InP-based planar photonic crystals: process development, characterization and infiltration

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Back cover: scanning electron microscope top image of a fabricated photonic crystal.

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Chapter 1
Introduction

The possibility to control the electrical properties of materials has revolutionized the society in the second half of the 20th century. The widely spread use of personal computers, (mobile) phones and the internet has lead to a demand for transmission and processing of information that is still increasing. Where the use of electrical signals for this purpose is running into its fundamental limits, the use of optical signals may facilitate the growing need for information. The long haul transport of information is already done with optical signals in glass fibers. Also for short haul transport, the electrical copper cables are more and more replaced by optical fibers (fiber-to-the-home). To fully benefit from such an optical network, the processing of the signals in network nodes must also be done optically, preferably by monolithically integrated, and thus compact, devices. Devices on present day optical integrated circuits have dimensions much larger than the wavelength of the light used. The ultimate miniaturization of these photonic circuits requires control of light on the scale of its wavelength. A promising development in this respect is the concept of the photonic crystal (PhC).

1.1 Description of the research field

A PhC is a material with a periodic modulation of the dielectric constant. For certain lattice structures and sufficient refractive index contrast it exhibits a
photonic band gap: a frequency range in which no electromagnetic (EM) modes are allowed, i.e. the photonic density of states is zero. This concept was developed independently by Yablonovitch [1] and John [2] in 1987. It can be deduced from Maxwell’s equations that electromagnetism in a composite of isotropic, linear and lossless dielectric materials with no free charges or currents is described by [3]:

$$\nabla \times \left( \frac{1}{\varepsilon(r)} \nabla \times \mathbf{H}(r) \right) = \left( \frac{\omega}{c} \right)^2 \mathbf{H}(r).$$  \hspace{1cm} (1)

Here $\mathbf{H}$ is the magnetic field vector, $\varepsilon$ the scalar dielectric constant, $\mathbf{r}$ the position vector, $c$ the speed of light in vacuum and $\omega$ the angular frequency of the light. The equation is given in terms of the magnetic field, because its electric field counterpart is more difficult to solve [3]. Equation 1 is a Hermitian eigenvalue equation, which implies that the eigenfunctions $\mathbf{H}(\mathbf{r})$ are orthogonal, can be catalogued by their symmetry properties, and have real eigenvalues. The properties of EM waves in a periodic dielectric medium therefore exhibit similarities to the properties of electron waves in a solid crystal [3]. Some important formalisms of solid state physics are also applicable here: light propagates in a PhC as a Bloch mode, the dispersion relations or photonic band structure can be visualized in folded representation in the first Brillouin zone, and defects in the PhC can sustain localized EM states [3, 4]. The possibility to engineer the photonic band structure (in analogy to the semiconductor band structure) and related defect states has led to a completely new research field with the primary focus on control of light at the scale of its wavelength.

No fundamental length scale is present in equation 1, which implies that the spectral position of the photonic band gap scales with the lattice constant $a$ of the PhC. For a given lattice structure and refractive index contrast, the photonic band structure and transmission spectrum can therefore be presented in general form as a function of normalized frequency $a/\lambda$, in which $\lambda$ is the free space wavelength. As in solid state physics, the opening of gaps occurs at the edge of the Brillouin zone where the wavelength in the medium is in the order of the lattice constant. Applications at optical wavelengths, e.g. for telecom or lighting, therefore require refractive index modulations with a period of a few hundred nanometer. Three main types of PhCs can be distinguished: 1D, 2D and 3D, referring to the number of spatial dimensions in which the refractive index is periodic, see Figure 1-1.
A multilayer film, see Figure 1-1a, is periodic in one direction and homogeneous in the plane and can therefore be regarded as a one-dimensional (1D) PhC. Long before the concept of photonic crystal was introduced, multilayer films have been applied as dielectric mirrors, using the high reflection coefficient within the 1D photonic band gap [6]. This concept is used in so-called Bragg reflectors, e.g. in vertical cavity surface emitting lasers [7]. The dispersion relation of 1D PhCs is exploited in distributed feedback lasers for wavelength selection purpose [8].

Truly zero EM density of states can be achieved in three-dimensional (3D) PhCs, see Figure 1-1c, which are periodic in all three directions. Several types of 3D PhCs have been realized. Drilling holes in a slab of material has shown to be a successful fabrication method, but is obviously feasible only for macroscopic PhCs [9, 10]. A so-called woodpile structure working in the infra-red was first fabricated by Noda et. al., but requires many lithography and wafer bonding steps [11]. The most widely used fabrication principle is sedimentation of polymer- or silica spheres from a colloidal solution. The spheres self-assemble into a regular lattice during drying, with relatively low defect density. This lattice forms a 3D PhC, but also serves as a template for fabrication of PhCs with higher refractive index contrast [12, 13]. When the self-assembled PhC is filled with elastomer and
the template is removed, it can be applied for taking fingerprints [14].

Two-dimensional (2D) PhCs are periodic in one selected plane and homogeneous in the perpendicular direction, see Figure 1-1b. These structures typically consist either of air holes in a dielectric material or dielectric pillars in air. For confinement of the light to the horizontal plane, the 2D lattice is usually embedded in a planar waveguide structure [15]. This configuration is no longer a genuine 2D PhC because the vertical symmetry is broken, and is therefore referred to in this thesis as planar PhC (PPhC). Many device proposals and -realizations based on PPhCs have been shown in literature, notably for integrated optics applications. Line defects in such a structure, see Figure 1-2a, can form efficient waveguides for wavelengths in the photonic band gap [16, 17, 18, 19, 20], while point defects, see Figure 1-2b, can form 2D cavities. These two basic elements are used in devices like bends [21, 22, 23, 24, 25, 26], splitters [27, 28, 29, 30, 31], crossings [32, 33, 34] and add-drop filters [35, 36, 37, 38], with an overall size of less than 10 µm. The quality factor of PPhC defect cavities has recently been improved dramatically, up to almost $10^6$ experimentally and $10^8$ in theory, using special designs [39, 40, 41]. The quality factor over mode volume (Q/V) ratio of PPhC point defects is orders of magnitude higher than in any other cavity design [39], reaching values in the order of $10^{24}$ m$^3$ or $10^6/\lambda^3$ ($\lambda$ in m). This property has been used to realize low-threshold short cavity lasers [42, 43], strong coupling of the EM cavity mode with a spatially confined exciton in semiconductor material [44, 45], and fluid sensors [46]. The curvature of the dispersion bands of PPhC line defects at the edge of the Brillouin zone results in modes with low group velocity (slow light), which can be used for enhanced interaction with the guiding material or optical memory applications [47, 48, 49]. Working exactly at the photonic band edge allows for lasing over large surface

Figure 1-2: Example (top view) of a) a line defect and b) a point defect in a 2D PhC.
areas [50]. It has also been shown, that PPhCs show anomalous refraction-like behavior in the vicinity of the photonic band edges [51, 52]. One consequence of this is the superprism phenomenon, which can be applied in wavelength filtering for wavelength division multiplexing (WDM) devices [53]. Another consequence is that PPhCs can exhibit negative refraction, which can be used for perfect near-field imaging [54].

1.2 Focus of this thesis

In this thesis the focus is on PhCs that can be applied for photonic integrated circuits (PICs) in long distance telecom applications at wavelengths near 1.55 µm. The base material system for active devices working at this wavelength is InP. The refractive index $n$ of this material for $\lambda = 1.55$ µm (3.17 [56]), requires $a \sim 400$ nm. PPhCs are the natural choice for such applications because they can be integrated with planar laser structures and allow for much freedom in choice of crystal parameters and introduction of defects. Furthermore, they can in principle be fabricated with available patterning and etching techniques [57, 58, 59, 60, 61]. For compatibility with existing PICs, deeply etched- rather than membrane type PPhCs are investigated here. Hole type PPhCs are favorable over pillar type PPhCs, because they have a connected InP matrix and are thus expected to have more efficient guiding in the PhC region.

A standard PIC InP/InGaAsP/InP planar waveguide structure is adopted in this work. The $\text{In}_{0.73}\text{Ga}_{0.27}\text{As}_{0.57}\text{P}_{0.43}$ core layer is 500 nm thick and has $n_{\text{core}} \approx 3.34$ at $\lambda = 1.55$ µm [62]. The InP upper cladding layer is also 500 nm thick while the lower cladding is InP buffer layer and substrate. This planar waveguide is single mode for $\lambda = 1.55$ µm and the effective refractive index $n_{\text{eff}}$ of the transverse

![Figure 1-3: a) Schematic cross-section of the InP-based PPhC with the calculated [55] intensity profile of the TE guided mode of the planar waveguide shown on the right. b) 3D impression of the PPhC.](image-url)
electric (TE) guided mode is 3.25. The intensity profile of this mode is shown in the schematic cross-section of the PPhC in Figure 1-3a. For low out-of-plane loss it is crucial that the holes are deep enough to fully overlap with the vertical mode profile [63, 64]. A 3D representation of the PPhC is shown in Figure 1-3b.

A 2D photonic crystal is mirror symmetric with respect to every plane perpendicular to the holes/pillars. As a result, the EM modes are classified into two polarizations: transverse electric (TE, electric field in-plane) and transverse magnetic (TM, electric field out-of-plane) [3]. The mirror symmetry is broken in PPhCs due to the vertical layer structure and the modes are strictly speaking only TE- or TM-like. In the remainder of this thesis, these modes will be referred to as just TE or TM. The optical properties of PPhCs are polarization-dependent. Due to the continuity of the electric field lines, TE band gaps are favored by hole-type PPhCs and TM band gaps by pillar-type PPhCs (for the full argument, see [3, 65]). The triangular lattice of circular air holes, see Figure 1-4a, is used in this work because it exhibits relatively large TE band gaps. This lattice is defined by unit vectors (1, 0) and (0.5, 0.5·√3) and hole radius over lattice constant ratio \( r/a \). In Figure 1-4b the hexagonal Brillouin zone is shown schematically. The irreducible Brillouin zone is only 1/12th of this, see the shaded region in Figure 1-4b, due to the six fold rotational- and inversion symmetry of the lattice. The two main symmetry directions, defined as \( \Gamma K \) and \( \Gamma M \), are depicted both in real and reciprocal space in Figure 1-4.

Figure 1-4: Triangular lattice both in a) real space and b) reciprocal space. The two main symmetry directions of the crystal, \( \Gamma K \) and \( \Gamma M \), crystal parameters \( a \) and \( r \), and the unit cell of the lattice are denoted in a). The shaded region in b) is the irreducible Brillouin zone.
For many applications in PICs it is crucial that the optical properties of PPhC components are externally tunable. Present WDM schemes have sub-nm channel spacing and the accuracy of the channel position must be even smaller to assure stable operation. This accuracy in the resonance of a PPhC cavity can not be achieved with current fabrication technology. For this kind of applications, static tuning, also called trimming, of the device characteristics is required. For modulation of an optical signal, dynamic PPhC tuning is needed. Current telecom networks, operating at 10 Gbit/s data rate per WDM channel, require tuning times < 100 ps. To change the optical properties of a PPhC, either the crystal geometry or the refractive index contrast has to be altered. The geometry can be changed by compression or expansion of the structure, which is difficult for InP-based PPhCs. The refractive index contrast can be changed by altering either the high- or the low index material. For the latter case, the air holes need to be filled with a material that has a tunable refractive index. This allows for tuning of the optical properties of the PPhC without compromising the properties of the InP that may be required for the application. Also it could be used to compensate changes in the refractive index of the InP, for instance to enhance the temperature stability of devices.

1.3 Contribution of this thesis to the field

This thesis comprises the fabrication, characterization and infiltration of InP-based hole-type PPhCs. The methods and materials used are described in detail in chapter 2 of this thesis. The main achievements that are new to the field are listed below.

1.3.1 Sidewall passivation

The crucial process step in the fabrication of deeply etched PPhC is the final etching of the high aspect-ratio holes in the planar waveguide layer structure. Several successful etching processes have been reported in the literature. Good results have been obtained with Cl₂/Ar- [60] and Cl₂-based [61] chemically assisted ion beam etching (CAIBE), Cl₂/Ar-based electron cyclotron resonance (ECR) etching [59] and SiCl₄-based inductively coupled plasma (ICP) etching [58]. In the present work, the etching is performed with ICP using Cl₂ as the reactive component. Passivation of the sidewalls to obtain cylindrical hole-profiles is introduced by addition of O₂ or N₂ to the plasma chemistry. This concept is commonly exploited in etching of high aspect ratio structures in Si [66], but explicitly used here for the first time for the fabrication of InP based PPhCs. The major advantage of this chemistry is the increased window in the
parameter space in which the quality of the etched holes is acceptable, i.e. the holes are nearly cylindrical and sufficiently deep. This is an advantage when other structures, e.g. ridge access waveguides, need to be etched in the same run. The latter has proven to be particularly useful in this work. The etching results are presented in chapter 3.

1.3.2 End-fire transmission measurements in combination with loss parameter approach

In this thesis the PPhCs have been characterized by transmission measurements on finite crystal sections with no intentional defects, using an end-fire technique. Here, light from an external source is guided to and from the PPhC fields. This is in contrast with the internal light source (ILS) technique where the probe light originates from optically excited nanostructures like quantum wells or quantum dots that are incorporated in the material [67]. The latter technique has been successfully used in combination with a loss parameter approach [68] for assessment of the quality of GaAs-based [63] and InP-based [69] PPhCs. In this approach, the transmission measurements are fitted to 2D calculations in which the out-of-plane losses are simulated by introduction of a non-zero imaginary component of \( \epsilon \). The advantage of the end-fire technique is that it is generally applicable to any wavelength range in which the material is transparent. In ILS measurements of InP-based PPhCs, anomalously high transmission in the band gap is reported [69]. It will be shown in the remainder of this thesis, that > 30 dB transmission decrease in the gap can be observed with end-fire measurements. Although the end-fire technique is widely used, it is exploited here for the first time in combination with the loss parameter approach. An important result is that a considerable sidewall angle in the core layer does not lead to excessive losses, in contrast to what is commonly believed. Also it is shown that transmission through a PhC is possible by continuous diffraction of the light into allowed directions, even if the forward direction is forbidden. The optical measurements are described and analyzed in chapter 4.

1.3.3 Infiltration with polymer and liquid crystal

The first proposal for PhC tuning with the low index material was done by Bush and John [70]. They showed theoretically the possibility of opening and closing the band gap of a 3D PhC, using the tunability of infiltrated liquid crystals (LCs). Recently, liquid crystal infiltration has also been applied for tuning of PPhCs [71, 72, 73, 74, 75, 76]. In this thesis, PPhCs are filled with a solid polymer by monomer infiltration and in situ polymerization for the first time.
Both liquid crystals (LCs) and polymers have their advantages for tuning purposes and the material choice depends on the application. The main advantage of LCs is the large attainable refractive index change of typically $\Delta n = 0.2-0.3$. On the other hand, < 1 ps tuning can in principle be achieved using optically non-linear polymers. Also, the solid nature of polymers allows for further (polymer) processing on top of the filled PhC and offers mechanical stability. In this thesis a simple, reliable and reproducible procedure for filling deeply etched PPhCs with polymer is presented. It is shown that the strong capillary forces exerted by a liquid inside the PhC holes allow for infiltration in ambient atmosphere. The polymer filling fraction is determined with optical measurements to be 80%. The analysis of the filling experiments with both polymer and LCs is presented in chapter 5.
Chapter 2
Methods and materials

For optical simulation and characterization, PhC sections without intentional defects are integrated with input- and output ridge waveguides in the transmission configuration as shown in Figure 2-1. The 2.5 μm wide ridges are formed by etching trenches on both sides and are aligned with either the ΓK- or the ΓM direction of the triangular lattice. The PhC section contains nine unit cells for the ΓK-direction and nine-and-a-half for the ΓM-direction, which are aligned as indicated in the figure. The center of the horizontally outer holes is placed on a distance 0.5·a (for ΓK, 0.5·√3a for ΓM) from the edge of the PhC section. The total distance between the waveguide ends is therefore 10.5·a for the ΓK- and 10.5·√3a for the ΓM-direction. In this region the light is not laterally guided but a significant fraction (>0.8) of the input light is collected by the output waveguide for the wavelength range considered here.
2.1 Simulation

Since the papers of Yablonovitch and John, much research effort has been devoted to modeling of the EM behavior of PhC structures. The objective of many of these efforts is to calculate the photonic band structure and the associated field distribution of the Bloch modes in the unit cell of the crystal by solving equation 1. Basic PhC properties as the spectral position and width of the photonic band gap can be derived from these calculations, as will be shown in section 2.1.1. Band structures are only valid for infinitely extended and perfect crystals however, while a real world PhC is finite, suffers from fabrication imperfections and interacts with its surrounding. Therefore, other methods are used for the modeling of actual device structures. These usually involve discretization of a finite PhC structure in space followed by numerical solving of Maxwell’s equations in either the frequency- or the time domain. It will be shown in section 2.1.2, that these simulations yield additional information. The tools for PhC simulation have matured over the years and some of them are incorporated in commercially available software now. In this thesis the program ‘Crystal Wave’ of the company ‘Photon Design’ is used. With this program, photonic band structures are calculated with the plane wave expansion (PWE) method.
The EM behavior of actual device structures can be simulated in Crystal Wave using the finite difference time domain (FDTD) method [78] or with the frequency domain (FD) solver.

In principle, full 3D calculations are required for PPhCs, because the in-plane and out-of-plane components of the electric field are not separable [68]. 3D calculations require long calculation times, while 2D calculations are feasible with current desktop computers. Because the vertical confinement of the planar waveguide used in this work is relatively low, the effective index approach can be used. In this approach, the PPhC is approximated by a purely 2D PhC with refractive index equal to \( n_{\text{eff}} \), the effective index of the planar waveguide structure [79]. 2D simulations are then sufficient to model the 2D PhC. The effective index approach is applied in combination with both PWE and FDTD throughout this thesis.

In the 2D calculations, out-of plane components of the k-vector are not accounted for. However, even perfect PPhCs with infinitely deep cylindrical holes exhibit so-called intrinsic losses for modes above the light line [80]. Deviations from the perfect crystal structure lead to so-called extrinsic losses [63, 64]. How to include losses in the 2D calculations is described in section 2.1.3.

### 2.1.1 Band structures

To determine the band structure of a 2D triangular lattice, it is sufficient to calculate the set of eigenvalues \( \omega_m \) for \( k \)-vectors along the edge of the irreducible Brillouin zone (the path \( \Gamma MK \Gamma \) in Figure 1-4b). Here \( m \) is the integer band number and \( k \) the wavevector. In PWE this is done by solving the Fourier transformation of equation 1, i.e. in the reciprocal space [77]. For the calculation of the Fourier transform of \( 1/\varepsilon(r) \), the unit cell of the PWE calculation in Crystal Wave is divided into a number grid cells that must be a power of 2. The eigenvalues \( \omega_m \) for \( m = 1-4 \) at the \( \Gamma \)-, \( M \)-, and \( K \)-point change with less than 1 % when the resolution is increased from 32x32 to 64x64 cells. All PWE calculations presented in this thesis are therefore performed with a resolution of 32x32 cells. The calculated TE and TM band structure for a 2D triangular lattice of air holes with \( r/a = 0.3 \) in a material with \( n = n_{\text{eff}} = 3.25 \) is presented in Figure 2-2a and b respectively. The first four bands (i.e. \( m = 1-4 \)) are calculated for 16 \( k \)-points in each part of the Brillouin zone edge (\( \Gamma M, MK \) and \( K \Gamma \)).
The so-called stop gaps for TE polarization between the $m = 1$ and $m = 2$ bands in the $\Gamma K$ and $\Gamma M$ direction are indicated in Figure 2-2a. They cover a different spectral region, because of the different periodicity in these crystal directions. The overlap of the stop gaps is indicated by the shaded region and covers frequency range $a/\lambda = 0.221-0.287$. No EM mode is available in this frequency region, which means that this is the TE photonic band gap of this particular crystal. As in semiconductors, the bands below and above the band gap have special names, dielectric band and air band respectively. These names refer
to the fact that the electric field of the mode is localized mostly in the dielectric material for the \( m = 1 \) band and more in the air holes for the \( m = 2 \) band. For a crystal with \( a = 400 \) nm and \( r = 120 \) nm, the band gap runs from 1394-1810 nm. The dashed line just above the \( m = 1 \) band is the light line of this configuration, which is just the \( m = 1 \) band of the 2D PhC formed by the perforated claddings [81]. Only the part of the band structure that lies below this light line represents truly guided modes. In Figure 2-2b it is visible that no band gap is present for TM polarized light, due to the degeneracy of the first two bands at the K-point. The relatively narrow stop gap for the \( \Gamma M \) direction is indicated in the figure.

Figure 2-2 represents the band structure for one particular value of \( r/a \). In Figure 2-3 the grey areas represent the lowest photonic band gap as a function of \( r/a \), ignoring higher order gaps in the graph. Such a graph is called a gap map. Only data for \( r/a \leq 0.5 \) is shown in the figure, because the holes overlap for larger \( r/a \). The TE band gap opens up at \( r/a = 0.15 \) and the relative gap width increases linearly up to \( r/a = 0.4 \). The center frequency of the gap increases with \( r/a \), because the effective refractive index of the structure as a whole decreases. For \( r/a > 0.42 \) also a TM gap opens up, between the \( m = 2 \) and \( m = 3 \) band. Qualitatively this is because for large hole radii, the lattice resembles a lattice of pillars, although connected by thin veins [3]. A complete polarization independent gap is present for these high \( r/a \) values where the TM gap overlaps with the TE gap. The fabrication of such structures is a difficult task however, because small (sub 100 nm) features are left between the holes. Furthermore, the
losses are high because most of the guiding material is removed. Therefore the focus here is on the more robust TE gap at moderate values of $r/a$.

### 2.1.2 Transmission spectra

The transmission spectrum of the PhC configuration shown in Figure 2-1 is simulated with FDTD. This means that the geometry is divided into a mesh of nodes, both in space and time. The field in each node is calculated from the field in neighboring nodes using a finite difference equivalent of Maxwell’s equations. For this purpose, the geometry is divided into a rectangular mesh that is matched to the triangular PhC lattice with $a = 400$ nm. A cell size of $10 \times 10\sqrt{3}$ nm proved to give sufficient resolution and is used for all FDTD calculations in this thesis. The time step for the evolution of the fields is related to the grid and equal to $1.4 \cdot 10^{-17}$ s here. In the simulation, a Gaussian (in time) pulse with a central wavelength of $1.52$ $\mu$m and bandwidth of $1$ $\mu$m is excited in the input waveguide on the left. The in-plane field profile of the pulse is identical to the fundamental mode of the $2.5$ $\mu$m wide waveguide at the central wavelength. The optical signal is monitored by two Crystal Wave software sensors, one before and one after the PhC. The sensors register the computed optical power in the fundamental waveguide mode in the propagation direction of the pulse at these locations. Spectral information is obtained by Fourier transformation of the time-evolution of the signal. The duration of the calculation, the number of time steps, therefore determines the spectral resolution. The signal of the output sensor is divided by that of the input sensor to obtain the normalized transmission of the PhC.
The transmission spectra for both symmetry directions are shown for TE and TM polarization in Figure 2-4a and Figure 2-4b respectively. In Figure 2-4a, the positions of the TE stop gaps, as calculated with the PWE method, are indicated by vertical lines. These are in agreement with the regions where the transmission is decreased by several orders, down to $10^{-6}$, with respect to the pass bands. In the band gap the transmission is inhibited for both symmetry directions. The transmission level in the dielectric band is lower for the $\Gamma M$ direction because of

![Figure 2-4: Calculated $\Gamma K$- and $\Gamma M$ transmission spectra for the geometry of Figure 2-1 and a) TE- and b) TM polarization. The dashed vertical lines indicate the dielectric- and air band edge as calculated with the PWE method.](image)
the larger \( w \), and thus higher lateral losses. For \( \Gamma M \) the transmission levels in dielectric- and air band are calculated to be equal, but for \( \Gamma K \) the level is significantly lower in the air band. This could be due to a difference in grating-like diffraction. First order diffraction is possible for \( a/\lambda > (n_{\text{eff}})^{-1} = 0.31 \) for the \( \Gamma M \)- and \( a/\lambda > (\sqrt{3}\cdot n_{\text{eff}})^{-1} = 0.18 \) for the \( \Gamma K \) direction [82, 83]. In the air band this is the case for both symmetry directions, but the diffraction efficiency is probably higher for \( \Gamma K \) at these \( a/\lambda \) values. The oscillations in the pass bands are due to Fabry-Pérot type interference between the boundaries of the PhC. At \( a/\lambda \sim 0.38 \) a second transmission minimum appears for both directions, although no stop gaps are visible in the band structure. This so-called pseudo gap is due to symmetry forbidden coupling of the ridge waveguide mode with the \( m = 3 \) band [15, 82, 83, 84].

The calculated narrow TM, \( \Gamma M \) stop gap is indicated by the vertical lines in Figure 2-4b and is in agreement with the calculated transmission spectrum in the figure. In the \( \Gamma K \) direction, no stop gap is present, but a relatively broad transmission minimum is observed. This is again due to symmetry forbidden coupling, this time with the \( m = 2 \) band. At higher frequencies, also the symmetry of the \( m = 4 \) band results in a pseudo gap. For both \( \Gamma K \) and \( \Gamma M \) the transmission significantly drops just above the diffraction cut-off, at \( a/\lambda = 0.18 \) and 0.31 respectively.

2.1.3 Introduction of loss parameter \( \varepsilon'' \)

The loss parameter approach was introduced by Benisty et. al. [68]. In this approach, a fictitious absorption in the air holes is used to simulate out-of-plane optical losses in 2D calculations (here FDTD). This is realized by attributing a complex \( \varepsilon (\varepsilon = \varepsilon' + i\varepsilon'') \) to the holes, while assuming the semiconductor to be lossless. The relation \( \varepsilon = n^2 \) leads to the following relations between the real and imaginary components of \( n = n' + in'' \) and \( \varepsilon \):

\[
\varepsilon' = n'^2 - n''^2 \quad \text{and} \quad \varepsilon'' = 2n'n''.
\]

In the Crystal Wave program losses are included by introduction of a power absorption coefficient \( \alpha \) in the holes, while keeping \( n' \) constant. \( \varepsilon'' \) is related to \( \alpha \) and \( n' \) by:
The influence of optical losses on the calculated TE, $\Gamma_M$, $r/a = 0.3$ transmission spectrum is shown in Figure 2-5 on linear transmission scale. The most important changes in the spectrum upon increase of the loss are the decrease of the transmission level, notably in the air band, and damping of the Fabry-Pérot fringes. The difference in sensitivity to $\varepsilon''$ of the dielectric- and air band arises from the sampling of the holes by the respective Bloch modes. The TM spectrum is less sensitive to the loss parameter than the TE spectrum, because the Bloch mode is concentrated more in the high dielectric for this polarization, specifically in the air band. The gray line in Figure 2-5 represents the case $\varepsilon'' = \varepsilon''_{\text{int}}$, see section 2.1.4, and shows already significant decrease of the transmission level. In Figure 2-6, the influence of $r/a$ on the TE, $\Gamma_M$, $\varepsilon'' = 0.10$ transmission spectrum is shown. Larger $r/a$ values lead to lower transmission levels, due to the larger absorbing area in the simulation. The influence of $r/a$ on the stop gap width, following Figure 2-3, is also apparent.

### 2.1.4 Loss mechanisms: $\varepsilon''_{\text{int}}, \varepsilon''_{\text{ext}}$ and $\varepsilon''_{\text{hole}}$

The loss parameter $\varepsilon''$ is the sum of an intrinsic contribution $\varepsilon''_{\text{int}}$, due to the perturbation of the vertical waveguide structure in the air holes, and an extrinsic contribution $\varepsilon''_{\text{ext}}$, due to fabrication imperfections. In ref. [68], a semi-quantitative
expression for $\varepsilon_{\text{int}}$ was obtained by treating the lack of guiding in each hole as a perturbation with a dielectric constant $\Delta \varepsilon = n_{\text{core}}^2 - n_{\text{clad}}^2$, in which $n_{\text{clad}}$ is the refractive index of the cladding material. In first order approximation, the out-of-plane losses are due to the electric dipole radiation from this perturbation into non-guided modes. Under the assumption of the dipoles radiating incoherently, $\varepsilon_{\text{int}}$ scales as:

$$
\varepsilon''_{\text{int}} \sim \frac{d_{\text{core}} n_{\text{core}}}{\lambda} \left(\frac{r}{a}\right)^2 \left(\frac{a}{\lambda}\right)^2 (\Delta \varepsilon)^2 \eta \Gamma_{\text{core}}(\lambda). \tag{4}
$$

Here $d_{\text{core}}$ is the thickness of the core layer, $\eta$ the fraction of the dipole emission that escapes into non-guided modes and $\Gamma_{\text{core}}$ the confinement factor of the planar waveguide (fraction of the mode energy located in the core), which is equal to 0.73 for the planar waveguide used in the present work. A constant factor has been left out in equation 4 in comparison to ref. [68], because it is merely used as a scaling law here. The scaling of $\varepsilon''_{\text{int}}$ with $r/a$ and $a/\lambda$ implies that the intrinsic losses are different for the air band and the dielectric band. If $\varepsilon''_{\text{int}}$ is known for a specific wavelength and dielectric configuration, it can be rescaled to any wavelength and configuration for which the perturbation approach is valid. For a GaAs based PPhC, the value for $\varepsilon''_{\text{int}}$ was determined by Benisty et. al. with full 3D calculations to be in the range 0.024 - 0.048 [63]. Later on it was determined that $\varepsilon''_{\text{int}}$ is close to the lower limit of this range [85]. This value is rescaled with
equation 4 to the PPhC used in the present work, assuming $\eta = 1 - \cos(\varphi_c)$, with $\varphi_c$ the critical angle $\sin^{-1}(n_{\text{clad}}/n_{\text{core}})$ [68]. For $r/a = 0.3$, $a/\lambda = 0.3$ and $\lambda = 1550$ nm, $\varepsilon''_{\text{int}} = 0.024$.

The extrinsic loss parameter is the sum of different contributions arising from fabrication imperfections such as finite etch depth, non-cylindrical hole shape, sidewall roughness, and fluctuations in hole radius and position of its centre. It has been established that the first two are the dominant cause for optical loss with the current fabrication technology and can be translated in a loss parameter contribution $\varepsilon''_{\text{hole}}$ [64]. A similar approach as for the derivation of the scaling law for $\varepsilon''_{\text{int}}$ was followed by Benisty et. al. to analyze the influence of finite hole depth on $\varepsilon''_{\text{hole}}$ for cylindrically shaped holes [63]. It was found that $\varepsilon''_{\text{hole}}$ decreases

Figure 2-7: Calculated dependence of $\varepsilon''_{\text{hole}}$ on sidewall angle $\beta$ for truncated cone shape in the lower cladding, hole radius of 150 nm and wavelength of 1520 nm. The hole depth ranges from 1.5 to 3 $\mu$m and is increased with steps of 0.1 $\mu$m. The $\varepsilon''_{\text{int}} = 0.024$ level is indicated in the figure by the horizontal dashed line. The dependence for simple cone shape is also shown, for $r = 90$ and 150 nm. The simple cone lines differ significantly only for sidewall angles $> 3$°.
exponentially with etch depth, due to the exponential decrease of the guided mode intensity in the lower cladding. In first approach the focus in the fabrication process development should therefore be at maximal hole depth. Ferrini \textit{et. al.} obtained an analytical expression for this case \cite{64}, which can be written for hole depth $h$ as:

$$
\varepsilon_{\text{hole}}'' = \frac{8\pi^3}{3} \left(1 - n_{\text{clad}}^2\right)^2 n_{\text{clad}} \lambda^3 r^2 \Gamma_h(\lambda) L_{\text{decay}}. \quad (5)
$$

Here $\Gamma_h$ is the fraction of the mode energy below a distance $h$ from the top surface and $L_{\text{decay}}$ a characteristic decay length of the mode intensity in the lower cladding. Equation 5 shows that $\varepsilon_{\text{hole}}''$ scales with $r^2/\lambda^3$ and therefore it is higher in the air band than in the dielectric band for equal hole depth. The analysis was extended by Ferrini \textit{et. al.} to holes with (truncated) cylindroconical profile, a more realistic shape after etching \cite{64, 86}. This was performed under the assumption that the cylindrical section of the hole extends at least to the bottom of the core layer. Following the approach in refs. 64 and 86, the dependence on sidewall angle $\beta$ for increasing etch depth was calculated for the three layer heterostructure used in this thesis \cite{1}. The sidewall angle is defined here as the deviation from the vertical. The result for $\lambda = 1520 \text{ nm}$ and $r = 150 \text{ nm}$, with $1.5 \mu \text{m} < h < 3 \mu \text{m}$ in steps $\Delta h = 0.1 \mu \text{m}$, is shown in Figure 2-7. For $r/a = 0.3$, this is in the air band of the transmission spectrum. Above a certain sidewall angle, which depends on the hole depth, the truncated hole shape is simply conical. The simple cone lines for $r = 90$ and $150 \text{ nm}$ are also shown in the figure and differ significantly only for $\beta > 3^\circ$. Two regimes are distinctly visible in Figure 2-7. For small $\beta$, the truncated cone line saturates to a depth dependent $\varepsilon_{\text{hole}}''$ value that is lower than $\varepsilon_{\text{int}}''$ for $h > 2 \mu \text{m}$. For large $\beta$, $\varepsilon_{\text{hole}}''$ is primarily determined by the cone angle and $\varepsilon_{\text{hole}}'' < \varepsilon_{\text{int}}''$ only for $\beta < 0.8^\circ$. This highlights the demand for nearly cylindrical shape of the holes, particularly in the part just below the core layer \cite{64}. Thus, in the optimization of the etching process, both hole depth and sidewall angle need to be considered for significant reduction of the optical loss.

---

\textsuperscript{1} For these calculations the original Matlab-files developed by dr. R. Ferrini were used.
2.2 Process development

2.2.1 Sample design

For evaluation of the fabrication process, a design without ridge waveguides is used. The test samples are plain InP substrates and contain various PhC sections with different crystal parameters and 10 μm wide lines to determine the etch results of large areas in the fabrication procedure. The purpose of these samples is to enable cross-sectional SEM inspection of the holes, in any stage of the fabrication process. A convenient property of InP in this respect is that it easily cleaves along either the (011) or (01 1) crystallographic planes, leaving a shiny and near atomically flat cut section. The PhC sections are therefore placed along one of these InP crystal directions, using the edge of the sample as a reference for alignment. All fields are then cleaved with a single cut, by scratching the surface...
at the edge of the sample with a diamond scriber and applying force onto the sample next to the scratch. The symmetry axis of the PhC, either ΓK or ΓM, is tilted under a small angle (~3°) with respect to the cleavage so that a different part of the cross-section is visible for each hole. See Figure 2-8 for a schematic overview of the sample design.

### 2.2.2 Layer structure

The planar waveguide structure is grown on top of a (100) oriented epi-ready 2 inch InP wafer with metal organic chemical vapor deposition (MOCVD). A 1000 nm thick InP buffer layer is grown on the InP substrate, followed by the lattice matched 500 nm thick In$_{0.73}$Ga$_{0.27}$As$_{0.57}$P$_{0.43}$ core layer and the 500 nm thick InP upper cladding. The peak photoluminescence emission of the quaternary material is at 1265 ± 5 nm. After the MOCVD growth a SiN$_x$ hard mask is deposited on the full wafer by plasma enhanced chemical vapor deposition (PECVD). The thickness and average refractive index of this layer were measured with an ellipsometer to be ~370 nm and 2.0 (at visible wavelengths) respectively, giving the surface a green color. The wafer was then cleaved into nine pieces of approximately 16x16 mm$^2$. A layer of ZEP-520A (ZEON) electron beam (e-beam) resist solution in anisole was deposited onto the individual pieces by spin coating at 5000 rpm for 50 seconds. The layer was baked on a hotplate for 120 seconds to evaporate the solvent, leaving a 320 nm thick resist layer.

### 2.2.3 E-beam lithography

The 2D PhC patterns and the ridge waveguides are defined in the ZEP layer with e-beam lithography (EBL). The patterns are generated using electromagnetic deflection of an e-beam with a diameter of ~2 nm, in combination with a beam blanker. The exposure is performed with either 100 keV electron energy in a Leica 5000 e-beam pattern generator$^1$ or 30 keV in a Raith 150 system$^2$. The required electron dose for the PhC holes is in the order of 130 μC/cm$^2$ for 100 keV and 50 μC/cm$^2$ for 30 keV electrons. After exposure the sample is developed in n-amylacetate for 60 seconds, rinsed in methylisobutylketone:2-propanol = 9:1 for 30 seconds and blown dry with N$_2$. See Figure 2-9a for a realization of the design of Figure 2-1 after development of the resist.

$^1$ DIMES, Delft
$^2$ TU/e
2.2.4 Pattern transfer by reactive ion etching of the hard mask

The actual developed pattern deviates from the design because of the so-called proximity effect, which is due to scattering of the electrons in the material. These electrons travel through the resist and cause parasitic exposure outside the area that was directly exposed by the electron beam. For 100 keV, this causes a rather uniform background dose over a range of \( \sim 15 \) µm, while for 30 keV the range is limited to \( \sim 2 \) µm and the gradient in the dose is much larger [87, 88, 89]. In general, feature sizes are larger than designed due to the proximity effect. This is in particular the case for the PhC holes in the design of Figure 2-1, because of the proximity of the large exposed areas that define the waveguides, see Figure 2-9b. This can be corrected by either adjustment of the dose distribution or decrease of the hole diameter in the design. In this work, the latter option was used. From dose tests with the 100 keV e-beam for the actual device pattern, so including the large areas for definition of the waveguides, using SEM inspection from top it was determined that the radius of the holes should be designed \( \sim 30 \) nm smaller for the desired \( r/a \)-value of 0.3.

![Figure 2-9: a) Top view of the exposed PhC pattern after development of the resist. The ridge waveguides are visible at the top and the bottom. b) Illustration of the proximity effect at the edge of the PhC. The holes become progressively smaller when they are further away from the large exposed areas on the right top and bottom. The leftmost holes are not even fully opened.](image)
by a plasma that is generated above the sample in a cylindrical vacuum chamber by capacitive coupling, see Figure 2-10a for a schematic overview. Radiofrequency (RF) power is applied to the bottom electrode plate. This generates an oscillating electric field in the chamber, which ionizes and dissociates the gas that is introduced into the chamber by inlets at the top. The electrons in the plasma are much more mobile compared to the more massive ions and collide with the electrodes driven by the RF field. The bottom electrode is electrically isolated from the rest of the grounded chamber and acquires a net negative charge by the absorbed electrons. The resulting DC bias voltage $V_{\text{bias}}$ is typically in the order of a few hundred volts and accelerates the positive ions towards the sample. The plasma potential is slightly positive, in the order of several tens of volts, because of the loss of electrons. The chemical etching process is enhanced by the ion bombardment, which results in anisotropic etching profiles.

CHF$_3$ chemistry is used for the pattern transfer to the SiN$_x$-layer\(^1\). With a gas flow of 60 sccm CHF$_3$, 50 W RF power and chamber pressure $p$ of 2.0 Pa, the induced $V_{\text{bias}} = -(250-350)$ V and the large area etch rate is 20 nm/min for the SiN$_x$ and 15 nm/min for the ZEP. The etch rate is lower for the PhC holes, because of gas depletion at the bottom. This phenomenon depends on the aspect ratio of the feature (depth divided by lateral width) and is commonly called RIE lag [90]. The required time to open the smallest holes, with a diameter of ~160 nm, is therefore increased with a factor of 1.5 compared to large areas. The

\(^1\) Performed with the Plasmalab 100 RIE system from Oxford Instruments
etch rate is also influenced by the size of the sample. The values given are valid only for a sample size of approximately 15x15 mm². For a full 2 inch wafer the etch rates decrease with a factor of 1.5 due to loading effects [90]. A cross-sectional SEM view of a PhC pattern in the SiNₓ mask is shown in Figure 2-10b. After the RIE pattern transfer, the residual ZEP is removed with an O₂ plasma in a barrel etcher.

### 2.2.5 ICP etching

The PhC holes are etched in the InP with ICP etching, using the SiNₓ as patterned masking layer. The etching plasma is generated in a cylindrical vacuum chamber by inductive coupling, see Figure 2-11 for a schematic overview of the system. RF power (Pₛ) is applied to a helical coil outside the chamber, which induces an oscillating magnetic field along the coil axis. This in turn produces azimuthal electric fields that ionize the gas that is fed through the coil. The plasma expands to the lower part of the chamber, where the sample is located on a plate electrode. This electrode is powered by a separate RF source (with plate power Pₚ) to produce a bias voltage. The bias voltage and plasma density are

![Schematic of the ICP etching system.](image-url)
therefore controlled independently. This allows for higher plasma (ICP) power and thus larger plasma density (hence usually higher etch rate) than in the RIE parallel plate configuration of Figure 2-10.

The challenge in dry etching of InP-based material is the removal of In, i.e. the formation of volatile In-containing etch products. The chemistry used for this purpose is usually based on either halogens or hydrocarbons. With hydrocarbons, the volatility of the main etch product In(CH₃)₃ allows for etching at room temperature [91]. A drawback of this chemistry is the polymer formation (due to CH presence), which limits the etch rate and requires thorough cleaning and preconditioning of the system. Also, the incorporation of H-atoms in the material can degrade device performance [92]. Of the halogen based chemistries, Cl is the most widely used, although I [93] and Br [94] based InP etching is also reported. At typical ICP etching conditions with a Cl₂-based chemistry, an InCl₃ corrosion layer forms on the surface, which hinders the transport of etch gas to- and reaction products from the underlying InP [95, 96, 97]. The InCl₃ is not enough volatile at room temperature to evaporate from the surface. The etching is therefore performed at elevated sample temperatures, typically > 200 °C. High etch rates are reported, also without intentional heating of the sample [98]. This can be explained by significant sample heating induced by the ion bombardment [99]. The heating by the ion bombardment complicates the temperature control of the sample, which will be further discussed in section 3.4.

Two ICP systems were used in this work: an Alcatel MET reactor¹ and an Oxford Instruments Plasmalab 100 system², both load-locked. Samples are glued with thermal paste onto a 4 in. carrier wafer, which is clamped to the substrate holder. The carrier wafer is made out of stainless steel (MET) or Si (Plasmalab). The temperature of the substrate holder, \( T_{\text{sub}} \), was kept to a preset value by a combination of constant liquid nitrogen- (MET) or water (Plasmalab) cooling and regulated resistive heating. Thermal contact between the carrier wafer and the substrate holder is provided by a He backside pressure \( p_{\text{He}} \). This causes a constant He-flow of several sccm into the chamber, which slightly reduces the partial pressure of the etching gases. In the MET-system, the temperature was measured with a Luxtron fluoroptic probe. This probe was placed in the He-backflow, less than 1 mm from the wafer. In the Plasmalab system, the temperature of the substrate holder was measured by a thermocouple.

¹ DIMES, Delft.
² TU/e.
A Langmuir probe was used to measure the ion current density, \( J \), to the sample and the plasma potential, \( V_p \). The value for the ion current density can be used to determine the ion yield (atoms removed per incoming ion) by the relation:

\[
Y = \frac{\rho_{InP}}{M_{In} + M_P} N_A R \frac{e}{J}.
\]  

Here \( \rho_{InP} \) is the density of InP (4.81\( \cdot \)10\(^3 \) kg/m\(^3 \) ([100], page 12-98)), \( M \) the molar mass of either In (0.115 kg) or P (0.031 kg) ([100], page 1-13), \( N_A \) Avogadro’s number, \( R \) the measured etch rate, and \( e \) the electronic charge. It is assumed here that the ions are singly charged. If ions with higher charge are also present, the yield will be underestimated by equation 6. The ions incident on the surface are accelerated over a total voltage that is the difference of \( V_p \) and \( V_{bias} \). The ion energy, \( E_{ions} \), is therefore equal to \( V_p - V_{bias} \), for singly charged ions, in units of eV.
2.3 Optical characterization

2.3.1 Optical set-up

A schematic overview of the optical set-up for characterization of the fabricated PPhCs is shown in Figure 2-12. It is called and end-fire set-up, because light from an external source is coupled into the ridge waveguides at the edge of the sample. The ridges are cleaved perpendicular to the long axis to obtain end facets, see Figure 2-13. It is important to remove the residual SiN$_x$ before cleaving, as the strain redistribution invoked by the cleaving causes the high
aspect ratio ridges to break.

The external light source is a Tunics BT diode laser with external cavity that is tunable in wavelength from 1470 nm to 1570 nm with a resolution of 1 pm. The polarization-controlled output light with a power of 1 mW is radiated to free space from a polarization maintaining (PM) fiber and collimated with a microscope objective with a numerical aperture (NA) of 0.25. The polarization output of the PM fiber is set to TE or TM with respect to the sample. The polarization of the light is further defined with a Glan-Thompson prism. The collimated beam is chopped at a modulation frequency of 310 Hz to allow phase-sensitive detection via a lock-in technique and focused at the end facets by microscope objectives with NA = 0.65. The Gaussian beam waist at the input-facet is estimated to be \( \sim 4 \, \mu m \), which implies a coupling loss of \( \sim 5 \, dB \) with the fundamental waveguide mode\(^1\). The first order mode of the waveguide is not excited because it is anti-symmetric with respect to the Gaussian beam profile. The second order mode (highest order for a waveguide width of 2.5 \( \mu m \)) will be excited, but with an intensity that is calculated to be 17 times lower than for the fundamental mode\(^1\). Also it will scatter more at waveguide imperfections than the fundamental mode. For these reasons only the fundamental mode is regarded in the remainder of this thesis. The sample and in- and out-coupling objectives are positioned on translational stages with differential screws for alignment in three directions. To optimize the position of the beam waist with respect to the waveguiding layer, the input objective has translational piezo control in three directions. This optimization is done by maximizing the detector signal. The focal point of the objectives is wavelength dependent, which causes transmission loss up to 50 % at the boundaries of the wavelength range, when the signal is optimized at \( \lambda = 1520 \, nm \). Therefore each crystal is measured in two wavelength intervals: 1470-1520 nm and 1520-1570 nm, with signal optimization at \( \lambda = 1495 \) and 1545 nm respectively. This reduces the transmission loss to less than 10 %. In the stop gap, the low signal only allows optimization of the lateral alignment. Structures that have transmission in the stop gap for the entire tuning range of the laser are laterally aligned at \( \lambda = 1520 \, nm \) and measured in one interval. If signal optimization is not possible at \( \lambda = 1495 \, nm \) (1545 nm), because it is below the stray light level for that wavelength, the crystal is measured for the entire wavelength range after signal optimization at \( \lambda = 1545 \, nm \) (1495 nm). A lens-

\(^1\) Calculated from the overlap of the eigenmodes of the ridge waveguide with the Gaussian beam waist. Performed by L. Augustin, Optoelectronic Devices (OED) group, faculty of Electrical Engineering, TU/e.
pinhole system after the output objective is used to spatially filter the transmitted signal and reduce the contribution of stray light. The signal is detected with an InGaAs photodiode in combination with a lock-in amplifier. An infrared camera is used to image the output facet for alignment purposes.

Lithographic tuning [15, 101] is employed to cover the photonic band gap with the limited tuning range of the laser. This means that transmission spectra of photonic crystal structures with various $a$, ranging from 250-600 nm, are measured with 1 nm wavelength steps. The trenches that define the ridge waveguides are 20 $\mu$m wide at both sides and the distance between the structures is 100 $\mu$m. The total length of the waveguides is 1-4 mm, with usually a similar segment on either side of the PhC. For normalization and alignment purposes several uninterrupted ridge waveguides, i.e. without PhC section, are included in between the PhCs. The total number of structures on a completed single sample with both $\Gamma K$- and $\Gamma M$ oriented crystals is $\sim$50 and the size of the chip is typically 2 x 8 mm$^2$. A schematic overview of the sample is shown in Figure 2-13. The six cavities that may lead to Fabry-Pérot oscillations in the measured spectrum are also indicated in the figure.

### 2.3.2 Incoupling with lensed fibers

Some measurements were performed with in-coupling using microlensed optical fibers instead of microscope objectives [102]. This set-up contains two tunable diode lasers, providing a wavelength range of 1410-1585 nm. The results with fiber in-coupling are compared to the measurements performed with objectives in Figure 2-14. Note the logarithmic vertical scale in the figure and the $\sim$50 dB extinction in the stop gap for the fiber coupling. No uninterrupted ridge waveguides were included on this sample. The measurements were therefore normalized with respect to one another using the average transmission in part of the dielectric band, $a/\lambda = 0.196$-$0.202$. Unfortunately, some of the ridge waveguides were broken, so that only parts of the spectrum could be assessed. The crystals with $a = 280$ nm and $a = 486$ nm were only measured with the objectives, while the crystal with $a = 336$ nm was only measured with fibers. Nevertheless, the measurements correspond very well to one another. The difference in transmission level in the stop gap, $a = 405$ nm, is most likely due to a difference in detection of stray light. The stray light arises from scattering in the

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1 These measurements were performed at the set-up of dr. A. Talneau in the Laboratory of Photonics and Nanostructures (LPN) in Marcoussis, France.
2 Sample reference MO139-S4A.
sample, because even in the stop gap, the signal drops several orders when the laser is deliberately turned off. Probably less stray light is captured at the output by the microlensed fiber compared to the microscope objective, because the physical aperture of the latter is orders of magnitude larger. No TE/TM conversion is induced in the PPhC by the vertical waveguide structure here, because it is symmetrical around the core [103]. However a small polarization conversion due to the slightly conical shape of the holes cannot be ruled out.

### 2.4 Infiltration

In this thesis, the approach to obtain tunable PPhCs is replacement of the low index dielectric (air) with another material. This material must satisfy two requirements: 1) The refractive index $n$ is tunable by external means (e.g. temperature, electric field, optical field); 2) The refractive index is small enough, so that the infiltrated PPhC still exhibits a photonic band gap. Figure 2-15 shows that the latter requirement is achieved for materials with $n < 2.25$, which includes all organic materials. In this thesis two classes of organic materials that also satisfy the first requirement are used: liquid crystals (LCs) and polymers. For LCs the possible tuning range is large but inherently slow, while for polymers, making use of non-linear optical properties, the tuning range is small but fast. These materials will be discussed in the following sections.

![Comparison of the PPhC transmission spectra with different in-coupling.](image)

Filling of the etched holes involves infiltration with a liquid material, which is
either the LC itself or a monomer that is subsequently polymerized. This infiltration relies on the capillary action of the liquid inside the ~200 nm diameter air holes. The wetting of the solid by the liquid, described by the contact angle $\theta$, is therefore an important parameter [104].

### 2.4.1 Polymers

Filling of submicron high aspect ratio features with polymer can be envisaged in two ways: 1) Infiltration with the polymer in solution followed by thermal evaporation of the solvent. 2) Infiltration with a liquid monomer followed by *in situ* polymerization. The first method is used in a first experiment to fill the holes with the e-beam resist polymethyl(methacrylate) (PMMA) from a solution in Anisole. The second method is applied here to obtain complete polymer filling of the PhC holes.

The monomer trimethylolpropane triacrylate (TMP-3A, Aldrich), see Figure 2-16a, is chosen for its cross-linking efficiency and relatively low viscosity. It is converted into poly-TMP-3A (PTMP-3A) by free radical polymerization [105]. A fraction of 0.5 wt. % azo-bisisobutyronitrile (AIBN, Fluka), see Figure 2-16b, is added to the TMP-3A to initiate the thermal polymerization process. Heat rather than ultraviolet radiation is used for dissociation of the initiator, because InP is not transparent for wavelengths below 1100 nm. The dissociation rate of the
AIBN must be carefully balanced, because the polymerization reaction is not properly initiated if it is too low and terminated too early if it is too high. The presence of unbounded oxygen severely inhibits the process, because it reacts efficiently with the radicals formed.

2.4.2 Liquid crystals

Liquid crystals [106] are materials that form one or more so-called liquid crystalline phases or mesophases in the transition from solid to liquid. These mesophases exhibit characteristics of both liquids (fluidity) and crystals (macroscopic order). This behavior arises from the anisotropic shape of the molecules, called mesogens, of which the rod-like or calamitic kind is discussed in this section. Several mesophases exist, but here only the so-called nematic phase is considered. In this phase, the long axis of the molecules is oriented on average along a certain direction, see Figure 2-17, called the director. Apart from this long range orientational order there is no long range translational order, so that the nematic phase can be considered as an anisotropic fluid. The order is in principle limited to domains, but LC in ~200 nm diameter holes is expected to form a single domain. The orientational order is characterized by the order parameter $S$ that is defined as $\langle 3/2 \cos^2(\beta) - 1/2 \rangle$. This is a statistical average over the angle $\beta$ between the mesogens long axis and the director. $S = 1$ in the crystalline solid phase, while $S = 0$ in the isotropic liquid phase, see Figure 2-17. In the nematic phase, $0 < S < 1$ and $S$ depends on the temperature $T$. The phase

![Figure 2-16: Structural formulas of TMP-3A and AIBN.](image)
transition between nematic and isotropic (liquid) at the so-called clearing temperature $T_c$, can be either continuous or discontinuous in $S$.

A nematic fluid is birefringent. Light experiences the ordinary refractive index $n_o$ if the electric field is polarized perpendicular to the long axis of the mesogen, and extraordinary index $n_e$ if it is polarized parallel to the long axis of the mesogen. In the isotropic phase, the birefringence is lost and the refractive index $n_i$ is approximated by:

$$n_i = \sqrt{\frac{1}{3} \left( 2n_o^2 + n_e^2 \right)}$$  \hspace{1cm} (7)
The average refractive index experienced by the transmitted light depends on the director field, the position dependent orientation of the mesogens in the holes. This average index can be modified either by thermal adjustment of the order parameter or by reorientation of the molecules with DC electric or magnetic fields. In all cases, the possible tuning range is determined by the initial director field in the PhC hole. For a nematic LC in a confined geometry, the director field is determined by the alignment of the mesogens at the boundary (surface anchoring) and the elastic properties of the LC. The surface anchoring is either homeotropic, i.e. the long axis perpendicular to the surface, or planar, i.e. parallel to the surface. The angle between the mesogen and wall in case of homeotropic alignment depends on the so-called anchoring strength. For parallel surface anchoring the director field is axial (AX), i.e. all mesogens oriented along the hole axis (see Figure 2-18a), because this configuration exhibits no defects or elastic deformations. In ref. [107] it is discussed that, for homeotropic alignment
and $r < 400$ nm, the director field is either planar polar (PP), i.e. the mesogens directed in the plane according to Figure 2-18b, or escaped radial (ER, possibly with point defects), i.e. the mesogens directed in the plane at the edge of the cylinder and ‘escaping’ to the vertical direction in the centre as in Figure 2-18c.

In this work the LC 4-pentyl-4-cyanobiphenyl (5CB, also called K-15) is infiltrated in the PPhCs. This material exhibits a crystalline-to-nematic phase transition at $T_{nc} = 23$ °C and a discontinuous nematic-to-isotropic phase transition at the $T = T_c = 35$ °C [108]. The refractive indices of the material at $\lambda = 1.5$ µm are $n_o = 1.516$, $n_e = 1.682$ and $n_i = 1.575$ [76]. It has been calculated for 5CB in cylinders with a size comparable to our PhC holes, that the director field configuration is ER in case of weak homeotropic anchoring of the mesogens at the sidewall [109].
Chapter 3
ICP etching results

The fabrication of low-loss PPhCs requires etching of the holes to a depth well below the waveguide core. In fact it was discussed in section 2.1.4 that fabrication imperfections are the dominant cause for out-of-plane optical losses if the sidewall angle in the lower cladding exceeds 1°, or if the etch depth is less than 2 µm. This prompts for an etching process that is anisotropic and capable of producing holes with aspect ratio (depth divided by diameter) larger than 10. Therefore dry etching techniques as ICP etching, ECR etching and CAIBE are applied for fabrication of PPhCs. Standard parallel plate RIE is less suited, because the ion energy and plasma density cannot be controlled independently with this technique.

Good etching results for InP-based PPhCs have been achieved with CAIBE [60, 61] and ECR [59] with Cl₂-based chemistry. The use of an ion beam in CAIBE requires relatively high vacuum, which makes it difficult to operate, notably when using corrosive gases like Cl₂. In ECR etching, the plasma may become unstable due to ‘mode-hopping’ [110, 111] and it has by now become replaced by ICP etching [111]. ICP etching is therefore a more reliable and versatile technique for high aspect ratio etching on a routine basis. Good results
on deep-hole ICP etching in InP with SiCl$_4$-chemistry have been reported [58].

In this chapter, detailed results are presented regarding ICP etching of PPhCs in InP-based material, using simple Cl$_2$ gas instead of SiCl$_4$ to provide the reactive species. It was shown in ref. [112], that sidewall passivation provides verticality of the sidewalls in pillar-type Si-based 2D PhCs. This was achieved by controlling the flux of O$_2$ to an SF$_6$ etching plasma. It was shown in ref. [113], that in that case the lateral etching is inhibited by the formation of an oxygen rich reaction layer on the sidewall with a thickness of less than a nanometer. On the bottom surface, this layer is removed by the ion bombardment. The selective etching of Si over its oxide then results in features with vertical sidewalls. In this thesis, the concept of sidewall passivation is extended to the etching of high aspect ratio features in InP-based material by addition of O$_2$ or N$_2$ to the Cl$_2$-plasma. The Cl$_2$/N$_2$-chemistry was recently used in an ICP etching process for PPhCs in GaInP/GaAs material [114]. However, any role of sidewall passivation was not discussed there.

### 3.1 Sidewall passivation: Cl$_2$/N$_2$-chemistry

To investigate the feasibility of sidewall passivation for InP, preliminary experiments were performed in the MET system (see section 2.2.5) for both Cl$_2$/Ar- and Cl$_2$/N$_2$ chemistry. The process parameters for the Cl$_2$/N$_2$ process are given in Table 3-1. For Cl$_2$/Ar, the process parameters are the same, only $P_p$ was set to 160 W to obtain the $V_{bias}$ of 100 V. The substrate holder temperature of 220 °C is the highest possible in the system, to promote the evaporation of InCl$_3$. These plasma parameters lead to an ion current density $J = 0.7$ mA/cm$^2$ for Cl$_2$/Ar and $J = 0.5$ mA/cm$^2$ for Cl$_2$/N$_2$, as measured with the Langmuir probe. The plasma potential was measured to be 25 V, so that $E_{ion} = 125$ eV. SEM images of ~350 nm wide trenches on a 500 nm pitch after 2 minutes of etching are shown in Figure 3-1. In the Cl$_2$/Ar process significant lateral etching occurs as can be concluded from the undercut at the sides of the structured field in Figure 3-1a. This undercut is so large, that the ridges defined by the trenches are completely

<table>
<thead>
<tr>
<th>Cl$_2$/N$_2$ flow (sccm)</th>
<th>$P_s$ (W)</th>
<th>$V_{bias}$ ($P_p$) (V (W))</th>
<th>$T_{sub}$ (°C)</th>
<th>$p$ (Pa)</th>
<th>$p_{He}$ (mbar)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25/75</td>
<td>1000</td>
<td>-100 (300)</td>
<td>220</td>
<td>1</td>
<td>14</td>
</tr>
</tbody>
</table>
etched away, leaving residual SiNₓ-mask parts. For the Cl₂/N₂-process no undercut is observed, as can be seen in Figure 3-1b. The sidewalls of the features are vertical, except for a slight bowing, which can be attributed to ion-induced effects [115, 116]. Evidently, the sidewalls are protected against lateral etching due to passivation effects. The composition of the material at the passivated sidewalls is unknown. In ref. [117] the formation of InN on the sample surface after ECR etching with Cl₂/N₂ chemistry is reported. It can therefore be speculated that the passivation layer on the sidewalls consists of InN. It may also be more complicated however, as it is found in ref. [118] for (001) InP that different chemical surface states are formed under irradiation by N⁺-ions, depending on the ion energy.

It was found that for this etching process a Cl₂:N₂ ratio of 1:3 is required for sufficient passivation, which implies that Cl₂ is the minority species in the plasma. This fundamental reduction of reactive chlorine supply lowers the InP etch rate compared to a Cl₂-only process. Furthermore, the InP etch rate is decreased by passivation of the bottom surface. These effects restrict the InP:SiNₓ selectivity, because the etching mechanism of the SiNₓ mask is mainly physical. Although photonic crystal holes with vertical sidewalls were achieved after some process optimization, the etch depth was limited to 1 µm with a SiNₓ mask thickness of 400 nm. PPhCs etched with this process would suffer from excessive optical losses, as discussed in section 2.1.4. Therefore other gases, notably O₂, were considered as passivation agents. No O₂ is available on the MET system, because also BCl₃ is used as an etching gas on this machine. Combination of BCl₃
and O2 can lead to formation of BO2-dust in the vacuum chamber. Therefore, experiments with Cl2/O2 were performed on the Plasmalab system (see section 2.2.5).

3.2 Cl2/O2-chemistry with moderate ICP power and high bias voltage

It was shown before, that high aspect ratio structures in Si can be obtained with fluorine (SF6) based etching using O2 for sidewall passivation [66, 112]. In refs. [119, 120] it was reported, that the native oxide can be used as mask material for free Cl2 etching of features in InP to a depth of 3 µm. This suggests that the addition of O2 to the Cl2-plasma may also be efficient in formation of a protective layer on the sidewall during InP etching. It has been shown by Smolinsky et al. that GaAs etches selectively with respect to its oxides in Cl2-plasma [121]. No selective etching of InP with respect to its oxides was observed in ref. 121. However, in their case the etching mechanism was probably rather physical as the substrate temperature (60°C) was significantly lower than required for chemical etching of InP. Cl2/O2 mixtures have been used in reactive ion etching (RIE) of InP laser stripe facets, producing vertical or even overcut sidewalls at 250°C [122].

In this section a first investigation of Cl2/O2-chemistry for etching high aspect ratio holes is described. The process parameters used for these experiments are given in Table 3-2. These process parameters lead to an ion current density of 0.4-0.7 mA/cm², and a plasma potential of 60-70 V, both depending on the O2 flow. The ion energy for this process is therefore ~600 eV. No backside cooling with He was used here, to prevent the influence of He-inflow on the plasma. Therefore the thermal contact between the carrier wafer and the substrate holder is limited to the physical contact at the edge of the carrier wafer. As a result, the heating of the carrier wafer by the ion bombardment [99] causes a radial temperature gradient over the wafer in this case. The sample was positioned on

<table>
<thead>
<tr>
<th>Cl2/O2 flow (sccm)</th>
<th>Ps (W)</th>
<th>Vbias (Pp) (V (W))</th>
<th>Tsub (°C)</th>
<th>p (Pa)</th>
<th>pHe (mbar)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7/variable</td>
<td>500</td>
<td>-530 (350)</td>
<td>250</td>
<td>0.3</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 3-2: Process parameters for the etching experiments of section 3.2.
the perimeter of the carrier, i.e. close to the substrate holder, to keep its temperature as close as possible to the preset temperature of the substrate holder. Furthermore, the heating of the sample was limited by short sequenced processing (etch steps of 30 s), allowing the sample to cool down between the etch steps. Due to the plasma stabilization time of less than 3 s the sequencing could lead to an underestimation (<10 %) of the etch rates.

### 3.2.1 Sidewall passivation by addition of O₂

In a first series of experiments the passivation capabilities of O₂ for the Cl₂ ICP etching process were investigated. Figure 3-2a-d show SEM images of a 10 µm wide trench after 60 s of etching (i.e. 2 etching steps) for O₂-flows of 0, 1.8, 2.6 and 7.0 sccm respectively. The Cl₂-only case (Figure 3-2a) shows a bowing of the sidewalls, which is probably due to direct impact of ions with a
slightly non-vertical trajectory [115]. By adding 1.8 sccm O\textsubscript{2} the sidewalls become more vertical, as can be seen in Figure 3-2b. The etch depth for the two processes is comparable: 4.7 µm (Cl\textsubscript{2}-only) and 4.9 µm (+1.8 sccm O\textsubscript{2}), while the bottom roughness slightly increases with the addition of oxygen to the plasma. With equation 6 and the measured value of \( J \) it is deduced that these etch depths imply an ion yield of \(~10^2\). In ref. [123], the ion yield for InP sputtering with 500 eV Ar\textsuperscript{+} ions was determined to be \(~1\). Therefore, \( Y = 10^2 \) suggests an ion-assisted chemical etching mechanism. When the O\textsubscript{2} flow is increased to 2.6 sccm (see Figure 3-2c), the bottom surface becomes very rough and the etch depth is significantly reduced (\(~2.5\) µm). This grass-like roughness has some darker features on top. These most likely consist of broken grass, clustered together during etching. This is observed more clearly when the O\textsubscript{2} flow is further increased (see Figure 3-2d). Evidently, at higher O\textsubscript{2} flows the enhanced passivation effect causes micro-masking at the bottom surface [116] and inhibits etching despite direct exposure to the ion bombardment. These results indicate that O\textsubscript{2} is indeed capable of protecting the InP surface against Cl\textsubscript{2}-etching, even under direct exposure to the ion bombardment.

The protection of the sidewalls is observed more clearly for high aspect-ratio holes. In Figure 3-3 cross-sectional SEM views of a 2D hole pattern with \( r = 100 \) nm and \( a = 400 \) nm are shown as etched with Cl\textsubscript{2} only (a) and with addition of 1.8 sccm O\textsubscript{2} (b). These crystals were on the same sample, and therefore etched in the same run, as the trenches shown in Figure 3-2a and b. In the Cl\textsubscript{2}-only case the
lateral etching in the top region is more pronounced than for the trench with openings in the sidewalls to the neighboring holes. This bowing in the top is well-known from deep silicon etching [116]. In that case it was shown with simulations, that the under etching is due to direct exposure of radicals from the plasma in combination with ions scattered from the mask. Upon addition of O₂ the sidewalls become more vertical, although a slight under etch is still visible. The holes are substantially deeper than for the Cl₂-only process (2.6 µm instead of 1.6 µm) and have a rough bottom surface. When O₂ is added, the sidewalls are protected against lateral etching and chlorine radicals are not consumed there. Consequently, more radicals are available at the bottom surface, which results in higher etch rate. In this way, an anisotropic etching process is obtained, leading to large hole depths.

### 3.2.2 Aspect ratio dependent etching as function of O₂ content

In the previous section it was shown that large and small areas behave differently upon O₂ addition. In this section a more systematic study of aspect ratio dependent etching (ARDE, [90]) is discussed. Figure 3-4 summarizes the dependence of etch depth, as measured from SEM images, on hole-diameter (200, 240, 400 and 960 nm) at different O₂ flows. The Cl₂ flow was 7 sccm while the O₂ flow was 0, 1.8, 2.6, or 7 sccm. For comparison, the etch depth of the 10 µm wide trench is also included. In the Cl₂-only case the etch depth decreases with decreasing hole diameter, also known as RIE-lag. When increasing the amount of

![Figure 3-4: Dependence of etch depth on hole diameter for a Cl₂ flow of 7 sccm and various O₂ flows.](image-url)
O₂ the RIE-lag is reduced, eventually leading to inverse RIE-lag (etch depth increase with decreasing hole diameter) at the largest O₂ flow. The consequences of O₂ addition for the overall etch behavior with respect to ARDE are twofold, each with a different impact. On one hand improved sidewall passivation leads to enhanced exposure of the bottom to reactive species, which will increase the etch rate. On the other hand improved bottom passivation leads inevitably to lower etch rates.

The results in Figure 3-4 indicate that the resultant of the two effects varies largely with aspect ratio (AR). In the lower AR regime (960 nm diameter hole, 10 µm trench) the enhanced bottom passivation and slow down of the etch rate dominates for increasing O₂ content after a slight increase at the lowest O₂ addition (1.8 sccm). In the high aspect ratio regime (200 and 240 nm diameter) the impact of sidewall passivation and the correspondingly enhanced flux of reactive species to the bottom dominates. The hole depth is independent of the O₂ flow in the range investigated. The same etch behavior is also observed using a higher Cl₂-flow of 14 sccm, but then the hole depth is significantly larger. This suggests that, in this etching regime, the ion-enhanced etching is limited by the supply of neutral Cl species. The abundance of neutrals at the bottom surface is strongly dependent on the aspect ratio of the hole [115]. Apparently, in this pressure- and flow regime, insufficient O₂ is available to passivate the bottom surface of the holes under the ion bombardment.

It is pointed out that with a proper Cl₂/O₂ balance the etching process can be tuned to aspect ratio independent etching, as manifested by the 2.6 sccm O₂ curve in Figure 3-4. This may be very useful when structures with various feature sizes are to be realized in a single etch step. Apart from all surface chemical effects at sidewall and bottom, the addition of oxygen increases the dissociation of chlorine since the strong electron attachment mechanism for chlorine is suppressed [124]. The systematic increase of the etch depth when 1.8 sccm of O₂ is added could be partly ascribed to this effect.
Because the results of the previous section indicate a neutral limited etching regime, a decrease in ion flux to the sample is not expected to have a large impact on the InP etch rate under constant neutral flux. This would be advantageous because the (physical) etching of the SiN$_x$ is reduced upon a decrease in $J$, thus increasing the InP:SiN$_x$ selectivity. The ion current density was decreased here by lowering the ICP source power to 250 W, although this will also affect the dissociation of the Cl$_2$ and hence the supply of Cl radicals. In Figure 3-5a the cross-sectional SEM view of a 2D hole pattern with $r = 100$ nm and $a = 400$ nm is shown after 60 s of etching with the process with parameters given in Table 3-3. Also in this experiment, no He backside cooling is used, so etching was done in two runs of 30 s and the sample was positioned on the perimeter of the carrier wafer. For this process $J$ was measured to be 0.02 mA/cm$^2$ and $V_p$ to be 40 V.

### Table 3-3: Process parameters for the etching experiments of section 3.3.

<table>
<thead>
<tr>
<th>Cl$_2$/O$_2$ flow (sccm)</th>
<th>$P_s$ (W)</th>
<th>$V_{bias}$ ($P_p$) (V (W))</th>
<th>$T_{sub}$ ($\degree$C)</th>
<th>$p$ (Pa)</th>
<th>$p_{He}$ (mbar)</th>
</tr>
</thead>
<tbody>
<tr>
<td>14/1.8</td>
<td>250</td>
<td>-640 (400)</td>
<td>250</td>
<td>0.3</td>
<td>0</td>
</tr>
</tbody>
</table>

Figure 3-5: Cross-sectional SEM view of a) holes with $r = 100$ nm on a triangular lattice with $a = 400$ nm and b) wide trench as etched with a process with low ICP power and high bias voltage.

### 3.3 Cl$_2$/O$_2$-chemistry with low ICP power and high bias voltage

Because the results of the previous section indicate a neutral limited etching regime, a decrease in ion flux to the sample is not expected to have a large impact on the InP etch rate under constant neutral flux. This would be advantageous because the (physical) etching of the SiN$_x$ is reduced upon a decrease in $J$, thus increasing the InP:SiN$_x$ selectivity. The ion current density was decreased here by lowering the ICP source power to 250 W, although this will also affect the dissociation of the Cl$_2$ and hence the supply of Cl radicals. In Figure 3-5a the cross-sectional SEM view of a 2D hole pattern with $r = 100$ nm and $a = 400$ nm is shown after 60 s of etching with the process with parameters given in Table 3-3. Also in this experiment, no He backside cooling is used, so etching was done in two runs of 30 s and the sample was positioned on the perimeter of the carrier wafer. For this process $J$ was measured to be 0.02 mA/cm$^2$ and $V_p$ to be 40 V.
Therefore $E_{\text{ion}} = 680$ eV.

The holes in Figure 3-5a are deep ($\sim 3.4 \, \mu$m) with almost vertical ($< 1^\circ$) sidewalls in the upper 2 $\mu$m. It is therefore expected, that this etching process yields low-loss PPhCs, see section 2.1.4. The bottom part of the high aspect ratio holes is narrow, which appears to be a commonly encountered feature in PPhC etching in InP [59, 60, 61], and may relate to ion shadowing effects [90]. It is visible in Figure 3-5a, that a substantial part of the mask is still present after etching (200 nm). The InP:SiN$_x$ selectivity for large area etching is 20 for this process, similar to the process described in the previous section (but with Cl$_2$ flow of 14 instead of 7 sccm). This is probably due to the higher ion energy here. The residual SiNx visible in Figure 3-5a has a thickness of 140 nm, which would allow another $\sim 30$ s of etching.

For the trench, depicted in Figure 3-5b, the whole upper region of the sidewall is very rough and there is a marked transition to the smoother lower region. Similar abrupt transitions in sidewall roughness have been observed before with other dry etching processes, including Cl$_2$/O$_2$-RIE and were attributed to redeposition of non-volatile etch products [122, 125]. This transition is also observed in the Cl$_2$ only case, though less pronounced (Figure 3-2a). The trench profile would lead to important propagation losses in the ridge waveguide, which makes this process less suitable when both ridge waveguides and PhCs have to be etched in a single process step.

### 3.4 Cl$_2$/O$_2$-chemistry with high ICP power and low bias voltage: influence of sample temperature

In the previous sections, no He back flow for thermal contact between the substrate holder and carrier wafer was used. It was shown by Sabin however, that the ion bombardment may lead to significant heating of the sample, with a temperature increase up to 75 $^\circ$C [99]. The InP etch rate by Cl$_2$ gas shows an activated dependence on the temperature [95], so it is expected that accurate control of the sample temperature is important. In the following, the influence of the He backside cooling and the temperature dependence of the etching are discussed. The process parameters for these experiments are given in Table 3-4. Although no Langmuir probe measurements were performed for this particular process, the values for $J$ and $V_p$ were estimated from values for other processes to be $\sim 1$ mA/cm$^2$ and $\sim 40$ V respectively, the latter leading to $E_{\text{ion}} \approx 270$ eV. Etching
was performed here in a single step, i.e. without the 30 s sequencing of the previous experiments.

3.4.1 He backside cooling

The importance of the He backside cooling is illustrated in Figure 3-6. The SEM views in this figure are trenches after 60 s of etching and result from four samples in two different etching experiments. In each experiment one sample was positioned at the perimeter of the carrier wafer (right of Figure 3-6), as in the experiments described in the previous sections, and the other in the centre of the carrier wafer (left of Figure 3-6). One of the experiments was performed without He backside cooling (top of Figure 3-6) and the other with cooling (11 sccm He flow into the reactor, bottom of Figure 3-6). For both, the temperature of the substrate holder was set to 250 °C. The difference in etching result with (Figure 3-6a and b) and without (Figure 3-6c and d) cooling is striking. For the samples etched with He backside cooling, the trenches exhibit a grass type surface roughness, while no grass is present for the trenches etched without cooling. In a separate experiment with the sample positioned in the centre of the carrier wafer, the same He backside pressure (24 mbar) and a preset temperature of 300 °C, the bottom surface is similar to the sample etched without cooling in the centre of the carrier wafer (Figure 3-6a). The high temperature may cause an enhanced lateral etching of the grass pillars. It can be concluded, that the He backside cooling partly prevents heating by the ion bombardment, leading to a sample temperature that may be ~50 °C lower than without cooling. This reduces the dependence of the sample temperature on fluctuations in \( J \) and/or \( E_{\text{ion}} \) and therefore improves the reproducibility of the etching.

<table>
<thead>
<tr>
<th>( \text{Cl}_2/\text{O}_2 ) flow (sccm)</th>
<th>( P_s ) (W)</th>
<th>( V_{\text{bias}} (P_p) ) (V (W))</th>
<th>( T_{\text{sub}} ) (°C)</th>
<th>( p ) (Pa)</th>
<th>( p_{\text{He}} ) (mbar)</th>
</tr>
</thead>
<tbody>
<tr>
<td>14/3.8</td>
<td>1000</td>
<td>-230 (160)</td>
<td>variable</td>
<td>0.3</td>
<td>24</td>
</tr>
</tbody>
</table>

Table 3-4: Process parameters for the etching experiments of section 3.4.
The difference in trench profile between the samples at the perimeter and in the centre of the carrier wafer suggests that a significant temperature gradient exists over the carrier wafer. This gradient is probably radial, regarding the circular symmetry of the reactor, with the highest temperature in the wafer centre. In the case without He backside cooling the difference in etching result is more pronounced, but this does not necessarily imply a larger temperature gradient. To estimate the temperature difference over the carrier wafer, several experiments were performed at varying substrate holder temperature but otherwise similar conditions as for the sample of Figure 3-6c. The trench profiles obtained in these

Figure 3-6: Cross-sectional SEM view of wide trenches etched without He backside cooling (a and b) and with cooling (c and d). The samples were positioned in the centre of the carrier wafer (a and c) or on the perimeter (b and d).
experiments were compared with Figure 3-6c and d. From this comparison it was estimated, that the temperature difference between the centre and the perimeter of the carrier wafer was less than 25 °C in the case of He backside cooling. A similar comparison for Figure 3-6a and b requires experiments at substrate holder temperatures larger than 300 °C, which have not been carried out.

3.4.2 Temperature dependence of the large area etch rate.

The Arrhenius plot of the large area etch rate, using the preset temperature values, is shown in Figure 3-7. It must be pointed out here, that the actual sample temperature is probably significantly higher [99]. The experiments were carried out with a He backside cooling pressure of 24 mbar and an O$_2$ flow of 2.6 sccm. All other process parameters are equal to those described in the previous section. The etch depths are determined with step height measurements using a mechanical stylus (Tencor Alpha-Step surface profiler). In the figure, three regimes can be observed. For low substrate holder temperatures (< 160 °C), the etch rate levels off to ~0.6 µm/min, which is significantly larger than expected for physical sputtering of InP [123]. It was discussed by Vernon et. al., that in the low temperature regime the surface is fully covered with the InCl$_3$ corrosion layer [96]. The corrosion layer is removed by physical sputtering with a relatively high rate, because it is only loosely bound to the InP surface [96]. Hence the significant etch rate in this temperature regime. When the temperature exceeds a certain critical value (at a substrate holder temperature of ~160 °C in this case),
the etch rate shows an activated behavior [95]. Although only two temperature points in this range are shown here (160 °C and 180 °C), intermediate points were investigated for Cl₂-only chemistry, confirming the activated behavior. The activation energy is determined from temperature data of various etching parameters. This yields a value of ~1.7 eV, which is similar to the thermodynamically predicted value for InCl₃ evaporation from the corrosion layer, as estimated from ref. [95]. For high temperature of the substrate holder, above ~180 °C, the etch rate is limited by the supply of reactive species to the etched areas. In this region the activation energy is determined to be in the order of 0.05 eV, which is similar to the value found by Sabin for high temperatures [99]. The steady increase of the etch rate in the supply limited regime was also observed in ref. [96]. There it was speculated that the increase is due to surface modifications that change the Cl₂ sticking or to changes in the chlorine stoichiometry of the etch products.

For the Cl₂-only process with otherwise the same etching parameters the etch rate dependence on temperature is equal to Figure 3-7, except at high temperature. There, the etch rate is larger in the Cl₂/O₂ case, possibly because of increased dissociation of the Cl₂ molecules in the plasma [124]. The large area etch rate is therefore mainly determined by the surface chemistry of the InP-Cl₂ system for this particular Cl₂/O₂ flow ratio and the small amount of O₂ does not play a significant role. In general, the favorable temperature regime for etching is the supply limited regime, where the etch rate is large and only weakly dependent on temperature. For the process parameters used in this section, this implies that the substrate holder temperature should be set to a temperature > 180 °C.

### 3.5 Cl₂/N₂ chemistry on the Plasmalab reactor

Experiments with Cl₂/N₂ chemistry were also performed on the Plasmalab system with the plasma parameters given in Table 3-5. The value for \( V_{\text{bias}} \) was dependent on the N₂ flow, which is reflected by the range noted in the table. The He backside cooling lead to a ~10 sccm He flow into the reactor. No Langmuir

<table>
<thead>
<tr>
<th>Cl₂/N₂ flow (sccm)</th>
<th>( P_s ) (W)</th>
<th>( V_{\text{bias}} ) (( P_p )) (V (W))</th>
<th>( T_{\text{sub}} ) (°C)</th>
<th>( p ) (Pa)</th>
<th>( p_{\text{He}} ) (mbar)</th>
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<td>-(190-250) (400)</td>
<td>250</td>
<td>0.3</td>
<td>24</td>
</tr>
</tbody>
</table>
probe measurements were performed for this chemistry. Figure 3-8a-d show SEM images of holes with a diameter of ~500 nm after 60 s of etching for N₂ flows of 0, 3, 5 and 10 sccm respectively.

In contrast to the results for the MET system, significant sidewall passivation is already obtained for a Cl₂:N₂ ratio of 5:1, judged from the difference in sidewall verticality and etch depth between Figure 3-8a and b (0 and 3 sccm N₂). When the N₂ flow is increased to 5 sccm, the hole depth is reduced compared to the process with 3 sccm, probably due to increased passivation of the bottom surface. This is also indicated by the increased roughness at the bottom of the hole. A further increase of the N₂ flow to 10 sccm leads to even smaller hole depth and to conical hole shape, probably due to excessive passivation of the sidewalls.
The cause for the large difference in Cl₂/N₂ flow ratio required for sufficient passivation in the two ICP systems used is unknown. To make a meaningful comparison between the two systems, more (plasma) diagnostics, e.g. measurement of the density of neutral species in the etching plasma, is required. It highlights that process transfer between different etching systems is not straightforward. The results for the Plasmalab system show a similar passivation effect for N₂ and O₂. By now, an ICP etching process for InP-based PPhCs with Cl₂/Ar/N₂ chemistry on a similar Plasmalab reactor as used in this thesis, has been developed by P. Strasser [126].

3.6 Etching samples for optical characterization

The experiments described in the previous sections were performed on InP etch test samples. For functional optical devices, the holes must be etched in an InP/InGaAsP heterostructure planar waveguide, see section 1.2. The etching result may be different for InGaAsP than for InP, which is illustrated in Figure 3-9. The SEM views in this figure are taken from both InP (Figure 3-9a) as well as heterostructure (Figure 3-9b) samples etched with the process described in section 3.4, with a preset temperature of 200 °C and an O₂ flow of 3.8 sccm. It can be observed in Figure 3-9, that the holes etched in the InP have vertical sidewalls, while the holes in the heterostructure samples exhibit a considerable sidewall angle in the InGaAsP layer, possibly because of excessive passivation. In most cases investigated however, the etch result is similar for the
heterostructure and plain InP. If not so, a small adjustment of the process parameters is sufficient to correct for the different etching behavior. In the example of Figure 3-9, vertical sidewalls are obtained for the heterostructure sample by lowering the O\textsubscript{2} flow to 2.6 sccm. This confirms the approach of process development using InP substrates and subsequent fine tuning of the process with (more expensive) heterostructure material.

For optical characterization, ridge waveguides and PPhC areas are monolithically integrated and etched in a single process step. Therefore, the ICP process of choice is a compromise between the quality of the holes and that of the ridge. The sidewall passivation concept developed and described in this chapter.

Figure 3-10: Cross-sectional SEM view of holes with \( r = 100 \text{ nm} \) on a triangular lattice with \( a = 400 \text{ nm} \), etched with a Cl\textsubscript{2} flow of 14 sccm and a) no O\textsubscript{2} at 180 °C, b) 3.0 sccm O\textsubscript{2} at 200 °C, c) no O\textsubscript{2} at 250 °C, and d) 3.8 sccm O\textsubscript{2} at 250 °C.
proves to be useful for this purpose, which is illustrated in Figure 3-10. For Cl$_2$-only chemistry, vertical sidewalls can be achieved at a preset temperature of 180 °C with otherwise the same the process parameters as in section 3.4, see Figure 3-10a. The same process at 250 °C (Figure 3-10c) is useless because of the lateral etch in the top part of the hole, which was also observed in Figure 3-3a. The holes in Figure 3-10b are etched at 200 °C with an O$_2$ flow of 3.0 sccm. Increasing the etching temperature causes a slight barreling of the holes, which can be compensated for by an increase in the O$_2$ flow for a larger passivation effect. The holes in Figure 3-10d are etched with a preset temperature of 250 °C and an O$_2$ flow of 3.8 sccm. With the Cl$_2$/O$_2$ chemistry good quality holes are obtained in a larger window in parameter space, notably concerning temperature, compared to Cl$_2$-only chemistry. This increased degree of freedom accommodates the combined optimization of hole profile and ridge profile.

In the course of the process development, four different etching processes are used in the fabrication of optical transmission samples as in Figure 2-13. The process parameters used in the ICP etching of these samples are summarized in Table 3-6. All samples were etched for 60 s (in 2 runs of 30 s without He cooling) with a reactor pressure of 0.3 Pa. The SEM results and analysis of the optical measurements are discussed in Chapter 4.

### 3.7 Conclusion

A Cl$_2$-based ICP etching process for fabrication of PPhCs in InP is developed. It is shown that the addition of O$_2$ to the Cl$_2$ plasma induces sidewall passivation. The large area etch rate shows an increase upon the addition of small amounts of O$_2$, possibly due to increased dissociation of the Cl$_2$ molecules in the plasma. When the O$_2$ flow is further increased, the etch rate decreases due to passivation.

<table>
<thead>
<tr>
<th>Process #</th>
<th>Cl$_2$/O$_2$ flow (sccm)</th>
<th>ICP power (W)</th>
<th>$V_{\text{bias}}$ (V)</th>
<th>Temperature (°C)</th>
<th>He-pressure (mbar)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>7/0</td>
<td>500</td>
<td>530</td>
<td>180</td>
<td>0</td>
</tr>
<tr>
<td>B</td>
<td>7/1.8</td>
<td>500</td>
<td>530</td>
<td>250</td>
<td>0</td>
</tr>
<tr>
<td>C</td>
<td>14/3.8</td>
<td>1000</td>
<td>235</td>
<td>200</td>
<td>24</td>
</tr>
<tr>
<td>D</td>
<td>14/2.6</td>
<td>1000</td>
<td>220</td>
<td>200</td>
<td>24</td>
</tr>
</tbody>
</table>

Table 3-6: Details of the processes used for the fabrication of optical transmission samples
of the surface. The depth of holes with r = 100 nm is independent of O₂ flow in the flow regime investigated. Therefore, the etch depth is independent of aspect ratio for a specific Cl₂/O₂-ratio.

The temperature dependence of the large area etch rate reveals three etching regimes in the temperature range investigated. For a substrate holder temperature (which is not equal to the temperature of the sample) below 160 °C, the etch rate levels off at 0.6 µm. In this regime the etching is most likely due to physical sputtering of an InCl₃ corrosion layer that fully covers the InP surface. Above 160 °C, the InCl₃ is enough volatile to evaporate from the surface and the etching proceeds with an activated dependence on temperature. For a substrate temperature larger than 180 °C, the etch rate is limited by the supply of reactive species to the etched areas. It is shown that He backside cooling reduces the heating of the carrier wafer induced by the ion bombardment, which allows for better control of the sample temperature.

Etching experiments with Cl₂/N₂ chemistry are performed in two different reactors. Sidewall passivation can be achieved with N₂ in both reactors, but at very different Cl₂/N₂ ratio. The reason for this is yet unknown. However it is shown for the Plasmalab reactor, that O₂ and N₂ have a similar passivation effect.

Holes with good quality (i.e. sufficiently deep and cylindrical) are obtained for both a process with low ICP power (250 W) and high bias voltage (-640 V) and a process with high ICP power (1000 W) and relatively low bias voltage (-230 V). The processes developed for InP material may require some fine tuning for heterostructure samples, although in many cases the etching profiles are similar. It is shown that with Cl₂/O₂ chemistry, holes with good quality are obtained within a larger window in parameter space, notably concerning temperature, compared to Cl₂-only chemistry. The application of the sidewall passivation concept to InP material thus accommodates the combined optimization of hole profile and ridge profile in etching of optical transmission samples.
Chapter 4
Optical characterization

Various methods are used to experimentally assess the optical properties of PPhCs. Frequently, the in plane optical transmission of PPhC structures, which can include (a combination of) defects, is measured for this purpose. This requires an incoming guided wave on one side- and collection of the transmission on the other side of the PhC. The incoming light arises either from excited nanostructures inside the waveguide core (ILS method, see section 1.3.2) or from an external source (end-fire method, see section 1.3.2). In the latter case, incoupling of the light in the waveguide core layer is mostly done by focusing at end facets, but also prism coupling [127] or integrated grating couplers [128] are used.

In the transmission measurements, additional information can be obtained by imaging the light scattered upwards (out-of-plane) by the PPhC in the far field, see for instance [129]. A fundamentally different method is so-called near-field scanning optical microscopy (NSOM) or, equivalently, photon scanning tunneling microscopy, which is often used in combination with a transmission set-up. For a recent review on NSOM, see ref. [130]. The guided PhC modes are detected by coupling the evanescent field into a propagating wave inside an optical fiber probe, which has a subwavelength aperture (25-100 nm). The probe
is raster scanned over the sample surface, while keeping the distance to the surface constant at 5-50 nm, to obtain topological information as well. With NSOM the characteristics of PhC modes can be assessed with a spatial resolution $\ll \lambda$. With a heterodyne interferometric approach, this can even be done dynamically [131]. The latter has been performed for instance for fs-pulses [132] and low group velocity modes [49] in PhC line defects.

It is possible to obtain information on the band structure of a PPhC from transmission measurements. This is done by analysis of the fine structure in the transmission bands that arises from interference of the Bloch modes between the PhC boundaries [101]. Alternatively, band structure information can be obtained from the angle dependent out-of-plane reflection spectrum [133]. The incoming light is coupled to a PPhC mode if the parallel components of the wave vectors match. For a specific input angle of the probe light with the sample surface this leads to resonances in the reflection spectrum at specific wavelengths. From these, the $(\omega, k)$-pairs for the specific PhC modes can be deduced and the full band structure is obtained by scanning the input angle. This method only resolves the bands above the light line, as the ones below the light line cannot couple to free space modes.

Optical end-fire transmission measurements for PPhCs were first performed by Krauss et. al. with a configuration similar to Figure 2-1, based on (Al)GaAs material [15]. They observed clear signatures of the photonic stop gaps with $> 2$ decades optical extinction in the gaps. This was reproduced by Chow et. al., who showed evidence of polarization conversion in the PhC due to the asymmetry of the vertical waveguide structure [103]. As the waveguide structure used for the PPhC in this thesis is symmetric, it induces no polarization conversion.

In the remainder of this chapter, the end-fire transmission measurements performed are discussed in detail. The results are analyzed in the framework of the loss parameter model as discussed in sections 2.1.3 and 2.1.4, to assess the quality of the etched holes. This approach combines the advantages of the end-fire method (see section 1.3.2) with the versatility of the loss parameter model. In the course of the process development, several PPhCs with large differences in hole quality are produced. Four of these processes are evaluated here, and an unprecedented loss parameter range is observed.
In closer view of Figure 2-4, there appears to be transmission within the stop gap at the TE, ΓK air band edge, with a peak transmission of almost 0.2. This is better visible in the close-up of Figure 4-1. The explanation for this may be the following. At the PhC entrance, light is diffracted (non-grating like) into allowed modes due to the finite lateral width of the waveguide end, possibly aided by Bragg scattering. PhC modes are allowed in progressively more crystal directions when going from the ΓM to the ΓK band edge. Light can travel through the crystal and reach the output waveguide due to continuous diffraction into allowed directions, even though the forward ΓK direction is forbidden. This mechanism causes transmission in the stop gap, which increases with $a/\lambda$ between the ΓM- and ΓK band edge. With the FD solver in Crystal Wave, intensity profiles were calculated for various frequencies around the air band edge. The results are presented in Figure 4-2 and illustrate the mechanism described above.

### 4.1 Transmission in the ΓK stop gap

Figure 4-1: Close-up of Figure 2-4a, the air band edge in the calculated TE, ΓK spectrum. The band edges as calculated with the PWE method (see section 2.1.1) are indicated by the dashed vertical lines. The transmission in the ΓK stop gap is highlighted in the figure by the dashed ellipse.
4.2 Normalization and fitting

The measured transmission spectra must be normalized for comparison with the simulated spectra of section 2.1. This can be tricky, because the width $w$ of the PhC field, see Figure 2-1, or rather the ratio $w/\lambda$, and thus the lateral losses increase with $a$. In the simulation however, the spectrum is normalized by the plain waveguide input power and the variation in $w/\lambda$ is accounted for by the variation in $\lambda$. The measured PhC spectra are therefore normalized with the transmission of an uninterrupted waveguide, i.e. without photonic crystal section. The width of the waveguide is 2.5 $\mu$m in both simulation and experiment, but its ratio to $\lambda$ varies significantly in the simulation only. This causes a difference in

Figure 4-2: Top view intensity profiles calculated with the FD method for characteristic frequencies around the air band edge. The color scale in order of increasing field is black, red, yellow, white (= saturated). The scale is magnified to emphasize the important phenomena here. The waveguide mode is excited at the left and travels to the right. a) In the photonic band gap, no modes allowed. b) Just above the $\Gamma M$ band edge. Light is diffracted in the $\Gamma M$ direction only (depicted in the figure). c) More crystal directions are allowed than with b) which causes the light to penetrate further into the crystal. d) Some leakage into the output waveguide is already visible on this scale. e) Peak frequency of the transmission, still in the $\bar{K} K$ stop gap. f) Well within the air band. The first order grating like diffraction beams are visible here and indicated in the figure.
diffraction at the waveguide end between simulation and experiment for $a/\lambda$ values deviating significantly from 0.26. It was verified with FDTD calculations taking into account the scaling of the waveguide width, that this has an influence on the transmission level of less than 5%. Therefore it is neglected here.

The spectrum measured for a ridge waveguide etched 4.7 µm deep with process D (see Table 3-6), is shown in Figure 4-3a. The fast oscillations in the
spectrum are of Fabry-Pérot type due to reflections between the end facets, cavity 6 in Figure 2-13. The Fourier transform of a spectral region limited to 5 nm to obtain sharp peaks is shown in Figure 4-3b. The corresponding wavelength spectrum was measured with 0.01 nm steps to resolve the higher harmonic peaks, and is shown in the inset of Figure 4-3b. The first Fourier peak appears at a cavity length of 1.1 mm, which is equal to the sample width. This peak therefore indeed represents the cavity that is formed by the end facets. The subsequent peaks are the higher harmonics at integer multiples of 1.1 mm. The waveguide propagation loss is calculated from the wavelength spectrum using the ratio of maximum and minimum transmission of the fringes (equation 5 in ref. [134]. The dielectric-air facet reflectance was calculated\(^1\) to be 0.33. A propagation loss of 2 dB/cm is deduced from the analysis, which is similar to the value found for ridge waveguides etched with an ICP process that was optimized for low waveguide losses [135].

The raw waveguide data is not to be used directly for normalization of the PhC spectrum, because the interference of the respective Fabry-Pérot fringes may cause exaggeration of the oscillations in the normalized spectrum. To smooth the waveguide spectrum, three methods are considered here: the average value, a linear fit, or filtering out fast oscillations using the Fourier smoothing option in Origin\(^2\). Care must be taken in interpretation of the smoothed spectrum at the edges of the data range, because the smoothing algorithm may cause slight misinterpretation of the slope there. Instead of smoothing the data after the measurement, it is also possible to suppress the Fabry-Pérot oscillations in the measurement, e.g. by anti-reflection coating on the end facets or by spectral broadening of the tunable laser output.

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\(^1\) Calculations performed for 3 \(\mu\)m high, 2.5 \(\mu\)m wide ridge with the vertical waveguide structure used in this work, by L. Augustin, Optoelectronic Devices (OED) group, faculty of Electrical Engineering, TU/e.

\(^2\) Software for graphical data representation. Reference Microcal Origin v7.0475.
The measured TE, ΓK transmission spectrum of the sample etched with process D is shown in Figure 4-4a, before and after Fourier smoothing but before normalization. The missing parts of the spectrum are due to broken ridge access waveguides for the corresponding crystals. The spectrum is normalized by dividing the value at each data point by the corresponding value of each of the three processed graphs in Figure 4-3a. No large differences are observed between the different normalization methods, except for the transmission in the dielectric band. There the linear and Fourier filtered normalization curves lead to unphysical transmission > 1 for this sample. The exact origin of both the linear
dependence of the waveguide transmission and the slow oscillations that remain after Fourier smoothing is unknown. Furthermore these dependencies can be different for the PhC spectra because of the interference that may arise from the multiple cavities as shown in Figure 2–13. Therefore the average waveguide transmission value is used for normalization. The quality of the waveguides varies over the sample, which leads to a spread in the average transmission value with a range of a factor of 1.5. As the largest value is used for normalization, the normalized transmission level may be underestimated by this factor.

The simulated PhC spectra are fitted to the measured ones at the sharpest spectral features in the smoothed spectrum: the band edges. As an example, a zoom-in of the TE, ΓK air band edge of Figure 4-4 is shown in Figure 4-5, which is the normalized measurement of the crystal with \( a = 402 \) nm. The simulated spectrum with fit parameters \( r/a = 0.2645 \) and \( \varepsilon'' = 0.07 \) reproduces the measurement well. To check the influence of normalization uncertainties, the fitting was also performed for the spectrum multiplied by 1.5. A good fit was obtained with \( r/a = 0.2635 \) and \( \varepsilon'' = 0.06 \), see Figure 4-5. The additional uncertainty in \( r/a \) and \( \varepsilon'' \) induced by this underestimation of the transmission is therefore in the order of 0.001 and 0.01 respectively for this structure. The error margin for the fit parameters is determined by varying these around the optimum value until the deviation from the measurement is unrealistic, as judged with the
bare eye.

4.3 Results

In Table 4-1, the basic characteristics of the samples evaluated are summarized. All crystals were designed to have $r/a = 0.30$. Cross-sectional SEM views of the characteristic hole profile for these samples are shown in the following sections with the optical data. Also, the profile of a 10-20 μm wide trench is shown, indicating the quality of the ridge waveguides.

### 4.3.1 Conical holes with a sidewall angle of 3° and a lateral etch in the top part

Sample A\(^1\) was obtained from ICP etching process A, which is defined in Table 3-6. A heterostructure sample with test patterns was etched along with sample A. The cross-sectional SEM views of ~200 nm diameter holes and the wide trench are shown in Figure 4-6. The holes are conical from top to bottom, 2.2 μm deep and exhibit a 30 nm wide lateral etch in the top 300 nm. Because of the considerable sidewall angle of ~3°, the optical loss is expected to be high for

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\(^1\) Original sample reference is MO139-S3B.
this hole shape. The sidewall of the 3.6 µm deep line in Figure 4-6 is rough in the top 1 µm and a lateral etch is present in the top 500 nm. Despite of these sidewall irregularities, the transmission of the waveguides was sufficient to perform the optical measurements1.

High losses are indeed evident from the measured transmission spectrum of sample A shown in Figure 4-7. The dielectric band transmission is reduced to

1 Initial test measurements on this sample were performed in the lab of the Optoelectronic Devices (OED) group, department of Electrical Engineering, TU/e, in collaboration with A.A.M. Kok.
~0.05, while the air band transmission is at most only slightly above the stray light level of ~10⁻⁴. A good fit to the measurements was obtained with fit parameters \( r/a = 0.33 \pm 0.02 \) and \( \varepsilon'' = 0.65 \pm 0.1 \) for the dielectric band, as shown also in Figure 4-7. In the air band only the signal for \( a = 551 \) nm \( (a/\lambda \approx 0.36 \) in Figure 4-7) may be above the stray light level. A fit to this signal is not reliable, as the contribution of stray light is significant. Nevertheless, \( \varepsilon'' \) must be > 1 to explain the low air band transmission. The high optical losses make process A useless for most applications. It may find use however in applications where coupling to the vertical direction is required, e.g. in improving the efficiency of light emitting diodes.

4.3.2 Conical holes with a sidewall angle of 1° and a lateral etch in the top part

Sample B¹ was obtained from ICP etching process B, defined in Table 3-6. An InP sample with test patterns was etched along with sample B. It was verified for this process, that the hole profile is similar for plain InP and heterostructure material. The cross-sectional SEM views of ~200 nm diameter holes and the wide trench are shown in Figure 4-8. The sidewall angle of the holes is improved with respect to sample A to 1°, at the expense of a slight ~10 nm widening of the lateral etch in again the top 300 nm. The depth of the holes is similar, but the line is much deeper, 6.2 \( \mu \)m. However, the sidewall profile of the line in the top 3 \( \mu \)m is similar to that of sample A. Therefore the waveguides on sample B are expected to be of equal quality. Unfortunately, no uninterrupted ridge waveguides are present on this sample. Normalization of the signal is therefore not straightforward. To normalize the spectrum it is assumed that the in-gap signal (stray light level) here is also at ~10⁻⁴ of the ridge waveguide signal. This is reasonable considering that the waveguide quality is similar to that of sample A, but will result in larger normalization errors.

¹ Original sample reference is MO139-S4B.
The TE, ΓK transmission spectrum of sample B is shown in Figure 4-9. From this figure it is clear, that the improvement in sidewall angle has a major effect on the optical properties of the PPhC, notably in the air band. While the dielectric band transmission is improved with a factor of ~5, the air band transmission is recovered with > 2 orders of magnitude. The band edges are steeper and the extinction in the ΓK stop gap is > 30 dB compared to the dielectric band transmission. Also, the pseudo gap can be observed around \( a/\lambda = 0.38 \). A good fit
was obtained here with fit parameters $r/a = 0.29 \pm 0.01$ and $\varepsilon' = 0.35 \pm 0.1$ for the dielectric band and $r/a = 0.295 \pm 0.01$ and $\varepsilon' = 0.35 \pm 0.1$ for the air band. The measured band edges are reproduced well by the simulated ones, which is an indication that the normalization was indeed correct. The error margins are taken larger than usual here to account for the normalization uncertainty. Despite the large improvement in hole quality, the transmission level in the air band is only $\sim 0.04$ which means that the out-of-plane losses are still excessive for this hole profile. The measured transmission level in the pseudo gap is an order of magnitude lower than in the simulation.

### 4.3.3 Holes with a sidewall angle of 2.5° only in the core

Sample C\(^1\) was obtained from ICP etching process C, defined in Table 3-6. A test pattern was included on the chip and cleaved off for SEM inspection after etching. The cross-sectional SEM views of $\sim 200$ nm diameter holes and the wide trench are shown in Figure 4-10. The hole depth is increased compared to process B to $3.4 \, \mu m$ and the hole profile is cylindrical, except for the constriction at the core level, which leads to a sidewall angle of $2.5^\circ$ in the core. The taper in the bottom $1 \, \mu m$ is a common feature in deeply etched PPhCs [59, 60, 61]. The improvement in the hole quality is at the expense of the waveguides for this process, which suffer from large, $120$ nm wide lateral etch in the top $1 \, \mu m$ and severe ‘grass’ formation in the open area. It was found that these waveguides are indeed very lossy with a transmission decrease of 2 orders of magnitude

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\(^1\) Original sample reference is MO139-S6A.
compared to sample A. Even with these low quality waveguides, the transmission spectrum of the PPhC could be measured. Both $\Gamma K$ and $\Gamma M$ configuration are included on sample C and both are measured for TE and TM polarization. The TE, $\Gamma M$ and TM, $\Gamma K$ spectra are shown in Figure 4-11a and Figure 4-11b respectively.

Even though the level in the transmission bands differs slightly between $\Gamma K$
and \( \Gamma \)M (see Figure 2-4), it is obvious from Figure 4-11a that the TE spectrum of sample C is significantly improved compared to that of sample B. Notably, the air band transmission level is \( \sim 0.6 \). Due to the low waveguide transmission, the stray light level is more than an order of magnitude higher. This is because the output signal is attenuated in the output waveguide, while the stray light is not. Another consequence of the high waveguide loss is suppression of the fast Fabry-Pérot fringes in the original data. A good fit was obtained with fit parameters \( r/a = 0.24 \pm 0.01 \) and \( \varepsilon' < 0.15 \) for the dielectric band and \( r/a = 0.21 \pm 0.01 \) and \( \varepsilon' = 0.09 \pm 0.03 \) for the air band. An error margin for \( \varepsilon' \) at the dielectric band edge was difficult to obtain because only the rising edge could be measured, where the spectrum is relatively insensitive to this parameter. The pseudo gap is apparent in the graph, but again it is much deeper experimentally than in the simulation.

In the TM, \( \Gamma \)K spectrum, the expected pseudo gap around \( a/\lambda = 0.23 \) is clearly observed. A second pseudo gap is observed around \( a/\lambda = 0.34 \). The simulated spectrum with crystal parameter \( r/a = 0.225 \) and \( \varepsilon' = 0.11 \) is also shown in Figure 4-11b. Also for these pseudo gaps the difference between experiment and simulation is apparent. Although the measured edge position and transmission profile of the first pseudo gap seem to coincide with the simulated one, it is not possible to make a quantitative fit.

### 4.3.4 Holes with vertical sidewalls in the upper 2 \( \mu \)m

Sample D\(^1\) was obtained from ICP etching process D, defined in Table 3-6. On this sample also, a test pattern was included and cleaved off for SEM inspection after etching. The cross-sectional SEM views of \( \sim 200 \) nm diameter holes and the wide trench are shown in Figure 4-12. The hole depth is further increased compared to process C to 4.1 \( \mu \)m and the hole profile is cylindrical without constriction at the core level, but with a tapered section in the bottom 1.5 \( \mu \)m. For this process, also the waveguide quality is good, with only a 90 nm wide lateral etch in the top 400 nm, a height of 4.7 \( \mu \)m, and a smooth bottom surface of the open area. Both \( \Gamma \)K and \( \Gamma \)M configuration are included on sample D, which are both measured for TE and TM polarization. After first cleaving of the facets, many ridge waveguides appeared to be broken, probably because the residual SiNx was not removed before cleaving. Fortunately, most of the fractures were close to the sample edge, so that new facets could be cleaved in the

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\(^1\) Original sample reference is MO350-S4A.
waveguide part that was still intact. The TE, ΓK and TM, ΓM spectra measured after the last cleaving are shown in Figure 4-13a and Figure 4-13b respectively.

The spectra are slightly improved compared to sample C, with a further reduction in the loss parameter with a factor of ~1.5. A good fit for the TE, ΓK spectrum was obtained with fit parameters \( r/a = 0.265 \pm 0.01 \) and \( \varepsilon'' = 0.24 \pm 0.07 \) for the dielectric band and \( r/a = 0.265 \pm 0.005 \) and \( \varepsilon'' = 0.07 \pm 0.02 \) for the air band. The spectrum for \( a/\lambda > 0.32 \) could not be measured because the access waveguides for the particular crystals were broken. The same holds for the TM, ΓM. Furthermore, also the waveguide for \( a = 337 \) nm was broken. The crystals with \( a = 317 \) and 357 nm were masked by dirt during e-beam exposure of the ZEP, which yields interrupted waveguides with lower transmission. A good fit for the TM, ΓM spectrum was obtained with fit parameters \( r/a = (0.235 \pm 0.01) \) and \( \varepsilon'' = 0.10 \). Note the high transmission level of 0.8 in the air band. The transmission level in the dielectric band is lower than in the fit, but also here the spectrum is too insensitive to \( \varepsilon'' \) to assign a meaningful error margin to this parameter.

Figure 4-12: Cross-sectional SEM view of a) hole profile and b) trench profile of test patterns on the same chip as sample D.
Discussion

A summary of the results is given in Table 4-2. Indicated are the lattice constant for the crystal(s) under consideration, fit parameters $r/a$ and $\varepsilon''$, the calculated value for $\varepsilon''_{\text{int}}$ (see section 2.1.4) and $\varepsilon''_{\text{ext}}$ as determined from $\varepsilon''_{\text{ext}} = \varepsilon'' - \varepsilon''_{\text{int}}$. Because no reliable fit could be found for the measured TM, $\Gamma K$ spectra,

Figure 4-13: a) Measured TE, $\Gamma K$ transmission spectrum of sample D. The simulated spectra for fit parameters $r/a = 0.265$, $\varepsilon'' = 0.24$ and $r/a = 0.265$, $\varepsilon'' = 0.07$ are also included. b) Measured TM, $\Gamma M$ transmission spectrum of sample D. The simulated spectrum for fit parameters $r/a = 0.235$, $\varepsilon'' = 0.10$ is also included.
For the dielectric band $\varepsilon'' = 0$ is assumed, because it is below the light line. For the TM spectra, only fit parameter $r/a$ is given, because the band edges are almost independent of $\varepsilon''$ for this polarization.

Table 4-2: Summary of the optical results.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Spectrum (D = dielectric band, A = air band)</th>
<th>a (nm)</th>
<th>$r/a$</th>
<th>$\varepsilon''$</th>
<th>$\varepsilon''_{int}$</th>
<th>$\varepsilon''_{ext}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>TE, ГК, D</td>
<td>325, 343</td>
<td>0.33 ± 0.02</td>
<td>0.65 ± 0.1</td>
<td>0</td>
<td>0.7</td>
</tr>
<tr>
<td></td>
<td>TE, ГК, A</td>
<td>-</td>
<td>-</td>
<td>&gt; 1</td>
<td>0.034</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>TE, ГК, D</td>
<td>325, 343</td>
<td>0.29 ± 0.01</td>
<td>0.35 ± 0.1</td>
<td>0</td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>TE, ГК, A</td>
<td>465, 486</td>
<td>0.295 ± 0.01</td>
<td>0.35 ± 0.1</td>
<td>0.025</td>
<td>0.3</td>
</tr>
<tr>
<td>C</td>
<td>TE, ГК, D</td>
<td>299, 317</td>
<td>0.23 ± 0.01</td>
<td>&lt; 0.3</td>
<td>0</td>
<td>&lt; 0.3</td>
</tr>
<tr>
<td></td>
<td>TE, ГК, A</td>
<td>379</td>
<td>0.235 ± 0.005</td>
<td>0.11 ± 0.04</td>
<td>0.010</td>
<td>0.10</td>
</tr>
<tr>
<td></td>
<td>TE, ГМ, D</td>
<td>282</td>
<td>0.24 ± 0.01</td>
<td>&lt; 0.15</td>
<td>0</td>
<td>&lt; 0.15</td>
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<tr>
<td></td>
<td>TE, ГМ, A</td>
<td>357</td>
<td>0.213 ± 0.005</td>
<td>0.09 ± 0.03</td>
<td>0.007</td>
<td>0.08</td>
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<tr>
<td></td>
<td>TM, ГМ, D</td>
<td>282</td>
<td>0.255 ± 0.01</td>
<td>-</td>
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<tr>
<td></td>
<td>TM, ГМ, A</td>
<td>299</td>
<td>0.217 ± 0.005</td>
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<td>-</td>
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<tr>
<td>D</td>
<td>TE, ГК, D</td>
<td>317</td>
<td>0.265 ± 0.01</td>
<td>0.24 ± 0.07</td>
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<td>402, 427</td>
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<td></td>
<td>TE, ГМ, D</td>
<td>266, 282</td>
<td>0.243 ± 0.01</td>
<td>0.10 ± 0.05</td>
<td>0</td>
<td>0.10</td>
</tr>
<tr>
<td></td>
<td>TE, ГМ, A</td>
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<td>0.235 ± 0.005</td>
<td>0.06 ± 0.02</td>
<td>0.010</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>TM, ГМ, D</td>
<td>282</td>
<td>0.245 ± 0.01</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>TM, ГМ, A</td>
<td>299</td>
<td>0.235 ± 0.005</td>
<td>-</td>
<td>-</td>
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</tr>
</tbody>
</table>
Both $r/a$ and $\varepsilon''$ vary significantly between the samples, for reasons that will be discussed in the following sections. For the individual samples, the different measurements (crystal direction and polarization) do show consistency. Specifically, the fitted $r/a$ values are consistent for different polarization and the fitted $\varepsilon''$ values are consistent for different crystal direction. The former is required in case that these are measurements for exactly the same structures. The latter should also be the case, because the hole shape is expected to be similar for $\Gamma K$ and $\Gamma M$ structures on the same chip. The slightly lower $\varepsilon''$ values for $\Gamma M$ can be explained by the lower $r/a$ for these structures, in view of the scaling law in equation 5. Also the air band edge is at lower $a/\lambda$ for $\Gamma M$ than for $\Gamma K$, because of the different periodicity in the two directions.

On top of the consistency in $r/a$ for different polarizations, there is also consistency in $r/a$ between the air band and dielectric band. This highlights the versatility of the patterning process, which allows for production of PPhC structures of different size with rather constant $r/a$. The only discrepancy in this respect is found for the $\Gamma M$, $a = 282$ nm structure on sample C. It was already observed with SEM from top after RIE etching of the mask, that this single structure had a significantly larger $r/a$ than the other crystals. This is most likely due to a design error, also considering that the TM measurement of the adjacent, $a = 299$ nm, crystal is consistent with the TE measurement of the air band.

The fact that higher values for $\varepsilon''$ are measured in the dielectric band than in the air band, notably for samples C and D, is not consistent with equation 5 (section 2.1.4). The scaling law is only valid though, if the respective crystals have similar hole quality. That is not the case here due to the etching lag effect in either the RIE of the Si$N_x$ or the ICP etching of the deep holes. For sample D it was observed that the smallest holes, which are in the dielectric band, are not completely opened in the Si$N_x$ mask. This results in decreased etching depth after ICP etching, see Figure 4-14a for holes with $r = 45$ nm. The hole depth shown in the figure would result in huge optical loss, but as the holes in the dielectric band of sample D have $r > 70$ nm, the etch depth will be considerably larger there. The etching lag in ICP process C causes considerable decrease of etching depth for the smaller holes on sample C. The hole profile for $r = 70$ nm, which resembles the hole size of the dielectric band edge of sample C, is shown in Figure 4-14b. The cylindrical part of the hole extends to a depth of $\sim 1.4$ $\mu$m only. With a cone angle of $\sim 4^\circ$ in the remaining part of the hole, considerable optical loss can be expected.
4.4.1 Variation in \( r/a \)

The fitted \( r/a \) values for \( \Gamma K \) structures exhibit large variation between the samples and differ significantly from the designed value of 0.30 for all but sample B. It can be seen in close inspection of Figure 4-8b however, that the hole diameter at the waveguide core level is larger than the mask opening for this sample. This widening is due to a lateral etch component in the ICP process and implies that the patterns in the SiN\(_x\) mask had a smaller \( r/a \) than the fit value of 0.30. Sample D shows no hole widening and the measured \( r/a = 0.265 \) therefore represents the pattern in the mask. Assuming \( r/a = 0.265 \) also for the mask pattern of sample B, the resulting \( r/a \) in the core is estimated using the SEM results shown in section 4.3. This is done by measuring \( r \) at the top and bottom of the core layer and comparing the average with the mask opening. This indeed explains the measured \( r/a = 0.30 \) for sample B. Also for sample C, the narrowing in the core and the measured \( r/a \) are consistent, assuming \( r/a = 0.265 \) in the mask pattern. For sample A the optical measurement and hole widening are consistent only if \( r/a = 0.30 \) is assumed in the SiN\(_x\). Only for sample A the e-beam patterning was performed in the Raith at 30 kV, which may explain the discrepancy here. It is concluded that the patterning process developed for the Leica e-beam system leads to smaller holes than designed with the specific electron doses used, \( r/a = 0.27 \) instead of 0.30, but with a constant \( r/a \) over the lithographic tuning range. This could be caused by a systematic overestimate of the hole radius in the ZEP when measuring from top in the SEM. Cross-sectional evaluation of dosetests may be required instead.

Figure 4-14: Cross-sectional SEM view of: a) Hole pattern from sample D with \( r = 45 \) nm. b) Hole pattern from sample C with \( r = 70 \) nm.
A difference in \( r/a \) of \(~0.02\) is observed between \( \Gamma K \) and \( \Gamma M \) configuration, for both sample C as sample D. Also this difference probably originates from the e-beam patterning. The \( \Gamma M \) field is wider by a factor of \( \sqrt{3} \) and the holes are therefore on average further away from the large exposed areas for this configuration. This results in a lower total dose and thus a smaller hole radius than for \( \Gamma K \). This effect should be largest in the air band because \( w \) is largest there. The somewhat larger \( r/a \) found for the TE, \( \Gamma M \) dielectric band of sample D compared to \( \Gamma K \) dielectric band of sample D is therefore in line with the explanation. The dosetests are only performed for \( \Gamma K \) configuration and need to be performed for \( \Gamma M \) as well to correct for this effect.

### 4.4.2 Contributions to \( \varepsilon_{\text{hole}} \) in the air band

Various deviations from perfect cylindrical shape are present in samples A-D: lateral etch in the upper cladding, taper in the core layer and (truncated) cone shape in the lower cladding. The contributions of these features to \( \varepsilon_{\text{hole}} \) are estimated in this section, by combining the measured values for \( \varepsilon_{\text{ext}} \) in the air band, assuming \( \varepsilon_{\text{ext}} = \varepsilon_{\text{hole}} \), and using the analysis of the hole shape in the lower cladding in section 2.1.3.

The model described in section 2.1.3 assumes perfect cylindrical hole shape from top to the bottom of the core layer. Any serious deviations, notably in the core layer, are assumed to cause excessive loss, because the electric field is largest there [64]. The results for sample C do not confirm these expectations. Despite the severe sidewall angle of \( 2.5^\circ \) in the core layer, which is comparable to that of the very lossy sample A \((\sim 3^\circ)\), \( \varepsilon_{\text{hole}} \) is only \( 0.09 \) in the air band. A possible explanation for this based on geometric optics is illustrated in Figure 4-15. In the 1D picture of this figure, a ray traveling to the right is refracted at the two sidewalls. At the left of this figure, a ray traveling downward is shown, at angle \( \phi_h \geq 0 \) with the horizontal. The ray is directed further away from the horizontal by the double refraction. The increase in \( \phi_h \) can be calculated by application of Snell’s law. For the InGaAsP-InP interface, the critical angle \( \phi_c \) is \( 71^\circ \) (\( 19^\circ \) with the horizontal). For a sidewall angle of \( 2.5^\circ \), starting from a horizontal ray, \( \phi_h < \phi_c \) still after passing four holes. Within this distance, the ray has reached the bottom InGaAsP-InP interface and is directed upwards by total internal reflection. Upward traveling rays are directed back to the horizontal by the double refraction at the sidewalls, as shown at the right of Figure 4-15. A considerable sidewall angle in the core layer will therefore not lead to excessive loss, because the
refracted light is recaptured.

The contribution to $\varepsilon_{\text{hole}}'$ of the lateral etch in the upper cladding can now be estimated using the optical results for sample B and the discussion above. In view of the results for sample C, the small sidewall angle in the core would lead to a contribution $\ll 0.1$ to $\varepsilon_{\text{hole}}'$ for this sample. In the bottom cladding, the hole has truncated cone shape with $h = 2.2 \, \mu m$ and $\beta = 1^\circ$, with can be estimated from Figure 2-7 to contribute $\sim 0.05$ to $\varepsilon_{\text{hole}}'$. Therefore it is concluded that the lateral etch in the upper cladding leads to $\varepsilon_{\text{hole}}' \approx 0.3$.

Sample A exhibits a similar lateral etch as sample B, hence it is assumed that it contributes $\sim 0.3$ to $\varepsilon_{\text{hole}}'$ for this sample also. The hole profile in the core is similar to sample C, so this may add $\sim 0.1$ to $\varepsilon_{\text{hole}}'$ for sample A. The part of the hole in the lower cladding has simple cone shape with $\beta = 3^\circ$ so its contribution to $\varepsilon_{\text{hole}}'$ can be estimated from Figure 2-7 to be 0.3. These contributions add up to $\varepsilon_{\text{hole}}' = 0.7$, which is equal to the measured value in the dielectric band. For these high values of $\varepsilon'$ and with $n'$ fixed to 1 in the simulation it can be deduced from equation 2, that $\varepsilon' < 0.9$. It is therefore questionable if the perturbative approach and the scaling laws derived from it are still valid in this regime. Even if they were, simple application of the scaling law is not straightforward here, because the quality of the holes may differ for the dielectric band and the air band. Also, the uncertainties in $\varepsilon_{\text{hole}}'$ are high. A deviation of $1^\circ$ in the sidewall angle in the lower cladding leads to a difference of 0.2 in $\varepsilon_{\text{hole}}'$. For these reasons, a reliable
quantitative comparison of sample A with the other samples is not possible.

The fitted value of $\varepsilon_{\text{hole}} \approx 0.05$ in the air band for sample D cannot be explained from the tapered profile in the bottom part of the hole, judged from Figure 2-7. Most likely it is caused by the local fluctuations in hole radius and sidewall angle that are visible for the holes in Figure 4-12a, notably the slight widening just below the core. The loss parameter value found here is comparable to those reported for ICP etched PPhCs ($\varepsilon'' = 0.056$ [58]) and close to those reported for PPhCs etched with CAIBE ($\varepsilon'' = 0.045$ [136]) for the InP material system. This highlights the versatility of the Cl$_2$/O$_2$- chemistry used for the ICP etching, although the $r/a$ value and the combination of hole shape and vertical waveguide structure must be taken into account for a precise comparison [136].

4.4.3 Pseudo gaps

Pseudo gaps for different polarization and crystal direction are observed for sample B, C and D. In all cases, they could not be accurately reproduced in the FDTD simulation, the measured transmission level being significantly lower. Recall that the cause of the pseudo gaps is the symmetry mismatch between the waveguide mode and the PhC mode. When the access waveguides are removed in the simulation and plane wave input is used, the simulated transmission level in the pseudo gap decreases with 2-3 orders of magnitude. The fundamental difference is that the symmetry mismatch is present along the entire input for plane wave excitation, while it is strictly valid only at the centerline of the waveguide in case of excitation with the eigenmode of a ridge waveguide. Coupling to the anti-symmetric PhC mode is possible, probably due to the significant field gradient towards the edges of the ridge. This could also explain the discrepancy between experiment and simulation because the under etch causes a difference between the two at the edge of the ridge. Experimentally the intensity may be lower at the waveguide edge, because the light is scattered away by the imperfections there. Consequently, the experimental transmission is lower than simulated. The decrease of the in gap transmission with frequency for TM, $\Gamma K$, was also observed in the similar measurements of ref. [15] and is reproduced in the simulation.

4.5 Conclusion

End-fire transmission measurements are performed for both polarizations on deeply etched InP-based PPhCs with different hole quality. 2D FDTD
calculations, in which optical losses are simulated by a fictitious absorption in the holes, are fit to the optical measurements using $r/a$ and $\varepsilon''$ (the imaginary component of the dielectric constant in the holes) as free parameters.

For all samples, the $r/a$ value found for the dielectric band is similar to that of the air band. Also it was deduced from a comparison with SEM results that the pattern in the SiN$_x$ mask has $r/a = 0.27$ for all samples patterned with the Leica 5000 e-beam pattern generator. This indicates that the patterned $r/a$ is rather constant over the lithographic tuning range and for different samples and highlights the reproducibility of the patterning process. The deviation from the design value of $r/a = 0.30$ may be due to a systematic overestimation of the hole radius in the SEM evaluation of EBL dosetests from top.

The out-of-plane losses induced by imperfect hole shape are evaluated for the different samples using $\varepsilon'_{\text{hole}}$ as a figure of merit. This hole shape induced loss parameter is obtained from the measured $\varepsilon''$ and the calculated extrinsic loss parameter $\varepsilon'_{\text{int}}$. The values for $\varepsilon'_{\text{hole}}$ found for the air band are consistent for the $\Gamma K$- and $\Gamma M$-oriented crystals on the same chip, which are expected to have similar hole shape. This indicates that the $\varepsilon'_{\text{hole}}$ values found are reliable. The $\varepsilon'_{\text{hole}}$ values for the dielectric band compared to the air band do not satisfy the usual scaling with $r/a$ and $a/\lambda$. This is due to the lower quality of the holes with smaller $r$ in the dielectric band, caused by the RIE-lag in either the mask opening or the ICP etching of the holes.

It was shown that a considerable sidewall of $2.5^\circ$ in the core layer does not lead to excessive losses, in contrast to the common belief that cylindrical hole shape in the core is imperative. Qualitatively, this is probably due to recapture of the refracted light in the core. Using this knowledge, the contribution to $\varepsilon'_{\text{hole}}$ of a $\sim$40 nm wide lateral etch in the top 300 nm is estimated to be $\sim$0.3. A value for $\varepsilon'_{\text{hole}}$ as low as 0.05 was found for the best quality PPhC. This is similar to values reported in the literature by other groups for deeply etched InP-based PPhCs.

Significant transmission within the $\Gamma K$ stop gap was observed both in simulation and experiment. It is shown that this is due to the finite width of the input waveguide, which causes diffraction into allowed crystal directions. Light can travel through the crystal by continuous diffraction into allowed directions and a significant part is captured by the output waveguide, even if the forward direction is forbidden.
Chapter 5
Filling the air holes

The functionality of PPhC structures is increased when their optical properties are dynamically tunable by external means as heat and electric, magnetic or optical fields. In practice this can be achieved in semiconductor based PPhCs by adjusting the refractive index of either the high- or low index part, although tuning using micro-electro-mechanical systems [137], deformation [138, 139], or an atomic force microscope tip [140], is also reported. Tuning of PPhCs using the high index has been proposed [141, 142] and demonstrated [143], but the properties of this material are usually dictated by the (active) application. An attractive possibility is therefore infiltration of the air holes with an organic material that has a tunable refractive index. This allows for increased design freedom, because the properties of the semiconductor and the infill can be independently optimized for the application.

An important material class in this respect comprises liquid crystals (LCs), in particular the nematic type, because these materials exhibit large birefringence; $\Delta n = 0.2-0.3$ for a conventional LC like 5CB, but much higher values are also reported [144]. Since the first proposal of Busch and John [145], several tuning proposals and experiments and have been reported with 1D [146, 147, 148, 149],
2D [109, 150, 151, 152, 153, 154], 3D (see ref. 155 for an overview) and planar [71, 72, 73, 74, 75, 76] LC-infiltrated PhCs. Also, device proposals based on LC tuning have appeared in the literature [156, 157, 158, 159, 160], although the only experimental demonstration so far is the PhC laser of refs. [72, 73]. The tuning speed of LCs is limited to ms, down to µs for certain ferroelectric or polymer-dispersed LCs [161], because it relies on the physical reordering of molecules. This is sufficient for many applications, but higher speeds are required for e.g. signal modulation in the backbone of optical networks. Furthermore, the liquid state could prevent certain applications or limit the compatibility with other processing steps. Therefore also other materials, notably electro-optically active or optically non-linear (NLO) polymers must also be considered as tunable low-index material.

The possible tuning speed of NLO polymers is intrinsically high (< ps) [162]. The achievable $\Delta n$ is rather low however, so that cavities with considerable Q are probably required [149]. In a recent proposal, the Kerr nonlinearity of a polymer infiltrated in a 1D PhC was used to realize a Q-switched laser [163]. Also, the filling of 2D PhC lasers with photoaddressable polymer has been proposed [73]. Apart from the active application, also the passive effect of the refractive index change has many applications. In contrast to membrane type structures [72], the infiltration of deeply etched photonic crystals is expected to increase the vertical confinement due to improved index guiding, which was used to decrease the diffraction loss of a 1D PhC laser [164]. Furthermore, it was recently shown that selective filling opens up a new way to create components like single mode waveguides, waveguide bends, crossings and splitters from “bulk” 2D PhCs [165, 166]. Although the latter two applications are also possible with LC filling, the solid polymer offers better mechanical stability of the devices. Also, after planarization of infiltrated structures, further polymer processing may be used to create extra functionality above the PhC.

### 5.1 Infiltration parameters

A schematic cross-section of a fluid column in a cylinder with submicron diameter is shown in Figure 5-1. For these dimensions the capillary action is dominant and the gravitational force is negligible [167]. The capillary pressure $p_{\text{cap}}$ thus provides the driving force for the liquid flow and is given by:
Here \( r \) is the hole radius, \( \gamma \) the surface tension of the liquid and \( \theta \) the contact angle between the liquid and the solid. For successful infiltration it is crucial that the liquid wets the surface, i.e. \( \theta \ll 90^\circ \). The typical surface tension for most liquids is in the order of 10-100 mN/m (see [100], table 6-150), implying that the capillary pressure is in the range 2 to 20 bar for cylinders with a diameter of ~200 nm. The surface tension of TMP-3A is 36.1 mN/m and that of 5CB is comparable. If the infiltration is performed under a pressure \( p_0 \), a residual volume \( (p_0/p_{\text{cap}})V_{\text{hole}} \) of compressed air would remain unfilled, provided that the air cannot escape. This is a substantial fraction of the total hole volume \( V_{\text{hole}} \) for filling at ambient pressure \( p_0 = 1 \) bar. In previous work [71, 74, 75, 76] infiltration of deeply etched PPhCs was performed under vacuum conditions, which means that \( p_0 \ll 1 \) bar.

The air permeability of most liquids would be sufficient for the small holes to
degas efficiently under a pressure up to several bars. The degas time can be estimated from the volume gas flow given by [168]:

\[ \Phi_{\text{gas}} = P \frac{A \Delta p}{h}, \]

where \( P \) is the gas permeability of the liquid, \( \Delta p \) the pressure drop across the liquid column, \( A \) its cross-sectional area and \( h \) the hole depth. The degassing time \( \tau_{\text{degas}} \) can then be estimated from \( \tau_{\text{degas}} \approx V_{\text{hole}}/\Phi_{\text{gas}} \) with \( \Delta p = p_{\text{cap}} \). The exact value of \( P \) for the infiltrants used is not known, but the possible range may be obtained using values for the N\(_2\)-permeability of polyethylene (PE) [168] and water ([100], tables 6-201 and 8-88). This yields hole degassing times in the order of 1 s for 3 \( \mu \)m deep holes for the PE permeability and even two orders of magnitude shorter times when using the water permeability value. It is therefore concluded that the residual air at the bottom of the hole diffuses through the liquid well within the timescale of the infiltration procedure. This result indicates that vacuum conditions are not required for successful infiltration.

A typical time scale for the infiltration can be estimated from the Hagen-Poiseuille flow law for the liquid flowing into the hole, yielding

\[ \tau_{\text{fill}} = \frac{4 \mu h^2}{r \gamma \cos(\theta)}. \]

Here \( \mu \) is the viscosity of the liquid, and the pressure gradient is estimated by \( p_{\text{cap}}/h \). The liquid monomer used in our experimental work has a viscosity \( \sim 150 \) mPa\( \cdot \)s so that the infiltration time for holes of \( \sim 3 \) \( \mu \)m deep would be \( \sim 1 \) ms. Even very viscous liquids (e.g. glycerine with \( \mu \approx 1 \) Pa\( \cdot \)s) yield fill times < 1 s, short compared to the time scale of the infiltration. From this it follows that viscosity should not be critical for the infiltration.

### 5.2 Polymer filling by in situ polymerization

In this section the photonic band edge shift by filling the air holes with a polymer is demonstrated. A simple filling procedure is described, consisting of infiltration with liquid TMP-3A at room temperature and ambient atmosphere
followed by thermal polymerization\(^1\). The solid state of the infill allows for direct inspection of the infiltrated holes by cross-sectional scanning electron microscopy (SEM). The effect of the filling on the photonic band gap was investigated by optical transmission measurements.

It is explained in the previous section, that good wetting is crucial for successful infiltration. In [169] the wetting of GaAs by a LC was improved with an organic self assembled monolayer on the substrate. Here chemical treatment of the InP surface with acidic fluids is used to improve the wetting. Various chemical treatments of ICP-etched InP substrates were examined with contact angle measurements. This is done by inspecting a droplet of TMP-3A on the sample surface with a microscope; see the inset in Figure 5-2. A summary of the results of one set of measurements is given in Figure 5-2. All treatments improve the wetting compared to the untreated surface, which points to the role of removal of the native oxide layer. The best result was obtained after a rinse with 10% hydrofluoric acid in water, which reduced the contact angle from 33° to 7°. To protect the surface from oxidation during transport to the infiltration set-up, the sample is immersed in 2-propanol. After removal of the sample from the 2-

\(^1\) These experiments were performed in the laboratory of Polymer Technology, department of Chemical Engineering, TU/e, in collaboration with B. K. C. Kjellander.
propanol and blow-dry of the surface, the measured contact angle is still 11°. The wetting depends on the exact chemical state of the surface and the ambient atmosphere. This causes a considerable spread in the measured contact angles over different sets of experiments. In a separate set, even a contact angle of 3° was measured after surface treatment with HF and 2-propanol.

After the surface preparation, a droplet of the liquid monomer is deposited onto the sample to cover the hole pattern, either in vacuum (20 mbar) or ambient atmosphere, at room temperature. The thermal polymerization is performed on a hotplate under N₂ flow, to minimize oxygen reaction with the radicals formed. To suppress formation of cracks in the polymer layer, the temperature is gradually increased from room temperature up to 50 °C, where polymerization is allowed for 10 minutes. The temperature is further increased to 70 °C in order to increase the mobility of the unreacted monomers and the sample was baked for 20 minutes at this temperature to complete the polymerization. The refractive index of the polymer was measured separately by spectroscopic ellipsometry on a layer that was spin-cast on a Si-substrate and subsequently polymerized. For wavelengths around 1550 nm the measured refractive index is 1.465 ± 0.005; the absorption in this wavelength region is negligible for our purpose.
The filled hole pattern is inspected with cross-sectional SEM. In Figure 5-3, a SEM view of the holes is shown after infiltration in a) vacuum (20 mbar) and b) ambient atmosphere. Nearly complete infiltration is apparent for both conditions. The detachment of the polymer plug from the sidewall, visible for the center holes in Figure 5-3a, is attributed to the polymerization shrinkage, which is expected to be 10-15% for poly-acrylates. Some holes in Figure 5-3a appear to be unfilled. It was observed that the polymer plugs are not cleaved, but remain on either side of the cleavage. This is illustrated in Figure 5-4. The experiments were carried out on etch test samples with varying hole shapes. In all cases investigated it was observed that the polymer fills the holes down to the bottom, even for the irregular and conical hole shapes as in Figure 5-3.

Optical characterization of polymer filled PhCs was performed with sample B.

Figure 5-4: a) The top image is a composite of various SEM images along the cross-section of a filled PhC (infiltration in vacuum). The bottom is the mirror image of a similar composition for the other side of the cleavage. The corresponding holes are connected with lines and it is clearly visible that each empty hole has a filled counterpart. The polymer plug is stuck at the side where the largest part of the hole is situated, as illustrated in b).
After measurement of the empty structure, the holes were infiltrated with TMP-3A under ambient conditions. A thick polymer droplet remained on the surface after polymerization, which is shown in the SEM overview of Figure 5-5a. It is visible that the droplet is bounded by the ridges at both sides of the patterned area. Sharp edges are natural borders for a liquid spreading, because the contact angle is $> 90^\circ$ with respect to the perpendicular surface [170]. For this reason also, the monomer does not flow over the edges over the sample which ensures that the facets are clean. An illustration of this was already shown in Figure 2-13.

The spectrum of sample B after filling is normalized in the same way as the spectrum before filling (see section 4.3.2). The normalized transmission spectra for the empty and filled case are compared in Figure 5-5b. Upon filling, the air

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**Figure 5-5:** a) SEM view of the sample with the large polymer droplet on top of the PhCs. b) Measured TE, $\Gamma K$ transmission spectrum of sample B before and after polymer filling. The fit to the air band after filling is also included, with $r/a = 0.295$, $\varepsilon'' = 0.24$ and $n_{\text{hole}} = 1.372$. 
band edge exhibits a redshift of one third of the stop gap width, which is the optical evidence for hole filling under ambient conditions. No significant shift is observed for the dielectric band edge, because the electric field of the Bloch mode is located primarily in the semiconductor material \[75, 150\]. It was discussed in the previous chapter, that the \( r/a \) value of sample B is rather constant over the lithographic tuning range and it is therefore assumed to be 0.295 also for the air band edge of the filled sample. The filling efficiency \( \eta \) (volume of polymer plug divided by hole volume) is determined by fitting the air band edge to simulations with fit parameters \( \varepsilon' \) and \( n_{\text{hole}} \), the latter being the average refractive index inside the hole. The filling efficiency is then calculated via \( \eta = (n_{\text{hole}} - 1)/(n_{\text{polymer}} - 1) \), where \( n_{\text{polymer}} \) is the refractive index of the polymer. Holes with incomplete filling are thus approximated in the simulation by a uniform material with an average refractive index \( n_{\text{hole}} = 1 + \eta(n_{\text{polymer}} - 1) \). It has been verified with band structure calculations for a more realistic distribution of the refractive index, i.e. a polymer cylinder in the hole centre surrounded by a sheet of air, that this approximation is indeed valid here. The polymer filling reduces the average refraction angle at the semiconductor-hole interface (see Figure 4-15) and thus also the out-of-plane losses. This effect is not taken into account in the 2D simulation implicitly, so that the value of \( \varepsilon' \) for the empty structure is no longer valid for the filled. A good fit to the measurements is obtained with \( \varepsilon' = 0.24 \pm 0.05 \) and \( n_{\text{hole}} = 1.37 \pm 0.05 \), leading to a filling efficiency of 0.8 \pm 0.1. This number is consistent with complete infiltration of the holes followed by polymerization shrinkage of 10-15%. The filling yields an important decrease of the loss parameter, from to 0.35 to 0.24, which leads to improvement of the air band transmission with a factor of > 2.

5.3 Polymer filling by solvent evaporation

Polymer e-beam resists (EBRs) as PMMA and ZEP are usually deposited onto a substrate by spincoating from solution. A solid polymer layer is then obtained by thermal evaporation of the solvent. In this section, a first experiment to fill the PPhC holes with EBR from solution is described. This would create interesting possibilities, because it can be expected that removal of the infill from selected individual holes is possible with a subsequent EBL step. Such a process could be used to fabricate devices as described in refs. [34] and [165] with the EBR fill itself, or using the structure as a template for further infiltration steps as in section 5.2. Repetition of the latter procedure could make the filling of adjacent holes with different materials possible, to create for instance the optimized waveguide crossing design of ref. [34]. If appropriate holes are filled with NLO polymer, the
devices are tunable, with a large design freedom in tuning characteristics.

The experiment is performed with an InP test sample with a SiNₓ residue on top and a solution of 7 % PMMA 495K in Anisole. No surface treatment was performed prior to the infiltration, but good wetting of the InP surface by the liquid was apparent. A large droplet is deposited onto the sample, covering the PhCs. Subsequently, the sample was rotated at 3600 rpm to obtain a uniform film on the surface. The sample was baked on a hotplate at 200° for 2 min to evaporate the solvent, which is the standard procedure in EBL processing.

Cross-sectional SEM views of various infiltrated structures on the sample are presented in Figure 5-6a-d. Severe shrinkage is apparent in Figure 5-6a, due to the small volume fraction of polymer in the solution, yielding very incomplete filling of the 1 µm wide trenches. Figure 5-6b-d show holes with r = 120, 240 and
400 nm respectively. These all have in common that the polymer forms a sheet on the sidewall, probably due to the higher capillary force compared to the trenches. Although very incomplete filling is apparent for the holes, the polymer does extend down to the bottom, indicating full infiltration of the solution before solvent evaporation. Nearly complete filling can be obtained by multiple repetition of the procedure [171], which requires opening of the holes at the top between each step. This can be done by controlled etching of the polymer sheet.

The configuration in Figure 5-6c and d, a thin PMMA sheet covering the holes, allows opening of selected holes with an EBL step, using predefined markers in the vicinity of the PhC structure. This allows for subsequent selective infiltration, for instance using the method developed in section 5.2. It is possible that the PMMA inside the hole is not completely removed after EBL. For many applications this may not be an issue, because of the limited volume of the remaining PMMA. If complete removal is required, this can be done with an isotropic etch in oxygen plasma after EBL; the polymer layer on top is thick enough to withstand this.

5.4 Liquid Crystal infiltration

In this section the infiltration experiments with 5CB are described, which are performed with sample C\(^1\). The infiltration procedure is equivalent to that of section 5.2. The temperature of the sample during infiltration was ~23 °C with the LC just in the nematic phase. The measured contact angle for 5CB on InP substrate after treatment with HF and 2-propanol is 14°, so complete infiltration of the holes can also be expected for this material. After optical measurement of the empty sample, which was discussed in section 4.3.3, the sample was treated with HF and 2-propanol and infiltrated with 5CB at ambient conditions. A macroscopic droplet remained on the surface after infiltration. To check if the holes are indeed fully filled, the transmission spectrum is measured for sample temperature \(T > T_c\), because the average refractive index of the LC is known for this case. For this purpose, the sample is placed on a special optical sample holder with a resistive heater soldered into it. The temperature of the sample holder is determined by the applied current through the resistor and stabilizes in ~1 hour. A thermocouple is attached to the sample holder to measure the temperature.

\(^1\) These experiments were performed in the laboratory of Polymer Technology, department of Chemical Engineering, TU/e, in collaboration with B. K. C. Kjellander.
The TE, ΓK transmission spectrum is shown in Figure 5-7 for the empty crystal (at \( T = 23 \) °C) and the filled crystal at \( T = 48 \) °C, so with the LC in the isotropic phase. An air band shift of one third of the stop gap width and significant increase of the transmission level can be observed in the figure. The small shift of the dielectric band is caused by the temperature dependence of \( n_{\text{eff}} \)\(^{143}\). Using the same method as in section 5.2, \( \varepsilon'' \) and \( n_{\text{hole}} \) were used as free parameters to fit the measurement after filling and \( r/a \) is assumed equal to the measured value for the empty crystal, i.e. 0.235. The temperature dependence of the semiconductor material leads to an increase in \( n_{\text{eff}} \) of \( 2 \times 10^{-4} \) °C\(^{-1}\)\(^{143}\). This is accounted for by using \( n_{\text{eff}} = 3.255 \) instead of 3.25 in the calculations for \( T = 48 \) °C. A good fit was obtained with \( \varepsilon'' = 0.05 \) and \( n_{\text{hole}} = 1.41 \pm 0.03 \), where \( n_{\text{hole}} = n_i = 1.575 \) is expected for completely filled holes. Following the discussion in section 5.2, a filling efficiency of \( \eta = 0.71 \pm 0.05 \) is deduced from the measured \( n_{\text{hole}} \). The same procedure is followed for the TE, ΓM case, which yields \( \eta = 0.77 \pm 0.05 \). It was verified that the spectrum, and thus \( \eta \), is not changed after heating to above \( T_c \) and cooling down to room temperature.

The apparent incomplete filling with LC is not expected from the infiltration model of section 5.1 and from the efficiency found for polymer filling, which includes significant polymerization shrinkage. In refs. [71, 74, 75, 76], the experimentally obtained optical properties of PPhCs infiltrated with LC were also
explained by incomplete hole filling, although a significantly larger efficiency of $\eta \approx 0.9$ was found. In these previous works the infiltration was performed under vacuum conditions while heating the LC well into the isotropic phase. It could therefore be speculated, that the reduced $\eta$ is caused by formation of gas bubbles in the LC during infiltration. This effect would be more severe in the present work, because the infiltration is performed at ambient pressure. In fact it was suggested in ref. [75] that it is important to boil-out gas trapped in the LC before infiltration by heating and pumping the vacuum chamber. The cause of the incomplete filling is still an open issue, but following ref. [76], it will be assumed that $\eta = 0.74 \pm 0.05$ in the further analysis.

For application of the LC infiltrated PPhC in tunable devices, it is important to know the director field configuration below $T_c$, because this determines the position dependent dielectric tensor of the anisotropic LC. The average refractive index of the LC experienced by the PhC mode, $n_{LC}$, is determined by this dielectric tensor weighted by the electric field distribution of the light. The temperature dependence of $n_{LC} (= \eta^{-1} \cdot n_{hole})$ can be determined from measurement of the temperature dependent optical properties of the filled PhC and yields information on the director field [71, 74, 76, 150, 151]. Figure 5-8 shows preliminary data of the TE, $\Gamma M$ air band ($a = 337$ nm) after filling for sample temperatures of 23, 30 and 48 °C. For the first two cases, $T < T_c$, so the LC is in the nematic phase. No significant shift of the air band edge is observed when

\begin{figure}
\centering
\includegraphics[width=\textwidth]{figure5-8.png}
\caption{Measured TE, $\Gamma M$ air band edge ($a = 337$ nm) for the PPhC filled with 5CB at sample temperatures of 23, 30 and 48 °C.}
\end{figure}
heating the sample from $T = 23 \, ^\circ C$, which is just above $T_{nc}$, to $T = 30 \, ^\circ C$. This indicates that the order parameter is constant at least in a certain temperature range above $T_{nc}$. A similar behavior was shown for the eutectic LC mixture E7 infiltrated in macroporous silicon in ref. [150]. When the temperature is further increased to 48 $^\circ C$, so well above $T_c$, the air band exhibits a redshift of $\Delta a/\lambda = 0.001$. It is calculated that part of this shift, $\Delta a/\lambda = 3 \times 10^{-4}$, is induced by the increase in $n_{eff}$. The change in $n_{LC}$ due to the nematic-to-isotropic phase transition therefore contributes to the redshift with an amount of $\Delta a/\lambda = 7 \times 10^{-4}$. The direction of the shift indicates that $n_{LC} < n_i$ when the LC is in the nematic phase. This would rule out the PP configuration of the director field, see Figure 2-18, because for this configuration and TE polarized light $n_{LC}$ is expected to be approximately the in-plane average $1/2(n_o^2 + n_e^2)^{1/2}$ (1.601 for 5CB), i.e. larger than $n_i$.

Fitting of the air band for $T < T_c$ yields $n_{hole} = 1.37 \pm 0.03$ for $\Gamma K$ and $n_{hole} = 1.41 \pm 0.03$ for $\Gamma M$ for the LC in the nematic phase. Within the experimental uncertainties, these values are the same as found for the LC in the isotropic phase. Also, the temperature dependent measurements have not been done in sufficient detail to follow the exact temperature dependence of $n_{hole}$. Therefore, no quantitative conclusions can be drawn on the absolute values of $n_{LC}$ that are deduced from $n_{hole}$. Keeping this in mind, $n_{LC}$ is determined to be $1.50 \pm 0.04$ for $\Gamma K$- and $1.55 \pm 0.04$ for $\Gamma M$-structures. These values are close to $n_o$, which would indicate that the mesogens are oriented primarily perpendicular to the plane. For AX director field and TE polarized light, $n_{LC}$ is simply $n_o$, but for ER configuration $n_{LC}$ is a complex average of the position dependent dielectric tensor. It was discussed in ref. [150], that the electric field of the air band PhC mode is located primarily in the center of the holes, where the mesogens are aligned along the hole axis for both AX and ER configuration. Therefore, it is in general not straightforward to distinguish between the two director fields with this type of measurements. In the present work in particular, the experimental uncertainties are too large for this purpose. A larger $r/a$ would lead to larger shifts and a smaller error in $n_{LC}$. It is expected that additional information can be obtained from measurements with TM polarized light [76].

Although the exact director field is not known, the thermal tuning range for this PPhC infiltrated with 5CB is directly visible in Figure 5-8. The total corresponding wavelength shift is 7 nm, of which 5 nm due to the tuning of $n_{LC}$. The total band edge shift leads to a factor of $\sim 10$ difference in transmission level
due to the exponential decrease at the air band edge. This is achieved by heating the sample from 30 °C to above 48 °C so a temperature change of 18 °C would be enough for 10 dB extinction of an optical signal. The thermal tuning range of the LC may be enhanced by increasing $r/a$ or using LCs with larger birefringence.

### 5.5 Conclusion

InP based deeply etched PPhCs have been filled with both polymer and liquid crystal. An important step in the filling procedure is infiltration of the holes with a liquid, either the monomer as precursor for the polymer or the LC itself. It was estimated that the infiltration time is $\ll 1$ s. The entrapped air at the bottom of the hole is estimated to diffuse out through the liquid on a timescale $< 1$ s. From these estimations it is expected that the holes are fully infiltrated even at ambient conditions.

Polymer filling is obtained by thermal polymerization of the infiltrated monomer, trimethylolpropane triacrylate. The filled PhCs were inspected with SEM and complete filling down to the bottom of the hole was observed, both for samples infiltrated in vacuum and in ambient atmosphere. With optical transmission measurements it was observed that the polymer filling induces a shift of the air band edge of one third of the stop gap width. From the fit of 2D FDTD simulations a filling efficiency of $0.8 \pm 0.1$ was deduced, proving nearly complete filling. The deviation from complete filling is caused by polymerization shrinkage, which is typically 10-15 % for polyacrylates.

Filling with polymer e-beam resist was investigated with a first experiment by infiltration from solution and subsequent evaporation of the solvent. The low fraction of polymer in solution leads to very incomplete filling of the holes, order of 10 %. It was observed with SEM that the polymer is situated on the sidewall and that a sheet with a thickness of a few hundred nm covers the PhC. This may open the way to filling of selected holes by opening the sheet in defined areas with an EBL step and subsequent filling of the opened hole(s).

The PPhCs filled with the LC 4-pentyl-4-cyanobiphenyl were investigated with optical transmission measurements. The air band shift due to the filling is again one third of the stop gap width. The 2D FDTD simulations fit to the measurement with the LC in the isotropic phase, yield a filling efficiency of $0.74 \pm 0.05$. This incomplete filling is not expected and requires further investigation. In the preliminary temperature dependent measurements
performed, the experimental uncertainties are too large to conclude on the director field configuration of the LC inside the holes. However, the experiments do show thermal shift of the air band of $\Delta \lambda = 7$ nm, which is partly caused by the semiconductor (~2 nm) and partly by the LC (~5 nm).
Summary

The subject of this thesis is deeply etched planar photonic crystals (PPhCs) in InP material for eventual application in photonic integrated circuits. The PPhCs consist of a two-dimensional periodic pattern of holes etched through a standard InP/InGaAsP/InP planar waveguide layer stack. The periodic refractive index acts on light waves in a similar way as the periodic potential in a solid crystal acts on electron waves, yielding photonic band gaps. PPhCs with a photonic band gap centered at the telecom wavelength of 1550 nm are investigated in this thesis, which implies that the air holes have a diameter $d$ of ~200 nm and are arranged in a (hexagonal) lattice with a lattice constant $a$ of ~400 nm.

A Cl$_2$-based inductively coupled plasma etching process is developed for fabrication of the PPhCs, using a SiN$_x$ layer patterned with e-beam lithography and subsequent reactive ion etching as a mask. For low optical loss it is important that the etched holes exhibit vertical and straight sidewalls to a depth of at least 2 µm. The concept of sidewall passivation is exploited to promote this verticality. For this purpose, relatively small amounts of O$_2$ or N$_2$ are added to the Cl$_2$ flow into the plasma. With a proper Cl$_2$/O$_2$ flow ratio, here $\sim$14/3, good quality holes are obtained for a significantly larger window in parameter space, notably concerning sample temperature, than for Cl$_2$-only chemistry. At this Cl$_2$/O$_2$ flow ratio, the temperature dependence of the large area etch rate is determined by the surface chemistry of the InP-Cl$_2$ system. The sample temperature should therefore be raised sufficiently (typically $>200$ °C) to reach the regime where the InCl$_3$ corrosion layer formed evaporates from the etched surface. To improve the stability of the sample temperature, the heating by the ion bombardment must be compensated by cooling the carrier wafer with a He-gas backflow. With an optimized process, high aspect ratio holes with $d = 200$ nm are etched in 1 minute to a depth $>3.5$ µm with vertical sidewalls in the upper 2 µm.

Optical characterization of the fabricated PPhCs is performed with an end-fire technique. For this purpose, the hole-patterns are monolithically integrated with ridge waveguides in a transmission configuration and the samples are cleaved to obtain end facets. Light from a polarization controlled tunable diode laser (wavelength range 1470-1570 nm) is coupled into and out of the waveguides with microscope objectives. Transmission spectra were obtained on a normalized frequency scale by measurement of PPhCs with various $a$ on the same chip. Photonic band gaps are clearly observed, with an extinction of $>30$ dB compared
to the transmission bands. It is observed that significant transmission is still possible when the forward direction is forbidden (i.e. the frequency of the incoming light is in the stop gap), probably by continuous diffraction into directions that are allowed. The measurements are compared to two-dimensional finite difference time domain calculations, in which the out-of-plane losses are simulated by an artificial absorption in the holes. The imaginary component of the dielectric constant that is related to this absorption, referred to as loss parameter $\varepsilon''$, and the ratio of hole radius over lattice constant $r/a$ are used as free parameters to fit the calculated spectra to the measured spectra. The fitted loss parameter is a figure of merit for the quality of the etched holes. Hole qualities resulting from four different etch processes are evaluated and loss parameters ranging from 0.7 down to 0.05 are obtained. An important conclusion is that a significant sidewall angle in the core of the planar waveguide does not lead to excessive losses, as long as the sidewalls are vertical in the claddings. Analysis of the fitted $r/a$ values shows that the patterning process is reproducible.

The application area of PPhC structures will be greatly enhanced when their optical properties are tunable. An attractive possibility to achieve the desired tuning is filling of the air holes with a material that has a tunable refractive index. Solid polymer fills are obtained by infiltration with a liquid monomer and subsequent in situ thermal polymerization. Infiltration of PPhCs with a liquid relies on the capillary forces in the holes. Complete filling is obtained even in ambient atmosphere, as long as the wetting of the InP by the liquid is good. Residual air under the fluid column disappears from the holes by diffusion through the liquid. It was observed with scanning electron microscopy that all holes are filled down the bottom, for infiltration both under vacuum conditions and in ambient atmosphere. Upon filling of the PPhC with a polymer, the measured air band edge exhibits a redshift, yielding a 30% decrease of the stop gap. The filling degree is determined from fitting two-dimensional finite difference time domain calculations to the measured spectrum, using the refractive index in the hole and $\varepsilon''$ as free parameters. From this analysis, the filling degree is determined to be approximately 0.8, which is consistent with complete infiltration by the monomer followed by the expected 10-15% polymerization shrinkage. Preliminary tuning experiments were performed with PPhCs infiltrated with liquid crystal. The sample was heated to above the nematic-to-isotropic phase transition temperature of the liquid crystal. This caused a wavelength shift of the photonic band edge with a magnitude of 7 nm, which is partly caused by refractive index change of the semiconductor (~2 nm) and partly by that of the liquid crystal (~5 nm).
Dit proefschrift gaat over diep geëtste planaire fotonische kristallen (PFKs) in InP materiaal voor uiteindelijke toepassing in fotonische geïntegreerde circuits. De PFKs bestaan uit een tweedimensiona al gatenpatroon, geëtst in een standaard InP/InGaAsP/InP planaire golfgeleider. Door de periodieke brekingsindex bezitten deze kristallen een zogenaamde fotonische ‘band gap’, analoog aan het bestaan van een elektronische ‘band gap’ in halfgeleiders. In dit proefschrift zijn PFKs onderzocht met een fotonische ‘band gap’ gecentreerd rond de telecom golflengte van 1550 nm, wat betekent dat de gaten een diameter $d$ van ~200 nm hebben en zijn geordend in een (hexagonaal) rooster met een roosterconstante $a$ van ~400 nm.

Een inductief gekoppeld plasma etsproces met reactief Cl$_2$ is ontwikkeld voor fabricage van de PFKs. Een SiN$_x$-laag, gestructureerd met elektronstraal lithografie en daaropvolgend reactief ion etsen, is hierbij gebruikt als masker. Lage optische verliezen zijn alleen haalbaar als de zijwanden van de gaten recht en vertikaal zijn tot een diepte van tenminste 2 $\mu$m. Zijwandpassivatie is toegepast voor het bevorderen van de verticaliteit van de gaten. Voor dit doel zijn relatief kleine hoeveelheden O$_2$ of N$_2$ toegevoegd aan het Cl$_2$-debiet in het plasma. Met de juiste Cl$_2$/O$_2$-debietverhouding, hier ~14/3, is de kwaliteit van de geëtste gaten goed in een groter deel van de ets parameterruimte, in het bijzonder wat betreft de temperatuur van het sample, vergeleken met puur Cl$_2$ chemie. Voor deze Cl$_2$/O$_2$-debietverhouding is de temperatuursafhankelijkheid van de etssnelheid van grote vlakken bepaald door de oppervlaktechemie van het InP-Cl$_2$ systeem. De temperatuur van het sample moet daarom voldoende hoog zijn (typisch $> 200$ °C) om de gevormde InCl$_3$ corrosielag te laten verdampen van het oppervlak. Om de stabiliteit van de temperatuur van het sample te verbeteren moet de verhitting door het ionenbombarde ment gecompenseerd worden door koeling van de sampledrager met He-gas aan de achterzijde. Met een geoptimaliseerd proces zijn gaten met $d = 200$ nm geëtst tot een diepte van 3.5 $\mu$m, met verticale zijwanden in de bovenste 2 $\mu$m.

Optische karakterisatie van de vervaardigde PFKs is gedaan met een zogenaamde ‘end-fire’ techniek. Voor dit doel zijn de gatpatronen monolithisch geïntegreerd met golfgeleiders in een transmissie configuratie en zijn de samples gekliefd voor het verkrijgen van eindfacetten. Licht van een verstembare diode laser (golflengtebereik 1470-1570 nm) wordt in- en uit de golfgeleiders
gekoppeld met microscoopobjectieven. Transmissiespectra zijn verkregen op een
genormeerde frequentieschaal door meting van PFKs met verschillende \( a \) op
dezelfde chip. Fotonische ‘stop gaps’ zijn duidelijk zichtbaar, met een extinctie
van > 30 dB vergeleken met de transmissiebanden. Significante transmissie is
waargenomen voor de situatie waarbij de voorwaartse richting verboden is,
oftewel in de ‘stop gap’ voor het inkomende licht. Dit is mogelijk, waarschijnlijk
door continue diffractie in richtingen die wel zijn toegestaan. De metingen zijn
vergeleken met tweedimensionale ‘finite difference time domain’ berekeningen,
waarin de verliezen uit het vlak gesimuleerd zijn door een kunstmatige absorptie
in de gaten. De imaginaire component van de diëlectrische constante die
gerelateerd is aan deze absorptie, de zogenoemde verliesparameter \( \varepsilon'' \), en de
verhouding van de gatstraal tot de roosterconstante \( r/a \) zijn hierbij gebruikt als
vrije parameters om de berekeningen overeen te laten komen met de
实验en. De aldus verkregen verliesparameter is een maat voor de kwaliteit
van de gaten. Gatkwaliteiten resulterend van vier verschillende etsprocessen zijn
geëvalueerd en verliesparameters variërend van 0.7 tot 0.05 zijn gevonden. Een
belangrijke conclusie is dat een significante zijwandhoek in de kern van de
planaire golfgolfrichter niet leidt tot excessieve verliezen, zo lang de zijwanden
verticaal zijn in de bekleding. Uit de analyse van de gevonden \( r/a \) waarden volgt
dat het proces van de patroongeneratie reproduceerbaar is.

Verstembaarheid van de optische eigenschappen van PFKs zal het
toepassingsgebied van deze structuren aanzienlijk verruimen. Een aantrekkelijke
mogelijkheid om de gewenste verstemming te bereiken is het vullen van de gaten
met een materiaal dat een verstembare brekingsindex heeft. Vaste
polymeervulling is verkregen door infiltratie met een vloeibare monomeer
gevolgd door \textit{in situ} thermische polymerisatie. De drijvende kracht voor infiltratie
van PFKs is de capillaire werking van de vloeistof in de gaten. Complete vulling
is verkregen zelfs in omgevingsatmosfeer, zolang de benatting van het InP door
deur de vloeistof goed was. De overblijvende lucht onder de vloeistofkolom verdwijnt
via diffusie door de vloeistof. Met elektronenmicroscopie is waargenomen dat de
gaten tot op de bodem gevuld zijn, zowel onder vacuümomstandigheden als in
omgevingsatmosfeer. Door de polymeervulling ondergaat de gemeten flank van
de luchtband een roodverschuiving, die resulteert in een 30 % afname van de
‘stop gap’. De vulgraad is bepaald aan de hand van het aanpassen van
tweedimensionale ‘finite difference time domain’ berekeningen aan de metingen,
waarbij de brekingsindex in het gat en \( \varepsilon'' \) zijn gebruikt als vrije parameters. Uit
dezelfde analyse volgt een vulgraad van ongeveer 0.8, wat consistent is met volledige
infiltratie door de monomeer gevolgd door de verwachte 10-15 %
polymerisatiekrimp. PFKs geïnfiltreerd met vloeibaar kristal zijn gebruikt voor eerste verstemmings experimenten. Het sample is verhit tot boven de nematisch-naar-isotroop fase overgangstemperatuur van het vloeibaar kristal. Dit veroorzaakte een golflengteverschuiving van de fotonische bandflank ter grootte van 7 nm, deels veroorzaakt door de verandering in brekingsindex van de halfgeleider (~2 nm) en deels door die van het vloeibaar kristal (~5 nm).
List of publications

Journal papers


Publication of the results in chapter 4 is in preparation.

Conference papers


Dankwoord

Het promotieonderzoek en in het bijzonder het schrijven van een proefschrift is een grote inspanning. Een inspanning die ik zonder de, al dan niet expliciete, hulp van anderen nooit op deze manier had kunnen doen. Daarbij horen zeker ook de mensen van wie ik soms cruciale inzichten heb verworven via publicaties of discussies en de mensen in hun omgeving die dat weer mogelijk hebben gemaakt. De ruimte hier is te beperkt om al deze mensen met name te bedanken, maar ik wilde ze niet onvernoemd laten. Wel is er hier ruimte om de mensen in mijn omgeving te bedanken die veel voor mij en mijn promotieonderzoek betekend hebben. Ik heb sterk de behoefte om deze mensen in de schijnwerpers te zetten; daarom is het schrijven van een dankwoord ook zo leuk.

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Curriculum Vitae

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