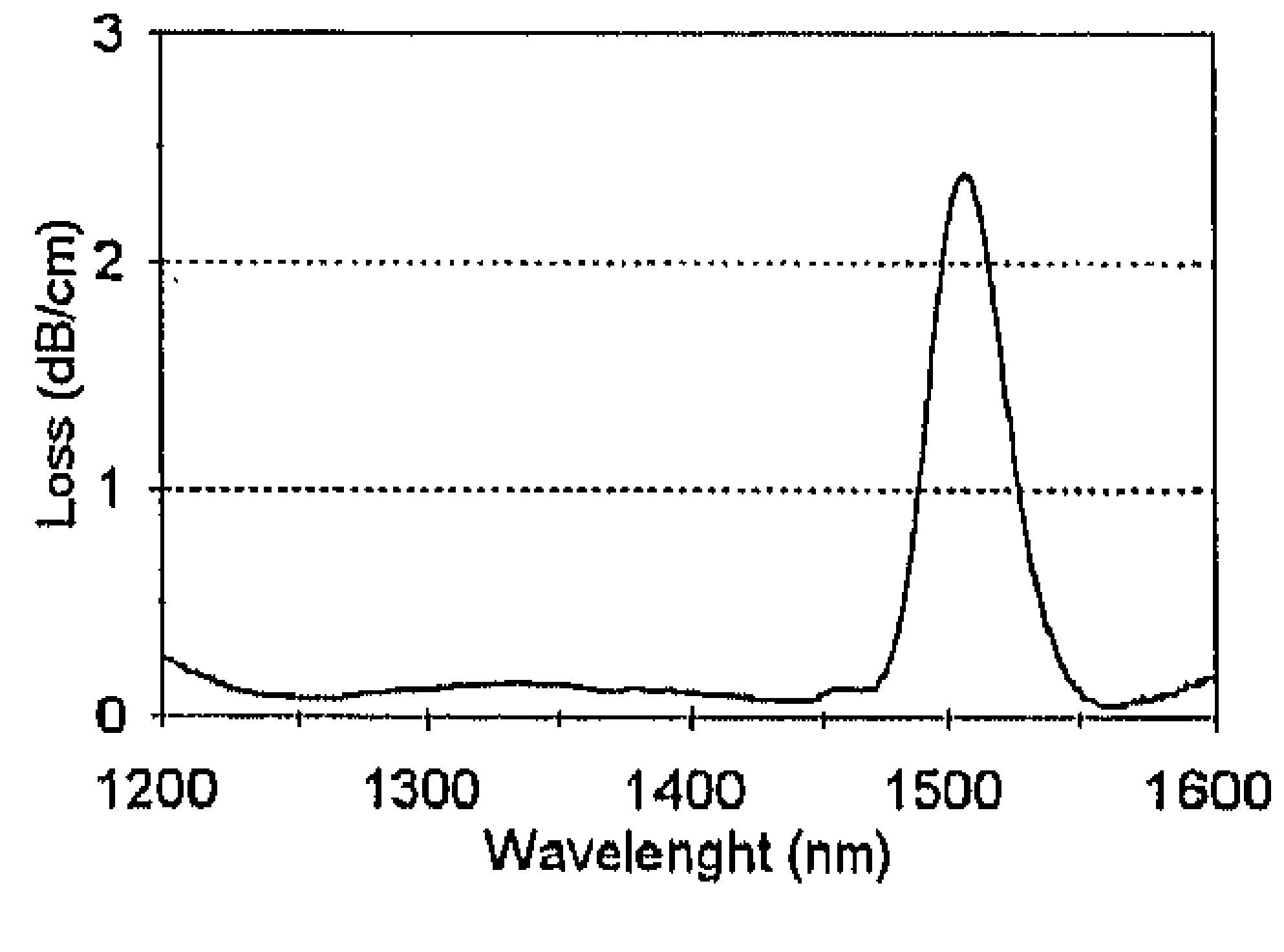


Fig. 8. Loss spectrum of an annealed (1150 °C) waveguide of PECVD SiO_xN_y with n=1.6. Residual hydrogen: 2 at%.

Applications

Layer uniformity is for integrated optic devices very important because the mode of propagation of light is directly coupled to the optical properties and layer thickness. Most basic functions are based on the phase of the optical signal. Such a situation is rare in other thin film or electronic devices. The demand is uniformity in propagation constant or effective refractive index of the waveguide. In principle a non-uniformity in thickness can be compensated with a non-uniformity in refractive index. In practice this is hard to realize and uniformity of both thickness and refractive index are needed. Not all functions or devices are as sensitive for non-uniformity. We will discuss first several functions with increasing demands and thereafter look at more complex devices build of functions on different positions on a chip.

Absorption sensor

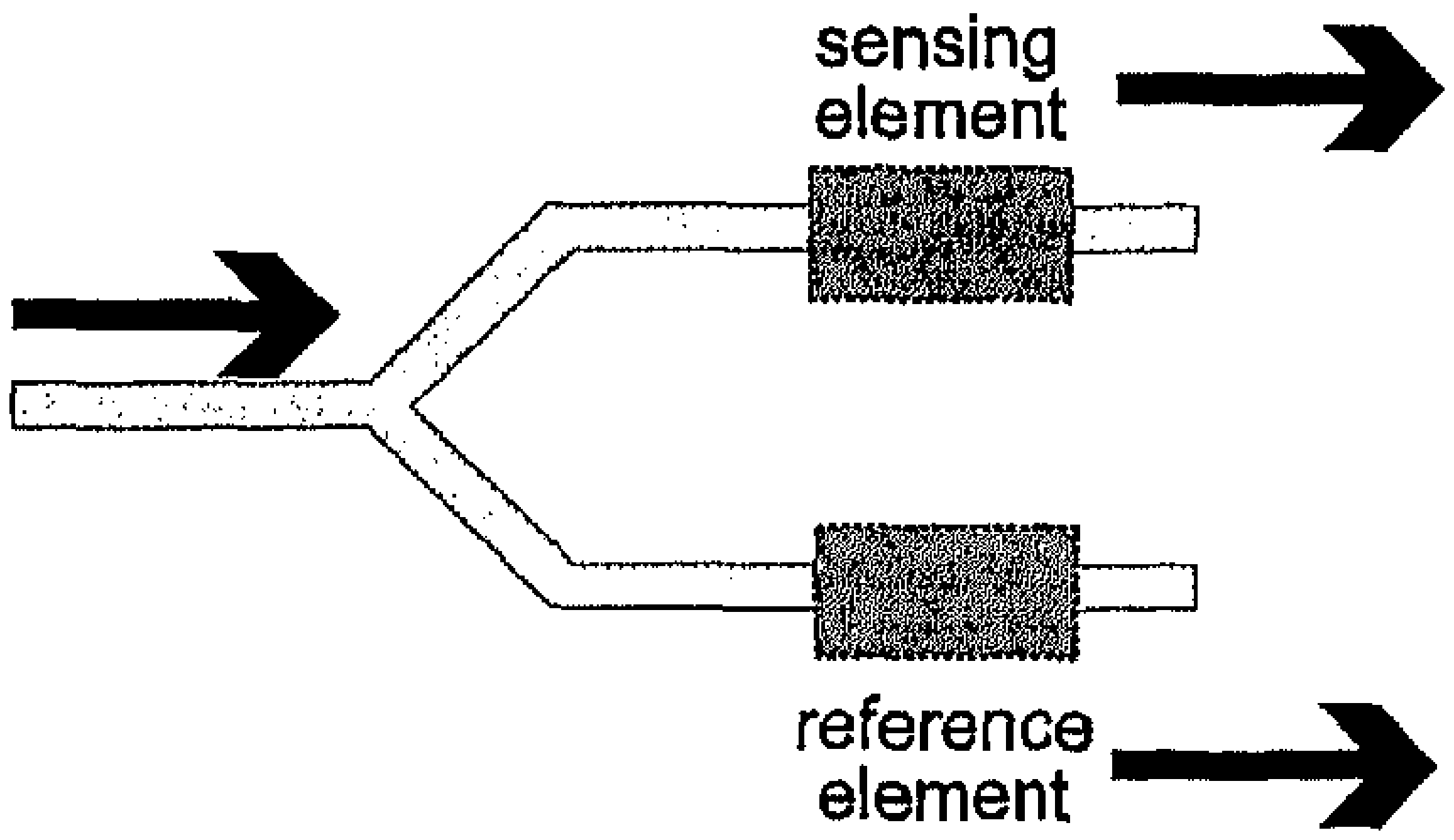


Fig.9. A schematic absorption sensor.

In this simple type of sensor (fig.9) the decrease in light intensity in a sensing waveguide channel is compared to that in a reference channel. The demands on waveguide uniformity are not very severe. Only intensity is important and no demands on phase are needed.

Mach-Zehnder interferometer.

A Mach Zehnder interferometer is an optical structure in which light is splitted in two branches and recombined. The output is maximal if the light arrives in phase at the point of combining. In figure 10. a general design for an integrated version is shown. The basic idea is that the propagation is changed in one of the branches, e.g. by a sensing element. Both branches are normally not identical and another source of difference e.g. non-uniformity of the effective index off the waveguide is no real problem. In some situations the branches are designed to be identical and the Mach-Zehnder is operated close to the symmetrical situation. As an example we will present the effect of non-uniformity for such a device. For the waveguide structure shown in figure 10. and a branch length of 30 mm and separation of 50 µm the effect of the thickness variation on the phase, the signal level and the sensitivity is presented in table V. The output signal of a Mach-Zehnder varies with the phase as:

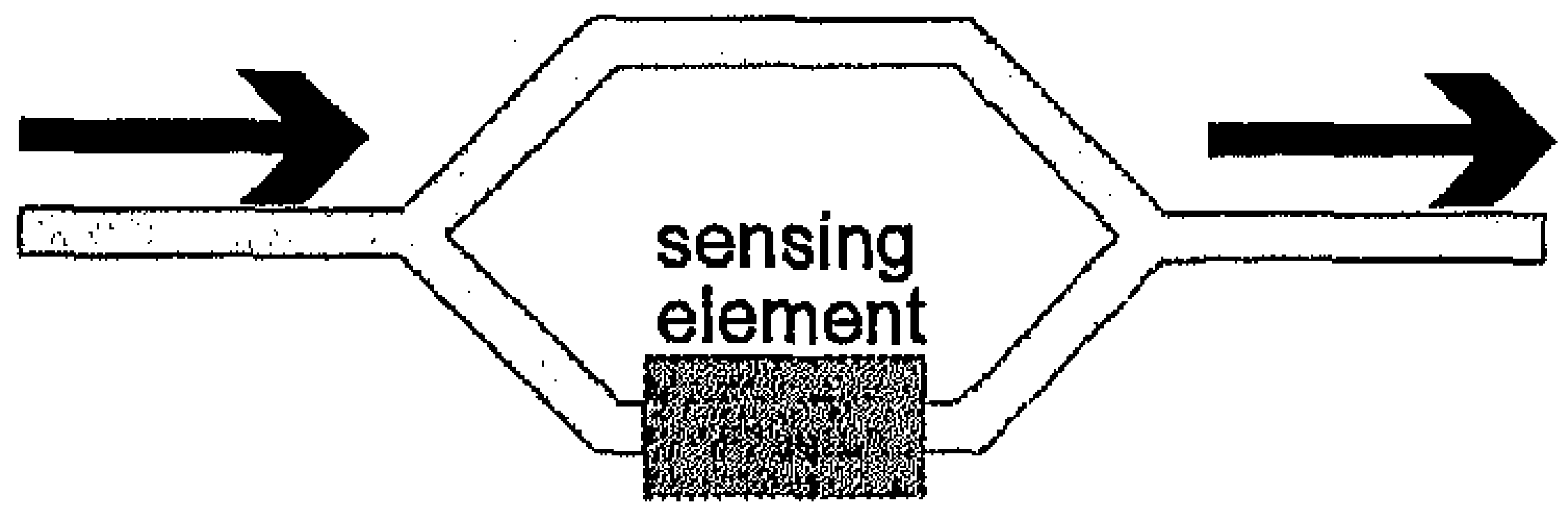
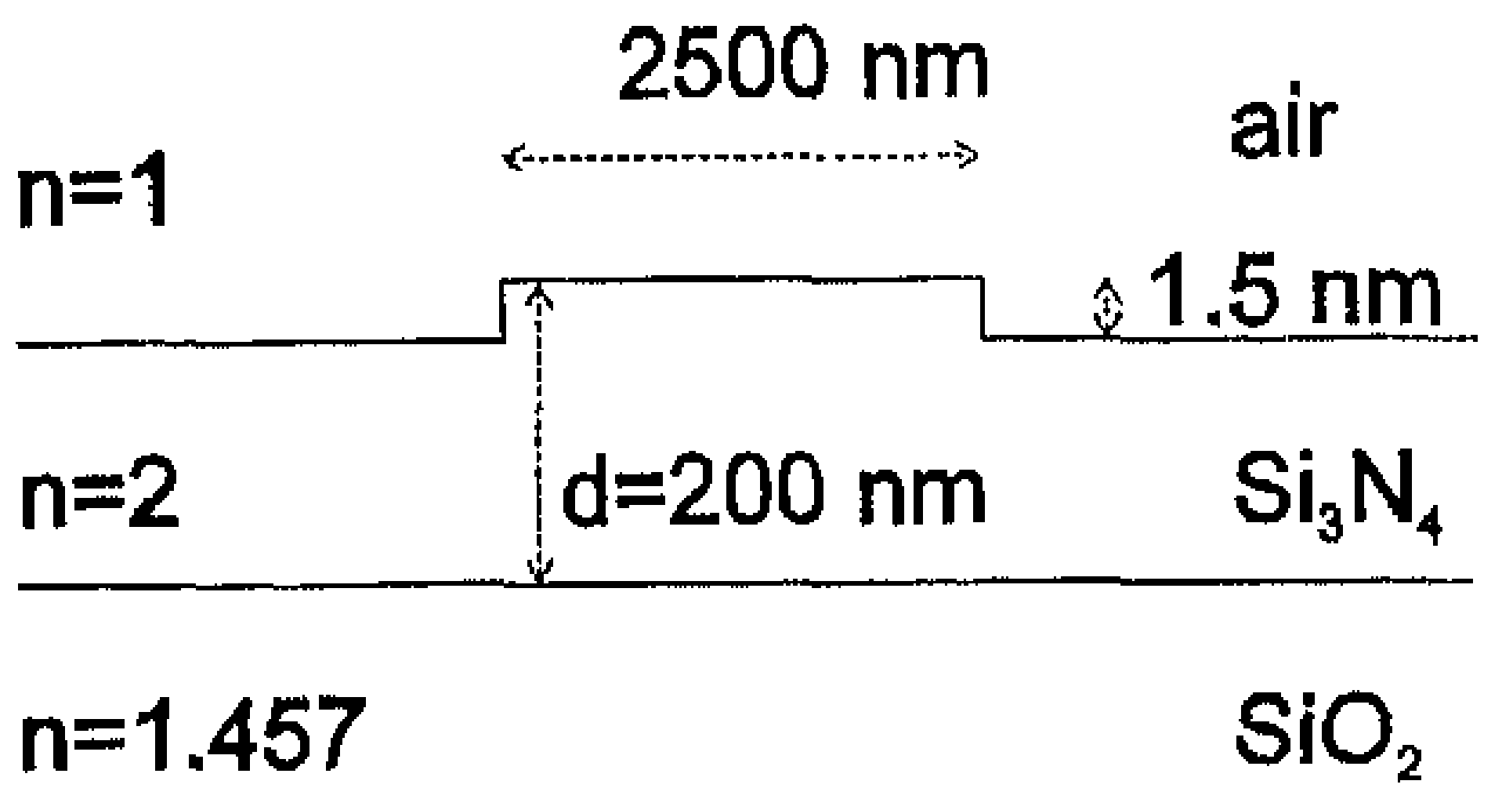


Fig. 10. A schematic Mach Zehnder interferometer.

$\frac{I_{out}}{I_{in}} = \cos^2 \frac{\Delta\varphi}{2}$ in which I_{out} and I_{in} are the intensities in the input and output and $\Delta\varphi$ the phase difference between the branches.



$$\frac{dN_{eff}}{dd} = 1.2\cdot10^{-3}\ \text{nm}^{-1}$$

Fig. 11. Cross-section of nitride waveguide used in the Mach-Zehnder interferometer.

Table V. Effect of non-uniformity on a Mach-Zehnder interferometer.

$\frac{\Delta d}{d}$ (%/cm)	$\Delta\varphi$ (rad)	$\Delta\left(\frac{I_{out}}{I_{in}}\right)$ (%)	$\Delta\frac{\partial\left(\frac{I_{out}}{I_{in}}\right)}{\partial\varphi}$ (%)
1	0.2 . 2π	-	4
0.5	0.1 . 2π	~30	2
0.1	0.02 . 2π	6	0.4

The phase difference corresponding with the steepest output change is chosen as working point: $\Delta\varphi = \pi/2$. For absolute measurements the change in signal level, $\Delta\left(\frac{I_{out}}{I_{in}}\right)$, is important, while for relative measurements the change in sensitivity, $\Delta\frac{\partial\left(\frac{I_{out}}{I_{in}}\right)}{\partial\varphi}$, is important. From table V. it is clear that absolute accuracy is hard to reach even with very uniform layers. Relative accuracy as presented here needs clearly a high uniformity. More general the Mach-Zehnder is not so demanding because when $\Delta\varphi$ is varied over more than 2π the exact position of the interference fringes is known.

Gratings

Gratings are functions with different applications in sensors:

- coupling free space light beam into a waveguide
- coupling light from a waveguide through free space to a detector
- coupling light between two modes in the same or different waveguides

  This function can be used to make a wavelength filter because the coupling condition:

  $\Lambda = \frac{\lambda}{\Delta n_{eff}}$ with Λ = grating period, λ = wavelenght and

  Δn_{eff} = the difference in effective refractive indexes of the modes

  If the Δn_{eff} is effected by the sensing layer, the read-out is in terms of a wavelength change. For a small bandwidth filter the length must be as large as possible. One can derive for a non-uniform waveguide the maximum useful length is restricted by the non-uniformity. This results in the following relation for the minimum bandwidth:

  $$\Delta\lambda_{FWHM} = \Lambda\sqrt{2\lambda\frac{\partial(n_{eff})}{\partial z}}$$

  In designing a waveguide structure with two vertical TE modes the sensitivity of Δn_{eff} for either thickness variations or refractive index variations can be minimized. In table VI. the minimum bandwidths and optimal lengths for both situations are presented.

Table VI. Grating length and bandwidth limited by non-uniformity.

n SiO_xN_y	Thickness insensitive $\Delta n = 0.001$ /cm		Refractive index insensitive $\Delta d = 0.2$ % /cm	
	L_{max} (mm)	$\Delta\lambda$ (nm)	L_{max} (mm)	$\Delta\lambda$ (nm)
1.6	2.1	1.9	5.1	1.5
1.7	2.1	1.1	3.6	1.3
1.8	2.1	0.8	2.9	1.1
1.9	2.1	0.6	2.5	1.0
2.0	2.1	0.5	2.3	0.9

The bandwidths are acceptable for many applications but this can only be reached for a good uniformity. Even in this case the optimum length is only a few millimeters.

Second Harmonic Generating Device

One way of generating visible light for use in integrated optical waveguide sensors is second harmonic generation (SHG) from cheap diode lasers with a wavelength of about 850 nm. A planar waveguide version of such a device has been realized on the basis of SiO_xN_y waveguides covered with non-linear calix[4]arene. In this device the modes at the fundamental wavelength (ω) and the second harmonic wavelength (2ω) must be phase matched very well. To reach a coherence length of 10 mm a $\Delta n_{eff} < 2x10^{-5}$ is needed for optimal efficiency. In practice this is a very hard to reach demand. Here every increase in uniformity pays off in much better efficiency. For a device with a refractive index of SiO_xN_y well matched with that of calix an efficiency of 1 % was reached with 500 W input power[11].

Complex devices

The above considerations are only for a single function. In practice devices will consists off at least some functions. The separation of the functions will be much more than the separations within a function. Often the performance of the same functions on different positions must be the same. Integrated optical components are large, so a complex device will cover a whole wafer or at least a significant part of it. As an example of such a situation we will consider here two Bragg reflector gratings[12] on different positions. The demand is that the bandwidths (0.2 nm) have sufficient overlap. For this type of grating we can derive:

$\Delta\lambda = 2 \cdot \Lambda \cdot \Delta n_{eff}$, so for a maximum change in wavelength of 0.2 nm the uniformity in n_{eff} must be better than 0.0005. In table VII. the maximum variations in parameters for a Si_3N_4 waveguide are presented.

Table VII. Maximum variations for refractive index (n), thickness (d) and channel width (w) for a Si_3N_4 waveguide resulting in a Δn_{eff} = 0.0005.

parameter	parameter value	variation
n	2.0	0.0007
d	120 nm	0.25 nm
w	2.5 μm	0.5 μm

Conclusions

SiO_xN_y is a flexible material for integrated optical applications. The deposition facilities are well developed for the IC production. It was shown that after some improvements in processing the properties are already very good and that further improvements can be expected. From the device examples it can be concluded that the demands range from very tolerant for variations in thickness and refractive index over the wafer to very hard to completely matched. This is a very fruitful situation in which the technology is already well enough developed to fabricate useful devices and on the other hand there is challenge for further improvement.

References

---

[1] S. Valette, J.P. Jadot, P. Gidon, S. Renard, A. Fournier, A.M. Grouillet, H. Denis, P. Philippe, E. Desgranges, "Si-based integrated optics technologies", Sol. State Technol., febr.'89, pp.69-75, 1989.

[2] C.H. Henry, R.F. Kazarinov, H.J. Lee, K.J. Orlowsky, L.E. Katz, "Low loss Si3N4-SiO2 optical waveguides on Si", Appl. Opt. 26(13), pp.2621-2624, 1987.

[3] H.Kreuwel, "Planar Waveguide Sensors for the Chemical Domain", Thesis, University of Twente, 1988.

[4] W. Gleine, J.Müller, "Integrated optical components on silicon substrates", Proc. EFOC/LAN 88, p.38-42, 1988.

[5] H. Bezzaoui, A. Baus, E. Voges, "Integrated optics on silicon with PECVD-fabricated waveguides", Micro System Technol., Ed. H.Reichl, p.283-288, 1990.

[6] "Interierte Optik auf Silizium", Abschussbericht des Verbundprojektes: "Entwicklung eines CMOS-kompatiblen Gesamtprozesses zur Herstellung integriert-optoelektronischer Schaltungen auf Silizium", 1989-1992, VDI/VDE Technologiezentrum Informationstechnik GmbH, 1992.

[7] P. Gidon, S. Valette, P. Schweizer, "Vibration sensor using planar integrated interferometric circuit on oxidised silicon substrate", Proc.SPIE Vol.514, pp.187-190, 1984.

[8] D. Peters, K. Fischer, J. Müller, "Integrated optics based on silicon oxinitride thin films deposited on silicon substrates for sensor applications", Sens. & Actuators A 25-27, p.425-431, 1990.

[9] E. Voges, "Integrierte optik auf Glas und Silizium für Sensoranwendungen", Techn.Messen 58(4), pp.140-145, 1991.

[10] H. Albers, L.T.H. Hilderink, E. Szilágyi, F. Paszti, P.V. Lambeck, Th.J.A. Popma, "Reduction of hydrogen induced losses in PECVD-SiO_xN_y optical waveguides in the near infrared, LEOS '95, IEEE Lasers and electro-optics Society Annual Meeting 1995, San Francisco, pp. 88-89.

[11] K. Wörhoff, O.F.J. Noordman, N.F. van Hulst, H. Albers and P.V. Lambeck, "Phase matched second harmonic generation in silicon[oxy]nitride - calix[4]arene waveguides", CLEO Pacific RIM '95, Technical Digest, p.199, 1995.

[12] G.J. Veldhuis, R.G. Heideman and P.V. Lambeck, "Bragg-reflector used as integrated chemo-optical sensor",This conference.