UNIVERSIDAD POLITÉCNICA DE MADRID

ESCUELA TÉCNICA SUPERIOR
DE INGENIEROS DE TELECOMUNICACIÓN

TESIS DOCTORAL / PH.D. THESIS

Tunable organic waveguides and microstructured devices

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M.Sc. Physics

M.Eng. in Electronics and Automated Systems

M.Sc in Physics of Condensed Matter and Biological System

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CENTRO DE MATERIALES Y DISPOSITIVOS AVANZADOS PARA LAS TIC

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ACRONYMS

ABS	Acrylonitrile butadiene styrene
AFM	Atomic Force Microscopy
BP	Blue Phases
CCD	Charge-coupled device
CP	Cyclopentanone
FDTD	Finite difference time domain
FDTS	Perfluorodecyltrichlorosilane
FFT	Fast Fourier transform
IFFT	Inverse fast Fourier transform
ITO	Indium tin oxide
LC	Liquid crystal
LDW	Laser direct write
LED	Light emitted diode
MFD	Mode field diameter
MLCL	Modal Liquid Crystal Lenses
MMI	Multi-mode interference
MZI	Mach-Zehnder interferometer
NIL	Nanoimprint Lithography
NOA	Norland Optical Adhesive
OAM	Orbital angular momentum

ACRONYMS

OVRP	Optical vortex retarder plate
P-NIL	Photo Nanoimprint Lithography
PDMS	Polydimethylsiloxane
PEDOT:PSS	Poly(3,4-ethylenedioxythiophene) poly(styrene sulfonate)
PGMEA	Propylene glycol monomethyl
PIC	Photonic integrated circuit
PLA	Poly(lactic acid) or polylactic acid or polylactide
PMMA	Poly(methyl methacrylate)
PVA	Polyvinyl alcohol
PVD	Physical vapor deposition
PWM	Pulsed width modulation
ROI	Region of interest
SDL	Spiral diffractive lenses
SLM	Spatial light modulator
SOP	State of polarization
SPP	Spiral phase plates
T-NIL	Thermoplastic nanoimprint lithography
TIR	Total internal reflection
UV	Ultra violet
UVO	Ultra violet oven

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RESUMEN

Introducción

El presente trabajo de Tesis se ha centrado en el desarrollo de una nueva línea de investigación en el Centro de Materiales y Dispositivos Avanzados para TIC (CEMDATIC) basada en el diseño, fabricación y caracterización de chip fotónicos integrados (PICs) de sus siglas en inglés. También se ha continuado con líneas de investigación existentes, como los dispositivos fotónicos de fase, desarrollando nuevos dispositivos y tecnologías que, con pequeñas modificaciones, puedan ser empleados en los PICs.

Durante la realización de la Tesis, se han modificado dos máquinas aportando sustanciales mejoras y se ha desarrollado un nuevo sistema de litografía por nano-impresión. Asimismo, se ha realizado un montaje óptico con acoplo de luz con precisión nanométrica para la caracterización de los chips, desarrollando además los módulos de control electrónico necesarios.

A continuación se enumeran **métodos** desarrollados para la fabricación:

- **Sistema de ablación láser.** El equipo consta de un láser pulsado a 50 kHz de Nd:YAG triplicado (355 nm) con 300 mW a la salida, un sistema óptico de colimación, enfoque y un sistema de movimiento en XYZ con precisión de $0.1\text{ }\mu\text{m}$. Se han desarrollado dos protocolos de fabricación para la creación de electrodos y guíaondas mediante la evaporación de material. El primero es la ablación superior, denominado *front scribing*. Permite la fabricación independientemente de si el sustrato es transparente al UV o no. El segundo protocolo sólo es aplicable a sustratos transparentes al UV denominado *back scribing* con mejores prestaciones pero limitado a ciertos materiales; en este caso la ablación se realiza enfocando el láser a través del sustrato.
- **Sistema de fotolitografía sin máscara o escritura directa por láser LDW** desarrollado en el mismo equipo. Permite el curado selectivo de fotorresina para el proceso de fotolitografía. Este método permite ahorrar muchísimo tiempo en procesos fotolitográficos cambiantes.
- **Sistema de fotolitografía por nano-impresión** Se ha fabricado íntegramente en el CEMDATIC. La máquina ha sido diseñada, fabricada y caracterizada en el desarrollo de la Tesis. Esta máquina puede estar fabricada por impresión 3D o en algún metal (dependiendo de los requerimientos). Con este dispositivo se han reproducido patrones de 233 nm con tamaños de $15\times 15\text{ mm}^2$.

A continuación se enumeran los **dispositivos** desarrollados durante la Tesis:

- **Matrices micro-lentes** Son matrices de lentes cuyo tamaño va desde 1 micrometro hasta centenares de micrometros. Las microlentes individuales se utilizan, por ejemplo, para acoplar luz desde fibras a sistemas ópticos, mientras que las matrices de microlentes a menudo se emplean para aumentar la eficiencia de la recolección de luz de las matrices CCD; recolectan y enfocan la luz que de otro modo habría caído sobre las áreas no sensibles del sensor. En algunos proyectores digitales, se emplean también matrices de microlentes para enfocar la luz a las áreas activas de la pantalla LCD, utilizada para generar la imagen que se proyectará.
 - **Matrices de micro-lentes con focal fija.** Se han desarrollado matrices de micro-lentes con focal fija (micro-lentes convencionales) con tamaños macroscopicos y resoluciones nanométricas gracias a la técnica de litografía por nano-impresión.
 - **Matrices de micro-lentes sintonizables.** Son micro-lentes en las cuales la focal puede variar desde infinito (no hay lente) a focales de pocos milímetros. Se han llevado a cabo distintas aproximaciones de fabricación mediante técnicas como la fotolitografía convencional y la fotolitografía por *front scribing*.
- **Dispositivo sintonizable de cristal líquido para la generación de vórtices ópticos.** Un vórtice óptico es una variación continua azimutal de la fase, produciendo una propagación espiral en vez de una propagación plana. Este dispositivo está caracterizado por la carga topológica (cuantos giros (variación de fase de 2π) efectúa la luz para una longitud de onda).

Figura 1: Esquema de dispositivo óptico para generar vórtices ópticos [1].

Un vórtice se puede generar mediante una lámina de fase espiral (SPP) donde la fase va aumentando en forma de espiral. Se ha desarrollado un dispositivo de cristal líquido, plano, que funciona de forma similar al SPP pero que además es sintonizable. Para ello se ha desarrollado un controlador capaz de conmutar el cristal líquido de manera que la fase cambie en la forma deseada, pudiendo además variar la carga topológica.

- **Separador de potencia por interferencia intermodal (MMI), sintonizable mediante onda evanescente con cristal líquido.** Se han desarrollado PICs que contienen diversas estructuras. Entre estas se encuentran los MMIs, desarrollados en plataforma híbrida $\text{SiO}_2/\text{SU-8}/\text{SiO}_2$ donde el cristal líquido se posiciona encima de la cubierta superior para modificar el campo evanescente de la onda propagada.

- **Dispositivo integrado para la conmutación/separación de polarizaciones de la luz acoplada a la guíaonda de entrada.** Otro de los dispositivos integrados en PICs, fabricado íntegramente en la plataforma polimérica Epocore/Epoclad, es un dispositivo capaz cambiar el camino óptico de una de las polarizaciones mediante el uso de la birrefringencia del cristal líquido (LC). El dispositivo cuenta con al menos una guíaonda de entrada y dos de salida (aunque se han diseñado dispositivos más complejos). El acoplo de luz de la quiaonda de entrada a las de salida se realiza a través de una lámina plano-paralela de LC. Estos dispositivos son además reversibles.

Sistema de ablación láser

La ablación láser es una técnica en la que un material es evaporado/convertido en plasma por la aplicación de energía mediante de un láser.

Figura 2: *Imagen del sistema láser disponible en el CEMDATIC.*

CEMDATIC posee un láser pulsado (Figura 2) de frecuencia de repetición de pulsos sintonizable entre 20 y 300 kHz con una potencia media de 300 mW y a una longitud de onda de 355 nm . El láser posee una plataforma CNC de 3 ejes controlada por ordenador con precisiones de movimiento por debajo de la micra donde el láser es enfocado mediante un objetivo de microscopio 20x. El equipo incluye tres montajes ópticos (Figura 3).

El primero (representado en amarillo) es el encargado de proporcionar una imagen visible del proceso. Está compuesto por un led, que mediante un divisor de potencia es reflejado hacia la muestra. Esta luz es reflejada a su vez en sentido opuesto, hacia una cámara donde es recogida la imagen. El segundo montaje óptico (representado en rojo) es el encargado de medir

Figura 3: *Esquema óptico láser.*

la distancia focal a la muestra mediante un láser de medida y retro-alimentar esa distancia para mantenerla fija. El último montaje óptico (representado en violeta) es el propio láser usado para la ablación. El sistema óptico está compuesto por varios espejos, lentes y un atenuador variable de potencia (para controlar la potencia). Este sistema láser se ha empleado para la ablación láser y para la escritura directa mediante láser (LDW) o fotolitografía sin máscara (descrito en la siguiente sección).

Se han desarrollado dos protocolos de fabricación por ablación láser en el transcurso de la Tesis (ablación superior y inferior), que a su vez se han empleado en dos aplicaciones: fabricación de micro-electrodos de ITO sobre vidrio y fabricación de guíaondas poliméricas.

1. Para la fabricación de electrodos se han optimizado las potencias y las velocidades de barrido para ambos tipos de ablación y para el vidrio empleado (0,7 mm de espesor, ITO de 100 $\Omega/\square$). Estos parámetros variarán ligeramente para otros sustratos de diferente grosor o diferente capa de ITO. Los parámetros optimizados son:
 - Para la fabricación por ablación superior, los parámetros optimizados para el láser son 10 mm/s con una potencia del 18% a 50 kHz a 5 A de corriente.
 - Para la fabricación por ablación inferior, en cambio, la potencia necesaria es mucho menor. Los parámetros optimizados son 10 mm/s a una potencia de 5% a 50 kHz y 5 A de corriente.

Figura 4: Medidas del microscopio de fuerza atómica para la ablación láser de ITO sobre láser. La figura superior muestra la ablación superior. La figura inferior muestra la ablación inferior.

En la Figura 4 se muestran dos cortes a una distancia de $10\ \mu\text{m}$. La imagen superior muestra la ablación superior, la imagen inferior la ablación inferior a través del sustrato. Para asegurar la ablación superior y que no haya conductividad entre electrodos, hay que aplicar mucha energía. Esto hace que además de eliminar el ITO parte del sustrato también se elimine, resultando en una anchura de corte mayor. En cambio para la ablación inferior, la eliminación de sustrato es casi inexistente y el ancho del corte es mucho más pequeño que en la ablación superior.

Figura 5: Perfiles obtenidos de las medidas del microscopio de fuerzas atómicas. En negro se representa el perfil de ablación superior, en rojo el perfil de ablación inferior.

La Figura 5 muestra los perfiles obtenidos de las medidas de AFM y ayudan a cuantificar la cantidad de material eliminado en ambos casos. La referencia es la superficie superior del ITO. La gráfica muestra diferencias sustanciales entre ambos métodos de fabricación. Mientras que para la ablación superior la profundidad de corte es de $0.6 \pm 0.05\ \mu\text{m}$ en la ablación inferior es de $0.13 \pm 0.02\ \mu\text{m}$. En cuanto al ancho, la diferencia es aún más notable. En la ablación superior se obtiene un ancho de $8.0 \pm 0.3\ \mu\text{m}$ mientras que la ablación inferior se obtiene un ancho $1.83 \pm 0.05\ \mu\text{m}$. Por los errores de las medidas

podemos observar que la ablación inferior no solo es más ajustada sino que también es más precisa.

2. La fabricación de guíaondas poliméricas mediante ablación láser fue una de las primeras aproximaciones para la fabricación de guíaondas (Figura 6).

Figura 6: A la izquierda se ve una imagen de microscopio, en ella se puede observar una guíaonda (WG) en SU-8 sobre vidrio. A la derecha es la misma imagen pero con la iluminación del microscopio apagada, en ella se puede observar el guiado de luz roja por la guíaonda; también hay luz guiada en el vidrio.

Para la fabricación de guíaondas sobre polímero se empleó ablación superior por el interés de dejar el máximo espacio entre la guíaonda y el resto de material depositado. Durante la caracterización acoplar la luz se empleó una fibra óptica convencional.

Para ensayar este método de fabricación se ensayaron como materiales de guiado SU-8 y NOA-81. La Figura 7 muestra el funcionamiento para este último material.

Figura 7: Guíaonda fabricada en NOA-81. El dispositivo consiste en un acoplador en Y donde una guíaonda se separa en dos. Estas últimas hacen un giro de 180° . Es posible ver que hay muchísimas pérdidas en el acoplo y en la unión en Y. Sin embargo es posible ver luz al final de las guíaondas pese al radio enorme de la curva.

Sistema de escritura directa por láser (LDW)

El mismo láser explicado en el anterior sección se ha empleado para LDW. Con ese sistema se puede realizar el proceso de fotolitografía sin máscara. Esto agiliza mucho los procesos de afinamiento de los dispositivos, pues un cambio en el dispositivo no requiere de una nueva máscara física.

El proceso consiste en los mismos pasos que una fotolitografía convencional, pero el paso de exposición UV se realiza con el láser. La resolución teórica de este método está por debajo del micrómetro, pero en el Centro se han logrado resoluciones de $1\ \mu m$.

Figura 8: *Círculos de SU-8 fabricados por LDW.*

Por ahora el láser solo permite el método vectorial, muy útil cuando los patrones son líneas, pero en el caso de querer exponer grandes superficies, el método de barrido sería el conveniente. Se prevé tener implementado este método de fabricación en el segundo cuatrimestre del año 2018.

Sistema de litografía por nano-impresión

La nano-impresión es un método de fabricación que proporciona resoluciones nanométricas en superficies macroscópicas. Durante el desarrollo de la Tesis se ha desarrollado una máquina de nano-impresión capaz de aprovechar al menos dos métodos de nano-fabricación (nano-impresión por temperatura y nano-impresión por luz).

Figura 9: *Dispositivo de nano-impresión diseñado y fabricado en el CEMDATIC.*

El sistema consta de dos máquinas y un controlador.

1. **Mold-maker.**- Se encarga de crear una copia intermedia en PDMS del patrón que se quiere fabricar. Está compuesta de tres partes (Figura 9 izquierda) para que luego sea sencillo desmoldar la copia intermedia de PDMS.
2. **Printer.**- Es la encargada de copiar el patrón deseado en el nuevo dispositivo está compuesto por dos partes, la primera (Figura 9 derecha A) es donde se coloca el PDMS, la segunda es donde se fija el sustrato donde se desea realizar la copia final (Figura 9 derecha B). Esta parte está regulada por un controlador de presión y temperatura, donde se controla la presión que el molde de PDMS ejerce sobre la muestra y la temperatura del sustrato.

El proceso de fabricación está representado en la Figura 10. Los pasos a realizar serían:

Figura 10: *Pasos necesarios para la nano-impresión.*

1. Hacer un molde negativo en PDMS del dispositivo a nano-fabricar con la ayuda del Mold-maker.
2. Copiar el patrón del PDMS en la muestra (previamente con el material depositado) con ayuda del Printer usando el controlador de presión.
3. Curar el material depositado (bien mediante la aplicación de temperatura o de luz).
4. Retirar el molde de PDMS y la muestra nano-impressa.

En el desarrollo de la Tesis se ha ensayado este equipo exitosamente hasta resoluciones de 233nm en superficies de $1.5 \times 1.5 \text{ cm}$.

Figura 11: *Master y copia en OrmoStamp para una red de difracción triangular de periodo de repetición 555nm.*

En la Figura 11 se pueden observar medidas de AFM para el master y la copia en OrmoStamp. Las medidas corresponden a zonas arbitrarias, tanto en el master como en la copia.

Matrices de micro-lentes de focal fija

Mediante la técnica de nano-impresión se ha fabricado matrices de micro-lentes de focal fija. Esta focal es dependiente del material empleado (Figura 12).

Figura 12: Foto del máster y la copia en OrmoStamp de microlentes

Los errores de la copia el método de nano-impresión son de pocos nm , errores totalmente irrelevantes para el funcionamiento de estas micro-lentes.

Matrices de micro-lentes de focal variable

Para el desarrollo de micro-lentes de sección variable se fabricaron lentes de cristal líquido modales (MLCL). Estos dispositivos afectan al camino óptico de la luz de manera análoga a como lo hace una lente convencional, pero variando el camino óptico mediante un cambio en el índice de refracción del LC, al variar la orientación de sus moléculas, en vez cambiar el espesor de la lente (Figura 13).

Figura 13: Imagen representativa del camino optico de la luz a traves de una lente (izquierda) y una MLCL (derecha).

Para la fabricación de los electrodos necesarios para crear el patrón de conmutación deseado se emplearon dos métodos:

- **Ablación láser.**- el electrodo se dibuja directamente mediante ablación láser (explicado en apartados anteriores.)

Figura 14: *Distintos motivos para la fabricación de electrodos por el método de LDW.*

Los cálculos de las resistencias de estos patrones oscilan entre los pocos cientos de ohmios hasta algunos kilo-ohmios. En el electrodo de la figura Figura 14 B teóricamente la resistencia hacia el centro es infinita y el LC conmuta por acoplo capacitivo.

Figura 15: *Lentes con electrodo fabricado por ablación láser a 10 kHz para distintas tensiones*

En la Figura 15 se puede observar que los resultados obtenidos mediante este tipo de electrodos no fue del todo satisfactorio.

- **Fotolitografía convencional.**- mediante este método se eliminó todo el electrodo del círculo interno de la lente quedando así una zona sin electrodo. Este tipo de lentes se conce como lentes modales. Se añadieron diferentes capas para intentar conseguir mejorar la lente. Estos diseños se ilustran en la Figura 16.

Figura 16: *Diferentes aproximaciones probadas para la fabricación de MLCL .*

En la Figura 17 se pueden ver los resultados de los ensayos de capas de SiO_2 y níquel. Se puede observar que el resultado final no es aceptable puesto que la caída del campo eléctrico no forma una lente de calidad.

Figura 17: *Micro-lentes con capa capacitiva de SiO_2 y Ni excitadas con una señal de 10 kHz con diferentes amplitudes.*

En la Figura 18 se ven los resultados para los ensayos con capa resistiva de PEDOT:PSS. El PEDOT:PSS se empleó como conductor de alta resistividad y como capa de alineamiento. La respuesta a 10 kHz no era la esperada y se aumentó hasta los 10 MHz donde a pesar de una leve mejora no se consiguió la calidad deseada. En este tipo de lentes la tensión umbral es mayor que para las que emplean capas de alineamiento de poliimida.

Figura 18: *Micro-lentes con capa resistiva de PEDOT:PSS excitadas con una señal de 10 MHz a diferentes amplitudes.*

Por último la aproximación más sencilla, las lentes modales referidas anteriormente, en las cuales se elimina todo el electrodo de la zona central, fueron las que mejores resultados ofrecieron. La Figura 19 muestra el resultado obtenido y a partir del cual se obtuvieron las diferentes distancias focales empleando la ecuación (1) y conociendo los desfases por análisis de variación del color (Tabla 3.2).

$$F = \frac{r^2}{2 \cdot \Delta n \cdot d} \quad (1)$$

Figura 19: *Micro-lentes sin capas adicionales excitadas con una señal de 10 kHz con diferentes amplitudes.*

Cuadro 1: *Distancia focal en función de la tensión aplicada.*

Voltage [V]	Retardation $\Delta n \cdot d$ [nm]	Focal length [mm]
12	800	2.6
16	700	2.9
20	620	3.3
24	570	3.6
28	520	3.9
32	480	4.3
36	440	4.7
40	400	5.0
44	370	5.4
48	350	5.8

Dispositivo sintonizable de cristal líquido para la generación de vórtices ópticos

Se ha diseñado, fabricado y caracterizado un SPP sintonizable de cristal líquido. Para ello, se realizó una aproximación de discretización de un SPP en 24 secciones (Figura 20).

Figura 20: Representación de la fase de un SPP con cargas topológicas 1 y 2, para (a) y (b) desfases continuos, (c) y (d) con discretización de los desfases introducidos en 24 segmentos

El dispositivo fue fabricado con un electrodo (Figura 21) de ITO sobre vidrio mediante la técnica de ablación láser superior.

Figura 21: En la imagen se observa el diseño del electrodo fabricado a escala 1:1. El sustrato de vidrio con el diseño del electrodo mide $50 \times 50 \text{ mm}^2$, y el contra-electrodo mide $30 \times 20 \text{ mm}^2$. El área activa (violeta) mide $10 \times 10 \text{ mm}^2$ limitada por el inicio de la salida en abanico (azul). El paso de electrodos es de 4 mm con un ciclo de trabajo del 50 %.

Para el control del dispositivo se fabricó un driver controlado por ordenador donde se puede controlar la tensión aplicada a cada uno de los 24 segmentos, controlando la anchura del ciclo de trabajo (PWM). Este controlador tiene una resolución de 12 bits por segmento (Figura 22).

Antes de generar los vórtices, el dispositivo se caracteriza colocándolo entre polarizadores cruzados para obtener el retardo en cada punto mediante la obtención intensidad ($I \propto \sin^2 \frac{\delta}{2}$) en una cámara.

La Figura 23 muestra el retardo inducido en función del campo aplicado para cuatro regiones aleatorias de la superficie del dispositivo. El resultado muestra que el desfase producido es

Figura 22: Driver de control del SPP de 24 segmentos con una resolución de 12bit por segmento.

idéntico independientemente de la posición. Las variaciones de amplitud se deben a diferencias de iluminación.

Figura 23: Intensidad en función de la tensión aplicada para cuatro regiones distintas. El eje vertical representa las cuentas de la CCD y el eje horizontal el ciclo de trabajo empleado

La diferencia de fase fue ajustada a una función pseudo exponencial $\delta = Ae^{-B \cdot dc} + C$ entre 3π y π . La Figura 24 muestra dicho ajuste.

Una vez calibrado el dispositivo, se procede a generar los vórtices. En la Figura 25 (fila

Figura 24: Representación de $\delta - \pi$ (eje izquierdo) y la intensidad normalizada (eje derecho) junto con su ajuste exponencial.

superior) se puede observar la imagen del dispositivo entre polarizadores cruzados para distintas cargas topológicas aplicadas (1,2,3,4,6,8 y 12). Tras eliminar uno de los polarizadores se pueden capturar los vórtices generados Figura 25 (fila intermedia). Por último en la fila inferior de la imagen se puede observar una simulación realizada mediante óptica de Fourier, que concuerda fielmente con los experimentos (Figura 25 (fila inferior)).

Figura 25: La fila superior muestra el retardo (δ) introducido por el vórtice óptico para diferentes cargas topológicas (1,2,3,4,6,8 y 12) observando el dispositivo entre polarizadores cruzados. La fila central muestra la distribución de potencia del haz a la salida del dispositivo. La fila inferior muestra la simulación realizada mediante óptica de Fourier.

MMI sintonizable mediante cristal líquido

El desarrollo de este dispositivo comienza con la selección de los materiales y las longitudes de onda de la luz a propagar. se escogió SiO_2 como cubierta y SU-8 como núcleo y longitudes de onda de 632.8 nm (roja) y verde a 543 nm . Conocidos los materiales y las longitudes de onda, se procede a simular los dispositivos.

Figura 26: *(Simulación de MMI 1x2 en COMSOL)*

Se puede observar, la propagación a través de un MMI 1x2 simulado mediante COMSOL donde a la salida observamos la potencia dividida al 50 %. De estas simulaciones se obtienen las medidas del MMI para fabricar la máscara fotolitográfica. Se diseñaron 2 máscaras. La primera constaba de 16 chips de $2 \times 2 \text{ cm}$, divididos en 8 chips para cada longitud de onda. Cada grupo de 8 chips se diseño para 4 anchuras de guiaonda (0.6, 1, 1.5, 2 micrometros). En el segundo diseño realizado se disminuyó el tamaño del chip a $1 \times 1 \text{ cm}$ y el nivel de integración creció hasta los 52 chips (Figura 27).

Figura 27: *(Izquierda) Primera máscara de 16 chips $2 \times 2 \text{ cm}$. (Derecha) Segunda máscara de 52 chips $1 \times 1 \text{ cm}$.*

Para garantizar pérdidas por curvatura razonables se restringió en el diseño el radio de curvatura mínimo a 150 micrometros.

Los dispositivos se fabricaron mediante fotolitografía convencional.

La Figura 28 muestra dispositivos MMI 2X2 fabricados en las instalaciones del CEMDATIC.

Una vez fabricados los chips e inspeccionados. Se procedió a cortarlos (Figura 29).

Por último se procedió a caracterizar los chips. Se empleó un montaje óptico de precisión nanométrica para acoplar luz en los chips. En primera aproximación, los chips fueron probados sin cristal líquido para posteriormente proceder a la integración del chip en una célula de LC y hacerlo sintonizable. La Figura 30 muestra como se guía la luz por el chip.

Figura 28: *Dispositivos fabricados mediante fotolitografía convencional.*

Figura 29: *(Izquierda arriba) Chip de la primera máscara sobre vidrio, (abajo izquierda) comparacion entre chips de la primera máscara y la segunda sobre Si, (derecha) chip con integración de cristal líquido.*

Figura 30: Imagen del acoplo (izquierda arriba), guiado (izquierda abajo) y salida (derecha) a través de un dispositivo MMI 2X2.

Por último se caracterizó la potencia de salida frente a la tensión (Figura 31) de los chips integrados en células LC (Figura 29 derecha) mediante una CCD.

Figura 31: Potencia relativa a lo largo del tiempo para una señal cuadrada aplicada al LC de 0.45 Hz.

El dispositivo es capaz de cambiar la potencia relativa a la salida entre un 70 %-30 % (Off) y un 53 %-47 % (On).

Dispositivo integrado para la conmutación/separación de polarizaciones

El ultimo dispositivo diseñado, fabricado y caracterizado en esta Tesis es un conmutador/separador de polarizaciones. Este dispositivo fue diseñado para una longitud de onda 633 nm en la plataforma polimérica Epocore/Epoclad. Fue fabricado en el CMST/IMEC en la Universidad de Gante, mediante técnicas de LDW.

Figura 32: *Diseño del dispositivo conmutador/separador de polarización. En la imagen se pueden ver los dos posibles estados de conmutación para el cristal líquido.*

En la Figura 32 se muestra la estructura básica del dispositivo. Este está compuesto de dos guíasondas paralelas, donde se interseca una cavidad plano-paralela que se llena de LC. Al conmutar el LC mediante campo eléctrico, varía su birrefringencia y se inducen distintos caminos ópticos para cada polarización.

Mediante una simulación FDTD en Lumerical se puede simular los distintos caminos ópticos para la luz (Figura 33). Se observa, que dependiendo de la polarización guiada (TE o TM), para un alineamiento del LC, una polarización se desvía a la guía-onda colindante mientras que la otra polarización continua recta. Para el otro alineamiento ocurre justo lo contrario.

Figura 33: *Simulaciones del camino óptico de la luz (con fondo azul). Y separado por polarizaciones (fondo verde) para los dos posibles estados de conmutación.*

En el dispositivo fabricado (conmutador de polarizaciones), una de las polarizaciones se radia por la cubierta, mientras que la otra es desviada a la guía-onda de salida. Al conmutar el LC lo que se consigue es el intercambio entre polarizaciones guiadas y radiadas. La Figura 34 muestra imágenes de la guía-onda y la cavidad plano-paralela fabricadas.

Figura 34: a) Guía-onda; b) Cavidad plano-paralela vista desde arriba; c) Perfil de la cavidad plano-paralela fabricada por ablación láser.

La estructura del dispositivo se muestra en la Figura 35.

Figura 35: Medidas, capas y diseño del dispositivo fabricado.

Se fabricaron varios chips, con numerosos dispositivos, donde la única diferencia entre dispositivos era la distancia entre guía-ondas.

Figura 36: Intensidades TE/TM para dos estados de conmutación del cristal líquido.

Para la caracterización, se acopló la luz a la guía-onda mediante una fibra monomodo. La intensidad óptica a la salida se recogió mediante una fibra multimodo y se llevó a un fotodetector. En la Figura 36 se muestra la potencia de salida para distintas distancias entre guía-ondas. Los maximos no coinciden debido a que el alineamiento del LC no es perpendicular al modo TE, haciendo que el índice efectivo del LC para este modo sea menor que para el TM debido a defectos en el proceso de fabricación.

ABSTRACT

This PhD work is focused on the development of a new research line at the Centro de Materiales y Dispositivos Avanzados para las TIC (CEMDATIC) based on the design, manufacturing, and characterization of photonic integrated circuits (PICs). Moreover, several previous research lines on phase-only devices have been continued, developing new devices and technologies that could be used in PICs with small adjustments. Two instruments have been modified and significantly improved upon PhD realization. A new nano-imprint lithographic system has been developed, as well as an optical setup for light coupling with nanometer precision for chip characterization; this setup includes the required modules for electronic control. The fabrication **methods** developed during the PhD Thesis have been:

- **Ablation laser system.** The equipment consists of a tripled Nd:YAG 50 *kHz*-pulsed laser (355 *nm*) having a 300 *mW* output, collimation optical system, focusing and XYZ mechanical stages with a numerical control system with 0.1 μm precision. Two fabrication protocols have been developed for generation of electrodes and waveguides through material evaporation: i) front scribing, *i.e.* ablation from the upper side, allowing fabrication regardless the substrate is transparent to UV or not, and ii) back scribing, that shows better performance but can only be used in UV transparent substrates; ablation is done focusing the laser through the substrate.
- **Maskless photolithography through laser direct writing (LDW).** This system has been developed on the same laser. It allows selective curing of photoresist for photolithography. This method is particularly suitable for research activities where a high number of different photolithographic designs have to be tested. Replacing mask construction by LDW leads to significant savings of time and money.
- **Nanoimprint lithographic system.** The full system has been design, fabricated and characterized as a part of the PhD work. The device has successfully manufactured $15 \times 15 \text{ mm}^2$ replicas of 233 *nm* feature size.

The **devices** developed during the PhD Thesis have been:

- **Micro-lenses array.** Lens matrices whose diameter ranges from 1 to several 100's μm . Single micro-lenses are used, for example, for fiber coupling to optical systems, whereas micro-lenses matrices are often employed to improve light collection in CCD matrices.
 - **Fixed focal distance micro-lenses.** Conventional micro-lenses matrices, having fixed focal distance, have been developed with nanometric resolution and macroscopic size using nanoimprint lithography.

- **Tunable focal distance micro-lenses.** In these micro-lenses matrices, focal distance varies from infinity (no lens) to a few *mm*. Several manufacturing approaches have been tested using conventional photolithography and by laser ablation photolithography.
- **Tunable liquid crystal device for optical vortex generation.** An optical vortex is a continuous azimuthal phase variation, leading to a spiral rather than plane propagation. The device is characterized by its topological charge, *i.e.*, number of full turns per wavelength performed by the helical beam.

Vortices can be generated by spiral phase plates (SPP), devices where phase delay increases with azimuthal angle in such a way that a full turn is always a multiple of 2π phase shift. A liquid crystal flat structure has been developed; it is able to mimic a tunable SPP. A controller (also developed in this work) drives the cell, which arranges phases as required, and allows modifying the topological charge.
- **Power distribution by intermodal interference (MMI),** tunable through liquid crystal interaction with evanescent wave. Several PICs containing different structures have been developed. Among these, some MMIs have been created on hybrid platform $\text{SiO}_2/\text{SU-8}/\text{SiO}_2$. Liquid crystal is deposited onto the upper lid, allowing its interaction with the evanescent field of the propagating wave.
- **Integrated device for switching/splitting orthogonal polarizations of the light coupled to the input waveguide.** Another device integrated in PICs. It has been made completely in polymers Epocore/Epoclad. The device is able to modify the optical path of one polarization through the liquid crystal birefringence. The device includes, at least, one input and two output waveguides -more complex geometries have been created as well. Light coupling from the input to the output waveguides is done through a planar parallel liquid crystal layer. Moreover, the devices are reversible.

Chapter 1

INTRODUCTION

Optical communications began with the invention of the laser in 1960. By these years glass-cladding fibers had attenuations about 1 dB per meter, fine for medical imaging but too high for communications. In 1966, Charles Kao demonstrates the possibility of lowering the attenuation by using sufficiently pure glass for light waves to bear information. Kao received the Nobel Prize in Physics in 2009 for this work.

Photonics has undergone a huge development since the appearance of first optical communications in the 70s, which made possible the communication at rates never seen before. The photons then became to take the place that had been occupied by electrons for many years. The fiber optic communication systems have revolutionized the telecommunication industry. The advantages of light-guiding transmissions instead the electrical transmission convert the optical communications into the main system to transmit large amounts of information at long distances. Every day the optical fiber network becomes wider; in many countries, it is customary to bring photons to the user's home in the so-called Fiber To The Home (FTTH). The connection rate is typically 100 *Mbps* or more, 20 to 100 times faster than a typical modem or Digital Subscriber Line (DSL).

It was in 1970 when fused silica glass became to be tested for optical fibers manufacturing, resulting in a patent in 1973 [2] by a team of researchers led by Robert Maurer with Donald Keck and Peter Schultz at Corning Glass. This fiber optic wire was capable to carrying 65,000 times more information than copper wire. This fiber was a titanium-doped single-mode (SM) fiber with loss of 17 *dB/km* at 633 *nm*. In 1972 the same team patented a germanium-doped multimode (MM) fiber [3] with a loss of 4 *dB/km*. In April 1977, General Telephone and Electronics tested and deployed the world's first live telephone traffic through a fiber-optic system. These fibers transmitted light at 850 nanometers (the so-called first window) from gallium-aluminum-arsenide laser diodes at data rates of 6.2 and 45 *Mbits* per second. Today more than 80 percent of the world's long-distance voice and data traffic is carried over optical-fiber cables.

Developers were bothered with early SM fibers development because the difficulty to achieve the micrometer-scale needed to couple light efficiently. Not satisfied with the low bandwidth of step-index multimode fiber, they concentrated on multi-mode fibers with a refractive-index gradient between core and cladding, and core diameters of 50 or 62.5 μm . A second generation soon appeared with the InGaAsP laser at 1300 *nm* (second window),

where fiber attenuation was about 0.4 dB/km . The necessity to cover long distance without repeaters turned the group at Bell Labs (and then the rest of communication companies after deregulation in the early 80s) to work with SM fibers again to avoid pulse dispersion of MM fibers.

Although it was clear to developers that the minimum attenuation of glass fibers was below 0.2 dB/km at 1550 nm , it was not until 1992 when this window began to be used owing to the difficulty of developing lasers at this wavelength and the chromatic dispersion.

The next big step was the arrival of the erbium-doped fiber amplifier (EDFA) in 1987 that allowed the possibility to amplify light at 1550 nm [4]. An all-optical amplifier was much simpler and faster than an electro-optic repeater. The developers also found that EDFAs could simultaneously amplify signals of different wavelengths transmitted through the same fiber, the wavelength-division multiplexing (WDM) that multiplied the potential transmission capacity of the fiber. The first transatlantic cable using optical amplifiers, TAT-12/13 in 1996, was able to transmit 5 Gbps through a single pair of fibers at one wavelength. Terrestrial technologies were capable to carry even higher bandwidths by packing the wavelengths close together the dense wavelength-division multiplexing (DWDM) transmitting dozens of channels simultaneously at 2.5 or 10 Gbps . By 2001 some manufacturers were offering DWDM systems able to transmit 100 wavelengths at 10 Gbps each, a total of one terabit per second.

Today light is propagated at different wavelengths that can travel independently by the same fiber with comparatively little crosstalk or interference. This allows to send 100 or more separate data streams by a single fiber at the same time achieving 20 Tbps [5]. Today, engineers also modulate the shape of the pulses, their phase, their polarization, and their physical spacing in the fiber. However, after great distance travels optical distortions creep in, causing errors to build up and degrade the data. This problem has been recently alleviated by ensuring that the laser variations were predictable rather than random [6].

Optical communications on standard fibers are nowadays a well-known technology where research has evolved from the materials and components to the systems. Yet research on new materials and devices that may improve the current media have never ceased. The underlying idea is that specific challenges or applications are best suited to alternative realizations. Nobody actually believes that any alternative medium will replace the present deployment of long distance optical fiber networks in the short or medium term. Yet a high number of applications for short (local area networks, LAN) and ultra-short distances exist, where the ends of the waveguide are in the same building (100 m , LAN), room (10 m), system (1 m) or circuit (0.1 m). Optical sensors, integrated optics, optical computing, optical cross-boards, add-drops or switches are just a few examples of this field. This is the field where the capabilities of new materials, devices and configurations must be tested, and the central subject of this work.

1.1 Optical waveguides

An optical waveguide is defined as any structure able to confine light from one point to another and preserve desirable properties along the propagation way. It is important to note that it is not referred only to the light is visible by humans but also a wider range of the spectrum including infrared and ultraviolet wavelengths. The basic structure in photonic integrated circuits is the planar dielectric waveguide; this introductory discussion is focused on

this particularly geometry. To better understand how light remains inside these guides some physical properties must be explained.

1.1.1 Reflection and refraction

Let us consider a monochromatic light that impinges onto a plane boundary surface between two media. While one part of this light is reflected, the other is refracted into the second medium (assuming there is not scattering, neither absorption). If this media have different refractive indices, the velocities of the light waves will be different in each medium. The incident angle and the refraction angle are related by Snell's Law of Refraction (Figure 1.1). The reflection angle is always equal to the incident angle [7, 8].

Figure 1.1: *Snell's Law of Refraction and critical angle. There are two medium represented with two different colors (blue (n_1) and green (n_2) and $n_1 > n_2$). The figure show the three main possibilities depending on the incident angle. (Left) transmission of the light; (center) critical angle; (right) total internal reflexion.*

This can be written in terms of the refractive index *i.e.* the ratio between the velocity of light in vacuum and the velocity of light in a particular medium.

$$v^2 = \frac{1}{\mu\epsilon} \approx \frac{1}{\mu_0\epsilon} \quad n = \frac{c}{v} \quad \frac{\sin \theta_1}{\sin \theta_2} = \frac{v_1}{v_2} \quad n_1 \sin \theta_1 = n_2 \sin \theta_2 \quad (1.1)$$

If the incident light impinges from a **higher** refractive index medium at a certain angle greater than a critical angle, all the light is reflected back into the incident medium. This optical phenomenon is known as total internal reflection (TIR). The critical angle is obtained when the refraction angle is parallel to the interface. Then:

$$\sin \theta_2 = 1 \quad \implies \quad \theta_c = \arcsin \left(\frac{n_2}{n_1} \right) \quad (1.2)$$

This total internal reflection can be employed to use one medium as propagation medium when placed between two media with lower refractive indices, thus it is finally gotten a three layer structure or slabs (core and two claddings) within which light is infinitely bounced. This phenomenon can also be achieved by rotating the simple structure around a revolution axis

placed at the edge of the core getting a circular waveguide as a result. These waveguides are commonly known as optical fibers and despite they have been widely used in telecommunications, they are not suitable for integrated circuits manufacturing. This is the reason why this work focuses on planar waveguides.

1.1.2 Planar dielectric waveguides

Slab or planar waveguides will be used as explanatory example due to its geometrical simplicity. They consist of planar layer of a high-refractive-index material which is sandwiched between a substrate and a cover with lower refractive indices. In this analysis two of the three space dimensions are considered infinitely wide.

When the guided light impinges at one end, there is a maximum angle of incidence for which it is guided. This angle is known as the acceptance angle. For higher incidence angles the light coupled to the guide would not meet the critical angle condition (in the substrate interface or for the cover interface, the one with the highest refractive index) and would be radiated. Therefore, the internal incidence angle must not exceed the critical angle in order to achieve TIR but also must fulfill the phase-matching criterion: when the light is reflected by TIR in a interface suffers a phase delay that depends on the incidence angle with respect to the interface. Phase difference must be zero or a multiple of 2π between each wavefront¹ and itself after reflected twice so that they do not cancel out each other by destructive interference (Figure 1.2) [8].

Figure 1.2: *Propagating rays in a parallel dielectric structure. Three media are presented with different colors. Blue refractive index is the highest. Light is propagating from left to right with the TIR condition; also the phase difference is 0 or 2π multiple.*

Therefore two conditions have to be met for light to be guided. First, an incidence angle within the acceptance angle. Second, a phase-matching condition of the non-propagating component. Every mathematical solution meeting these conditions is called a **mode** (propagation mode), and define the number of ways light can propagate in a given waveguide.

¹In a single mode waveguide, an important amount of the light is traveling outside the waveguide (evanescent field). In this case we cannot talk properly about a *wavefront*, but this restriction remains valid.

The propagation equation gives the propagation constant as a function of light frequency. When indices of substrate, cladding and guiding medium are very similar this is not very practical and a normalized frequency V is defined. If the waveguide is symmetric ($n_{clad} = n_{substrate}$) and $V < \pi (m = 1)$ only the fundamental mode travels along the guide while higher-order modes are cut off. This is called a single mode waveguide [9].

$$V = ka\sqrt{n_1^2 - n_2^2} = \frac{2\pi a}{\lambda}\sqrt{n_1^2 - n_2^2} \quad k = \frac{2\pi}{\lambda} \quad (1.3)$$

Where λ is the wavelength on vacuum and a is the waveguide height. The cutoff mode frequency can be calculate as:

$$V_0 = \arctan \sqrt{\frac{n_2^2 - n_3^2}{n_1^2 - n_2^2}} \implies \lambda_c = \frac{2\pi}{V_0} a \sqrt{n_1^2 - n_2^2} \quad (1.4)$$

The next modes can be calculated as:

$$V_n = V_0 + n\pi \quad (1.5)$$

Depending on the input light frequency, some modes propagate while others not. That means some of them confine most of power inside the core while others spread it through the cladding. Actually note that even in guided modes some light filter out the interface between media, as shown in the figure below. This filtering field is known as evanescent wave [7], and even though it decreases exponentially very quickly with distance away from boundary, it is crucial in integrated circuits because of other surrounding structures are very close (Figure 1.3).

Figure 1.3: *Evanescent field in a slab waveguide. The three first modes from TE polarization are represented.*

1.1.3 Guided modes

An optical mode represents a field distribution that satisfies the propagation condition for a particular waveguide. For every scenario satisfying the conditions mentioned above, two orthogonally polarized wave-fronts will be propagated at the same velocity so the number of modes traveling through a planar waveguide is always an even number. In practice, only one half is considered by convention. To better state this point let us consider a planar waveguide infinitely extending in the y and z directions and a monochromatic light. When Maxwell's Equations are solved for this particular case in the core region, assuming that there is no variation in y direction, this results in two overlapped plane waves propagating over z [10].

$$\nabla \times \vec{H} = \varepsilon_0 n_1^2 \frac{\partial \vec{E}}{\partial t} \quad \nabla \times \vec{E} = \mu_0 \frac{\partial \vec{H}}{\partial t} \quad \vec{E} = E_{x,y} e^{j\omega t - \beta z} \quad \vec{H} = H_{x,y} e^{j\omega t - \beta z} \quad (1.6)$$

Solving rotational and considering $\frac{\partial \vec{E}}{\partial y} = \frac{\partial \vec{H}}{\partial y} = 0$ two independent electromagnetic guided modes:

- TE Mode (Transverse Electric), since the electric field lies in the plane perpendicular to the propagation direction.

$$\frac{\partial^2 E_y}{\partial x^2} + (k^2 n^2 - \beta^2) E_y = 0 \quad H_x = -\frac{\beta}{\omega \mu_0} E_y \quad H_z = \frac{j}{\omega \mu_0} \frac{\partial E_y}{\partial x} \quad E_x = E_z = H_y = 0$$

- TM Mode (Transverse Magnetic), since the magnetic field lies in the plane that is perpendicular to the propagation direction.

$$\frac{\partial^2 H_y}{\partial x^2} + (k^2 n^2 - \beta^2) H_y = 0 \quad E_x = \frac{\beta}{\omega \varepsilon_0 n^2} H_y \quad E_z = \frac{-j}{\omega \varepsilon_0 n^2} \frac{\partial H_y}{\partial x} \quad H_x = H_z = E_y = 0$$

Although many analytical and numerical techniques, such as the perturbation method analysis, have being established for three-dimensional rectangular waveguides modeling (Figure 1.4), most of them are not reliable when non-linear behaviors exist. A common solution called the beam propagation method (BMP) consist on reducing the space under study until the fields in it could be approximated to a scalar wave and then solve the field distribution in slices along the propagation direction.

1.2 Photonic integrated circuits (PICs)

A photonic integrated circuit (PIC) is a device that integrates multiple (at least two) photonic functions and as such is similar to an electronic integrated circuit. The main difference is that photonic integrated circuits implement functions using light instead of electrical signals.

PICs have many potential applications [11, 12, 13] including next generation optical networks. The most employed platforms are silicon on insulator (SOI) and Indium Phosphide

Figure 1.4: *3D slab waveguide representation and 3D ridge waveguide representation examples.*

(InP) [14]. InP allows for manufacturing of passive and active devices in the same platform technology [15]. On the other hand, the SOI compatibility with CMOS technology leads to excellent processing control, low cost and high volume processing. Furthermore, SOI enables ultra-sharp bends in the optical waveguide, because of their high refractive index contrast [16] and thus allow for high device density and hence integration level.

Nowadays, polymer-based PICs are mainly being used in interconnections between conventional inorganic (SOI or InP) PICs [17, 18], ideal as intermediate (e.g. mode field converting) structures between a densely integrated SOI PIC (very small mode field diameter MFD) and telecom fiber (larger MFD).

On the other hand, polymer waveguides are low-cost with intrinsic low propagation loss in the visible range and near infrared, making them an attractive alternative platform to SOI and InP in a price league of its own [19], in case visible light operation is required or in case longer distances need to be bridged, e.g. for on-board chip-to-chip interconnections. Polymer

Table 1.1: *Comparative of the most common integrated optic platforms*

	Polymer	SOI	InP
WG Size	0.5-8 [μm]	0.2-0.5 [μm]	0.8-2 [μm]
Wavelength	VIS & NIR	NIR	NIR
Losses	<1dB/cm	<3dB/cm	<4dB/cm
Active	Yes	No	Yes
Price	Cheap	Same that electronic	Expensive
Flexible	Yes	No	No
Electronic integration	Organic Electronic	Yes	No

PICs are characterized by a relatively low index contrast, which does not allow realizing sharp bends. However, this reduced contrast also implies larger waveguide dimensions, so that the MFD can be tailored to that of an optical fiber to achieve very low coupling losses. Another possible drawback in some applications is their limited thermal and environmental stability. Table 1.1 shows the differences between platforms.

In this work several platforms have been tested in visible region, so SOI and InP are not

considered. The operating principles of several optical devices are presented below.

1.2.1 Couplers

An optical coupler is a device used in optical systems with one or more inputs and one or more outputs. The light that is introduced by one input and coupled to one or more exits. Its power distribution potentially depends on the wavelength and polarization.

1.2.1.1 Y-junction

The device consist in a waveguide that splits in a “Y” pattern. The idea is to split the power of the incidence signal in two signals with the desired power in each one.

1.2.1.2 Directional coupler

This device is composed of two single-mode waveguides placed close to each other (Figure 1.5). When the distance between them is small enough, light propagating through one of them is coupled into the other due to the evanescent field excitation. It should be noticed that mode coupling occurs not only in the straight region but also in the curved region. Therefore, the coupling length is actually an effective straight coupling length that includes the entire mode-coupling effect in the straight and curved coupling regions [8].

$$L_e = L_s + 2R \cos^1 \left(1 - \frac{d_c - d}{2R + w} \right) \quad (1.7)$$

L_e : effective coupling length.

L_s : straight coupler length.

R : bend radius.

d : gap between waveguides.

d_c : gap between waveguides where light start coupling in the bent region.

w : waveguide width.

Figure 1.5: Sketch of a directional coupler.

if both waveguides are identical, all the light is transferred after some propagation distance called **coupling length** L_c . At twice this coupling length, the power is coupled back to the original waveguide by the same process. Thus, the optical power can be transferred periodically from one waveguide to the other. This allows to reach any power ratio at the output by changing light propagation properties across the coupling region [20, 21].

The mode analysis of the two waveguides cannot be considered single-mode anymore because this geometry has its own set of propagation modes. It is usually solved by a superposition of two (even and odd) fundamental modes of the individual waveguides, each having its own propagation constant. Then the power exchange is defined by the coupling constant K that depends on the waveguide geometry, distance between the two waveguides and the

effective coupling length [8].

$$L_b = \frac{\lambda}{2(n_{\text{even}} - n_{\text{odd}})} \quad L_e = \frac{2L_b}{\pi} \sin^{-1} \sqrt{K} \quad (1.8)$$

Where **the beat length** L_b is the length required on a directional coupler for the light to be totally coupled to the waveguide where it was not injected.

1.2.1.3 Multi-mode interference coupler

This device uses the interference of normal modes so that input field is reproduced in single or multiple images (self-imaging) [22] at periodic intervals along the propagation direction. Although two normal modes are sufficient to implement common switching elements, improvements are possible if higher-order modes are employed [23].

Figure 1.6: Multi-mode interferometer (1×2). It is composed by one single mode waveguide, one multimode waveguide (size $W \times L$) and two single mode waveguides at the output.

For a multimode waveguide with width W and refractive index n , the propagation constants of the low-order normal modes can be approximated by

$$\beta_m = \sqrt{\left(\frac{2\pi n}{\lambda}\right)^2 - \left(\frac{(m+1)\pi}{W}\right)^2} \quad (1.9)$$

Then the propagation constant difference between the fundamental mode and other higher-order modes and the beat length of the two lowest modes can be expressed as:

$$\beta_0 - \beta_m = \frac{\pi\lambda}{4nW^2} m(m+2) \implies \beta_0 - \beta_1 = \frac{3}{4} \frac{\pi\lambda}{nW^2}, \quad L_\pi = \frac{\pi}{\beta_0 - \beta_1} = \frac{\lambda}{2(n_0 - n_1)} \quad (1.10)$$

Single-mode waveguides are connected to the multimode waveguide acting as inputs and outputs and two different configurations can be depicted according to the input position [21, 22] (Table 1.2):

Table 1.2: *MMI parameters for designs*

	General (N input)	Symmetric interference	Paired interference
Inputs X Outputs	N X N	1 X N	2 X N
First Single Image	$3L_\pi$	$3L_\pi/4$	L_π
N-fold image	$3pL_\pi/N$	$3pL_\pi/4N$	pL_π/N
Excitation requirement	none	odd	$\nu = 2, 5, 8, \dots$
Input location	any	$x=0$	$x=\pm W_e$

This structure has been simulated and manufactured in different devices. In both configurations, p factor denotes the periodic nature of the imaging along the multimode waveguide as it can be shown in the Figure 1.7.

Figure 1.7: *Multi-mode interferometer waveguide. Light input is propagate from left to right. The electromagnetic field power is presented. Multiples self images can be seen in the propagation of the light.*

1.2.2 Ring resonators

An optical ring resonator is a coupled structure in which one or more waveguides are closed on themselves forming a loop and others act as input or output ports. Light coming from the input waveguide can be transmitted inside the ring either by a directional coupler through the evanescent field or by MMI couplers. Circulating light is constructively interfered inside the resonating cavity before being coupled again into the output waveguide or into other resonators. The result is a filter in which light is accepted or rejected (notch filter) at some wavelength depending on the ring circumference length (Figure 1.8). This is because resonance only occurs when the optical path length of the ring is a whole number of times the coupled light wavelength [24].

$$\lambda_{res} = n \frac{2\pi R}{m} \quad m = 1, 2, 3, \dots \quad (1.11)$$

Where λ_{res} is the resonance wavelength, m denote periodicity and it is a integer.

Since its optical characteristics can also be externally modified, ring resonators are suitable for active optical devices. Thermal tuning is the most widely used technique because of the large thermo-optic response of silicon. Other solutions can be found for other core materials. Heating is achieved by placing high-resistivity metal micro-heaters either at the top or at the side of the resonating cavity [25].

Figure 1.8: Basic configuration of a single ring resonator structure. The light input is at the left of the top waveguide (red arrows). Light is coupled to the ring and the λ_{res} is represented in green and go through the left of the bottom waveguide. The remaining wavelengths go through the top waveguide (yellow arrow)

1.2.3 Mach-Zehnder interferometer

This device consist of as two coupling structures connected by two single-mode waveguides with a phase-shift between them (Figure 1.9). Light beam is split by the first coupling structure into two separated branches that must not interact with each other until they are combined again by the second coupling structure. The typical power division ratio used in both coupling structures is 3 dB.

Figure 1.9: Schematic top-view of a Mach-Zehnder interferometer. It is composed by 2 coupler and 2 single mode waveguide with a phase-shift represented in black $\Delta\phi$

If these two branches are identical, the input power must be symmetrically divided in each output waveguide. Since these branches are designed in order to cause intentionally a relative phase shift between light traveling through them, the combined power in the second coupler is modulated and hence the output power ratio changes [20]. The phase difference and the output at one of the two exits can be described as:

$$\Delta\phi = \frac{2\pi}{\lambda_{eff}}d \quad P_{out} = P_i(1 + \cos(\Delta\phi)) \quad (1.12)$$

Where $\Delta\phi$ is the phase shift and $P_{out}/2$ is the power of the output. There is a π delay

between outputs.

Looking the Equation (1.12), it can be observed that light can be switched from one port to the other by changing the phase shift $\Delta\phi = \Delta(d \cdot n)$ from zero to π (Figure 1.10). This could be accomplished by enlarging the optical length d or using a different material (different refractive index) in the phase tuning region of one of the two branches, so the light propagates at different velocities in each Branch.

Figure 1.10: *Normalized output power vs normalized $\Delta\phi$.*

Because of these properties, MZI are exploited as a chemical sensor in biotechnology applications by directly exposing the sensing region to molecules that interacts with the evanescent field. Beside this recent usage, MZI are commonly used also as power splitters and switches in conventional optical circuitry [26].

1.2.4 Tunable PIC

A tunable PIC is an optical integrated device that can change the working function with an external change. In terms of the electric field, the electromagnetic wave can be described as:

$$\vec{E} = \vec{E}_0 \exp\left(\frac{-\alpha L}{2}\right) \exp\left(\frac{j2\pi nL}{\lambda}\right) \quad (1.13)$$

So, it is possible to change the amplitude changing α (electro-absorption) or the phase, changing the refractive index n (electro-refraction). Both can be used to make an intensity modulator. The electro-absorption can be used directly; with electro-refraction it is necessary to combine phase change with an interferometer.

Non-linear effects must be used to modify the electro-absorption. Electro-refraction, typically, is modulated with temperature. The electro-refraction effect can be also used to tune filters like ring resonators.

1.3 Liquid crystals

1.3.1 An introduction to liquid crystals

A Liquid crystal (LC) is a state of the matter between solid and liquid with common properties of both; their molecules present a crystalline order but they can flow. In any case, LC is a mesogen with unusual dependence on temperature, the components of the mixture and the concentration. The LC exists in many mesophases: calamitic LCs, for example, show phases such as nematic, cholesteric and smectic.

There are three main LC kinds: *thermotropic*, *lyotropic* and *polymeric*. Thermotropics are the most common studied LCs, ordinarily used in displays. The most employed LCs in practical applications are usually elongated molecules called *calamitic*, typically represented as rods (Figure 1.11). Calamitic LCs adopt different phases as mentioned above; however, nearly all the applications employ nematic phase, a phase having orientational, but no positional, order.

Figure 1.11: Calamitic LC molecule with (right) and without (left) externally applied electric field

Similar to crystalline solids of non-center symmetric systems, liquid crystals have a large variety of properties showing anisotropy: dielectric permittivity, magnetic susceptibility, optical birefringence and viscosity. The rod-like LC molecules are uniaxial, which means that they have two equal ordinary axes and one extraordinary axis. They are also referred as perpendicular and parallel axes, respectively, that determine the LC anisotropy. Associated to each magnitude x , the corresponding anisotropy is defined as the difference $\Delta x = x_{\parallel} - x_{\perp}$. The definition determines the sign of the anisotropy. The magnitude x can be the refractive index n , the permittivity ε , the magnetic susceptibility χ , the viscosity ν or any other. Calamitic LCs are customarily magnetically and optically positive with $\Delta\chi$ and $\Delta n > 0$. However, $\Delta\varepsilon$ may be positive or negative.

From a practical point of view, LCs are interesting and useful materials inasmuch they can be reoriented with electric and magnetic fields (Figure 1.11); the reorientation having consequences on the optical properties of the material. Moreover their phases can be thermally tuned. The main phases of a uniaxial thermotropic LC are: *nematic* with orientational but no positional order, as mentioned above, and *smectic* with orientational and positional order. If the LC is made of chiral molecules, then the phases are chiral, producing new interesting effects:

some chiral smectic phases become ferroelectric, while chiral nematic phases spontaneously adopt a helical configuration that actually behaves as a 1D photonic crystal (Bragg reflector). Chiral nematic LCs were formerly known as cholesteric LCs, and deserve to be considered a separate family for their optical properties, phase sequence and, above all, their applications. These phases are briefly commented below.

1.3.1.1 Nematic and smectic LCs

Nematic phase is the most common phase in LCs. Nematic liquid crystals are aggregates of organic molecules whose orientation is determined by an order parameter S of intermediate value between liquids (0) and solids (1). This parameter determines the distribution of the molecules around an average direction called the director $\vec{n}$, and is defined as:

$$S = \frac{\langle 3 \cos^2 \theta - 1 \rangle}{2} \quad (1.14)$$

where θ is the angle between the liquid-crystal molecular axis and the local director. This is the simplest requirement in terms of orientation, and gives rise to the nematic phase [27, 28].

In nematic phase the calamitic molecules have no positional order. However, they tend to self-align to have long-range directional order with their long axes roughly parallel to the director vector $\vec{n}$. Thus, the molecules are free to flow and their center of mass positions are randomly distributed as in a liquid.

If another degree of freedom in the position of the molecules is restricted, then the smectic phase occurs, characterized by the distribution of molecules in layers, *i.e.*, featuring positional order. Ideally the molecules adopt plane-parallel geometry, with or without some angular tilt with respect to the layer normal. Smectics are much more viscous than nematics, severely limiting their applicability except in special cases like ferroelectrics, where the spontaneous polarization can be used to drive the material, achieving switching times in the μs range, being one or two orders of magnitude faster than nematics. Figure 1.12 shows differences between thermotropic LC phases

1.3.1.2 Cholesteric LCs and blue phases

If the nematic liquid crystal is made of, or contains, chiral molecules, they will present optical isomerism, and a characteristic helical rotation in the nematic distribution (Figure 1.12), resulting in the so-called cholesteric phase [30]. Specific molecular placement, gives to the cholesteric phase the property of rotating the plane of polarized light inducing a circular phase delay that causes the reflection one component of circular polarized light if non-polarized light impinges on a cholesteric liquid crystal. The reflection is restricted to a wavelength band where, the material effectively behaves as a 1D photonic crystal. The distance between two regions where molecules are parallel to each other is called half pitch. Hence, the pitch refers to the distance over which the LC molecules undergo a full 360° twist. The pitch typically changes when the temperature is altered, when an external electric field is applied or when other molecules are added to the LC host. If the pitch is in the order of the impinging light, a Bragg reflection occurs. In liquid crystal argot, when a LC phase have chiral components

Figure 1.12: *Thermotropic LCs phases [29].*

an asterisk is added to the nomenclature; for example a chiral nematic phase (cholesteric) is named N^* and a chiral smectic C phase is SmC^* .

Blue Phases (BP) are liquid crystal mesophases that show up in the temperature range between the cholesteric phase and the isotropic phase (Figure 1.13). Although blue phases were first detected in 1888 [31], their complex structure began to be fully understood in 80s and 90s [32, 33, 34, 35]. There are three blue phases in a typically very narrow range of temperatures, BPI, BP II and BP III. BPI and BP II, are known to exhibit cubic structures while BP III, also known as the foggy phase, is isotropic. The three blue phases differ in the amount of order they possess and the structures that the chiral molecules form. In chiral nematics, molecules lie in quasi-nematic layers and rotate when going from one layer to the next. However, the free energy is actually lower if the molecules twist in two dimensions simultaneously. This leads to the formation of the double twist cylinder, where all the molecules rotate about a central axis.

Figure 1.13: *BP are structured by intersecting orthogonally double twist cylinder [29].*

Blue phases present Bragg reflections associated with the photonic band-gap of chiral LCs. When BPs are seen under a microscope, different colors (predominantly blue, hence the name) are reflected in different regions depending upon both the spacing of reflective planes and the angle between the light and the reflective planes. BPs are self-organizing materials, not requiring any surface conditioning to align. Until now few studies have been carried out on this matter. Recently it has been developed a polarization-independent bistable light valve in a photonic crystal fiber [36]. It has been also reported that blue phase as fiber waveguiding cores are optically isotropic, polarization insensitive and relatively low-loss [37].

1.4 Tunable Lenses

The electro-optical properties of LC materials make them suitable to create many different optical devices, with the advantage that they can be tuned to change their optical behavior in real time by the application of a voltage difference in a range of frequencies where the LC has a response. Modal lenses are a good example of application of these LC devices. One of the goals of this work is to manufacture an array of Modal Liquid Crystal Lenses (MLCL) in the microscale *i.e.*, micro-lenses.

Figure 1.14: *Lens effect for solid lens (left) and modal lens (right).*

MLCLs are devices that affect the optical path of the light coming through, exactly the same as a glass lens would do (Figure 1.14). Moreover they are tunable from infinite to a minimum focal length, that depends on the size and thickness of the lens as well as on the birefringence, as seen in the equation (1.15), where F is focal distance, r is radius of the lens, d is the thickness of the cell and (Δn) the birefringence.

$$F = \frac{r^2}{2 \cdot \Delta n \cdot d} \quad (1.15)$$

The initial and simplest devices for microlenses arrays have the following symmetrical structure: a glass substrate with an electrode, then an alignment layer for the LC and finally the LC. All the layers in this structure are transparent. In order to achieve the lens effect, the LC cell or sandwich should not be uniform, so the electrode is patterned in at least one of the surfaces and may have extra layers, depending on the approach to get the lens effect.

1.5 Liquid crystal optical vortices

1.5.1 Spin and orbital angular momentum

Light rotation can be described by two ways: the first is related with the dynamic rotation of light rays around the main beam axis, and the second is the dynamic rotation of the electric and magnetic fields around the propagation direction. These two rotations are associated with two forms of angular momentum, namely orbital and spin angular momentum.

The orbital angular momentum OAM of a light beam is a component of angular momentum dependent on the field spatial distribution. It is independent of the state of polarization SOP and hence unrelated to the spin of individual photons [38]. OAM can be divided into internal and external OAM. The internal OAM is related to a helical or twisted wavefront (it is origin-independent). The external OAM is obtained as cross product of the light beam position (center of the beam) and its total linear momentum (it is origin-dependent).

An example of OAM appears when a paraxial light beam is in a ‘helical mode’. Helical modes of the electromagnetic field are characterized by a wavefront that is shaped as a helix, with an optical vortex in the center, at the beam axis. The vortex can be defined with a number, called topological charge (l). The topological charge is related to the number of times the light twists in one wavelength. The number is always an integer, and can be positive or negative, depending on the direction of the twist. The higher the number of twists is, the faster the light spins around its axis.

1.5.2 Spiral phase plates and spiral diffractive lenses

Optical vortices have received much attention in the last years [39] because of their applications in particle trapping [40], image processing [41], special phase contrast microscopy [42], and free-space communications [43]. To obtain a field vortex, a spiral phase profile has to be obtained. The spiral phase profile can be expressed as $\exp(jl\theta)$, where l is the topological charge and θ denotes the azimuth angle.

The easiest way to generate an optical vortex is by impinging a laser beam in an external spiral phase element like a computer generated hologram [44], or **Spiral phase plates (SPP)** [45, 46, 47], which have an improved efficiency. The ideal SPP has a continuous surface thickness topology that imposes the desired azimuthal phase. Usually, SPP is manufactured with multilevel quantized phase values, for example: multi-stage vapor deposition process [41] or direct electron-beam writing [48]. Generally high efficiencies can be obtained with these devices; however, the employed wavelength and the topological charge cannot be modified.

SPPs can be implemented as liquid crystal Spatial light modulator (SLM) [49, 50]. These SPPs can be tuned, but they suffer from a limited light efficiency due to the SLM pixelated structure. Each pixel consists of an active region with areas transmitting or reflecting light, and surrounded by opaque areas (dead zones) that contain control electronics and wires. This kind of structure has a drawback: the fill factor (the fraction of the incident intensity that is transmitted or reflected) is given by pixel spacing, and the intensity obtained in the main diffraction order is proportional to the square of the fill factor [51]. Therefore, the interpixels would limit the device performance. In order to avoid these effects, liquid crystal cells with a

special patterned ITO pie structure can be manufactured. The conversion of a Gaussian laser beam into a doughnut beam with efficiencies near 100% can be achieved with these cells [52].

When using liquid crystal cells stacked together larger topological charge numbers are achieved [53]. The generation of optical vortices up to $l=8$ can be achieved but the efficiency is reduced due to the stacking process.

There is a growing interest in developing efficient and programmable devices for the generation and manipulation of vortex beams [54, 55].

Figure 1.15: (a-d) *Spiral phase plate with different topological charges l .* (e) *Diffractive lense.* (f) *SPP with topological charge of $l=4$.* (g,h) *phase only spirals*

The transmittance function of a conventional SPP can be represented as (in polar coordinates):

$$g(\theta) = e^{jl\theta} \quad (1.16)$$

l is the number of 2π phase changes on the circle around the beam axis.

Figure 1.15 (a,b) illustrates how the transmittance function $g(\theta)$ is when $l=1$ and $l=2$. In general, due to the devices limited resolution, only quantized values of the phase modulation are produced, and multilevel SPPs must be considered. Figure 1.15 (c,d) shows the quantized values for $l=1$ and $l=2$ with different 24 levels (The same that it was fabricated in this PhD).

Spiral diffractive lenses (SDL) have been proposed for astronomical applications to allow viewing of a dim stellar object in the region of a much brighter object [56]. They have been manufactured with liquid crystal pixelated displays [50]. However, liquid crystal devices with specific SDL electrode structures have not been reported before.

A subject lately receiving attention is the generation of radially polarized beams [57]. Liquid crystal devices able to generate radially polarized beams have been manufactured [58, 59]. In this work, liquid crystal SPP devices can be used as pseudo-radial beam generators, avoiding

the efficiency losses due to the pixelated device employed in [58]. The word pseudo-radial comes from the fact that the generated beam is not a pure radially polarized beam, but it is accompanied with a spiral phase distribution. The pseudo-radial beam generation was achieved for different wavelengths by properly adjusting the voltage to different pie sectors of the liquid crystal cell and will be explained in next sections.

1.6 Hybrid devices (PICs+LC)

In order to make electronically active PICs it is necessary to combine the waveguides with electro-optics materials such as LCs.

Interestingly, LCs have been used in integrated optics as early as 1977[60], however due to their slow switching speed (*ms*) the number of applications of LCs is restricted to those where velocity is not an issue. Nonetheless there are many applications of this kind; for example polarization manipulation is often performed with passive devices [61, 62].

Hybrid devices combining a platform (InP, SOI or polymer) with electro-optic materials can have a high efficiency. Various examples have been demonstrated using slot waveguides filled with electro-optic materials [63, 64]. Tuning of waveguides based on liquid crystals has likewise been demonstrated [65, 66, 67].

PICs are not easy to integrate compared to electronics. However, the amount and speed of information that they are capable of transmitting make them a platform on the rise.

To modify the guiding properties of the waveguides, one must know the properties of the materials very well. The properties of the light / matter interaction must also be known. That is why the Thesis seeks to explore the modification of light guidance by modifying the evanescent field. Different forms of waveguides, procedures and materials are investigated in the development of the Thesis. To achieve these milestones new manufacturing processes have been developed and manufactured. Likewise, at least three new PICs devices have been manufactured.

One important part of this Thesis is explore the possibility of integration of LC materials in PIC platforms . The possibility of tuning the refractive index of the materials give the possibility to use the electro-refraction effect to tune filters, splitters, modulators, etc. Below a theoretical approach is introduced.

1.6.1 MMI splitter/modulator

Above in section 1.2.1.3 on page 11, the MMI splitter was presented. The L_π distance was defined like: $L_\pi = \frac{\lambda}{2(n_1 - n_0)}$. The dependence with the electro-refraction effect is marked in red.

When a LC cladding is applied (Figure 1.16), the MMI can work like a splitter (with a small tuning) but also like a modulator if the change in the refractive index of the cladding is enough.

Figure 1.16: *MMI with a LC cladding.*

1.6.2 MZI splitter/modulator

The output power in a MZI is given by $P_{out} = P_i(1 + \cos(\Delta\Phi)) = P_i(1 + \cos(\frac{2\pi}{\lambda_{eff}}d))$, again, the red color mark the dependence with the electro-refraction effect.

It is possible to tune the power in the output tuning the phase delay.

1.6.3 Ring resonator filter

A ring resonator is a frequency filter. The wavelength to filter is given by $\lambda_{res} = n_{eff} \frac{2\pi R}{m}$.

Figure 1.17: *Ring Resonator with a LC cladding.*

It can work like band-stop filter (yellow arrows) and band pass-filter (green arrows) simultaneously. With the electro-refraction effect (Red words in the formula) is possible to tune the frequency/wavelength of resonance (Figure 1.17).

1.7 Outlook of the present work

This work is centered on the manufacturing and characterization of light manipulation devices by refractive index change. The principal emphasis will be on the development and characterization of new device design and manufacturing technologies.

The later chapters will describe the work undertaken during the Thesis.

Chapter 2. Developed technologies for micro- and nano-fabrication: is a detailed description of the state of the art of the manufacturing process in the CEMDATIC clean room installations, with special emphasis in the machines developed to increase the resolution. In this chapter developed techniques of laser ablation, laser direct write (LDW) lithography and nanoimprint NIL are described.

Chapter 3. Microstructured devices: is dedicated to the design fabrication and characterization of microstructured devices like *microlenses arrays* and *reconfigurable optical vortex*. Some of the devices and techniques employed in this chapter are suitable to be used with small changes in PICs and tunable PICs described in the Chapter 4 *i.e. light coupling systems*.

Chapter 4. Photonic integrated circuits: describes a novel integration between polymeric waveguides and Liquid crystal (LC). The developed devices are a tunable *multi-mode interference coupler* and a *polarization manipulation PIC*.

Chapter 5. Conclusions and future research lines: shows a brief summary of the most important results and suggestions for future research lines.

Chapter 2

TECHNOLOGIES DEVELOPED FOR MICRO- AND NANO- FABRICATION

The R&D Center CEMDATIC has two clean room facilities, three characterization laboratories, three photonics laboratories, one biological laboratory and a workshop. The space allocated for every task depends on the type of processes being carried out.

Generally the manufacturing starts in one of the clean rooms, and the final device is characterized in a characterization laboratory. The manufacturing techniques used in the Thesis are mainly electronic micro- and nano- fabrication technologies.

In this chapter, three new systems are presented. The new systems are based and adapted from other technological areas to the fabrication of photonics devices.

2.1 Laser ablation for micro-fabrication. (*Developed system*)

A UV laser with a CNC controller has been acquired by CEMDATIC. The device consist of a UV laser (Nd-YAG tripled laser), which is focused onto a 3-axis CNC platform with closed feedback loop for maintaining the optical focus.

The optical setup consists of three optical subsets (Figure 2.1). The first is used to control the power and the quality of the engraving laser beam (**Laser setup**). The second is an IR (780 nm) laser used to monitor the focus (Z axis) when moving in the XY directions (**fixed focus setup**). The third is a visible LED (480 nm) in order to have a live image in a CCD camera (**illumination and microscope**).

The **fixed focus** and the **illumination and microscope** paths are composed by lenses and dichroic splitters or mirrors. The **laser setup** includes lenses to form a perfect Gaussian beam. An adjustable half wave retarder plate together with a polarizing beam-splitter and an optical trap is used to control the power. All the different light paths are focused by a 20x

Figure 2.1: Optics setup between the laser and the 3 axis CNC platform. The engraving laser beam path is shown in purple, the focus beam in red, and the live-image path in yellow.

microscope objective.

The 3-axis CNC controller is a *XPS MOTION CONTROLLER* from Newport. The precision of the stages is better than $1 \mu m$. In the XY directions the travel range is $160 mm \times 100 mm$ and in the Z direction the travel range is $\sim 10 mm$.

2.1.1 Working principles of laser ablation

Laser ablation is the process of removing material from a surface by irradiating it with a laser beam. At low laser flux, the material is heated by the absorbed laser energy and evaporates or sublimates. At high laser flux, the material is typically converted into a plasma. Usually, laser ablation refers to removing material with a pulsed laser, but it is possible to ablate material with a continuous wave laser beam if the laser intensity is high enough [68].

The depth over which the laser energy is absorbed, and thus the amount of material removed by a single laser pulse, depends on the material's optical and thermal properties, the laser wavelength and the pulse length. The total mass ablated from the target per laser pulse is usually referred to as ablation rate. The laser beam scanning speed and the overlap of successive scanning lines influence significantly the ablation process.[69]

Figure 2.2: *Laser system picture*

Laser pulse duration may vary from milliseconds to femtoseconds and the flux may likewise vary some tens orders of magnitude. Since both parameters can be precisely controlled, this makes laser ablation very flexible for both research and industrial applications.

The available laser system in the CEMDATIC has 300 mW output power at 50 kHz and the wavelength is 355 nm (tripled Nd-YAG laser) with a pulse width lower than 15 ns , it is working in the repetition frequency range between 20 kHz to 300 kHz , the power per pulse is reduced as the frequency is increased.

2.1.2 Fabrication protocol for laser ablation micro-fabrication

A protocol was developed in order to make micro-structures by laser ablation. It was employed to generate microscopic electrode patterns on ITO on glass and organic waveguide structures.

There are two ways to eliminate material; one is top ablation (front scribing) and the second one is bottom ablation (back scribing). The most suitable method depends of the material to eliminate and if the substrate material is UV transparent.

The parameters that are explained below were optimized to obtain the best results on the ITO covered glass used in the laboratory.

2.1.2.1 Substrate preparation

All glass employed in this work is ultra-flat polished float glass featuring a coating of indium tin oxide ITO on one of the surfaces.

Glass cutting

Glass is cut into pieces of the required size for further processing (Figure 2.3).

Figure 2.3: *Cutting substrate by hand.*

- Identify the ITO side with a multimeter
- Cut the substrates into the needed size with glass cutter on the side without ITO.
- Place the substrates into the holder in a systematic manner (*i.e.* always make ITO side face to you on the right hand).

Glass cleaning

Figure 2.4: *Ultrasonic bath.*

- Place the substrates into a ultrasonic bath with distilled water and KH-7, for 20 minutes (Figure 2.4).

- Wash the substrates with distilled water.
- Transfer the substrates into another ultrasonic bath with distilled water at 50 °C for 15 minutes.
- Wash glasses with distilled water.
- Transfer the substrates into the third ultrasonic bath with distilled water at room temperature, for 15 minutes.
- Place the substrates in the oven for more than 30 minutes at 200 °C.

Surface conditioning using short wavelength UV

The ultra violet oven UVO surface treatment is a photo-sensitized oxidation process ((Figure 2.5)). Absorption of short-wavelength UV radiation eliminates contaminant molecules of resins, grease from human skin , solvent residues, silicone oils etc. Atomic oxygen is generated when O_2 is dissociated by the energy associated to the wavelength 184.9 nm and O_3 by 253.7 nm UV. The 253.7 nm UV radiation is absorbed by most hydrocarbons and also by ozone [70]. The excited contaminants react with atomic oxygen to form volatile compounds

Figure 2.5: *UVO cleaner.*

which desorb from the surface. By placing pre-cleaned samples within a few millimeters of ozone producing UV source, such as the low pressure mercury vapor grid lamp, near atomically clean surfaces can be achieved. Before depositing any testing material onto the ITO face, they have to be UVO-exposed to improve the adhesion in spin-coating process, as this process not only eliminates impurities, but also oxidizes the surface fully, and leave very reactive surface.

- Expose the inner side of the glasses at least 10 min in the UVO.

2.1.2.2 Material spin-coating

A spinner is used for spin-coating of thin films from solutions of polymers in organic solvents and other dispersions. It has been used here to deposit different photoresists that allow to create the waveguides. Also, it is used for depositing alignment layers for rubbing process as described below in section 3.3.1.3 on page 69.

A Laurell WS-650Mz-23NPP spinner (Figure 2.6) is provided in the clean room, with rotation speeds up to 12000 rpm, controllable with 1 rpm increments. The holder diameter allows for the treatment of glasses up to 6" diameter (about 150 mm).

Figure 2.6: *Detail of the spinner used in the clean room.*

The final thickness of the deposited layer depends upon time and turning speed of the head. Viscosity is also an important parameter to take into account. The sample must be held tight to the spinner's head, for which a joint is placed between the platform and a vacuum chuck. This vacuum chuck goes inside the rotation axis which rotate over a hollow cylinder on which the vacuum is made.

The parameters to obtain the required size are taken from the materials data-sheet. In case that the thickness required is not in the data-sheet, a series of experiments is done to obtain the relationship between the spin-coating parameters and the thickness.

2.1.2.3 Hardening Material (UV curing / Hard Bake)

After the spin-coating process, the material remains liquid, so prior to the laser ablation a hardening of material is needed. The procedure to harden depends on the material. Usually, if the material is UV curable, it is needed to remove the solvent by temperature and a UV bake

is applied to polymerize. After the polymerization a hard bake in the oven makes the material stable at room temperature.

2.1.2.4 Glass positioning in the laser system

The substrate with the target thin film of material is placed in the xyz CNC stage, where the laser is focused in order to ablate the material. The substrate is fixed by Kapton tape to the xyz stage before running the program.

2.1.2.5 Running the program to executed the design

The different parameters of the laser system depend on the target material. Anyhow the process can be summarize:

1. Laser setup.
 - (a) 5A of current.
 - (b) 50 *kHz* repetition frequency.
 - (c) Check the stand by.
 - (d) Choose the power that is needed for the target material.
2. Choose the origin with *"Set relative home"* button.
3. Focus the laser on the correct face.
4. Run the program.
5. When the program is finished. Check the problematic regions to be sure that it is ok.
6. Remove the substrate from the laser.

The CEMDATIC has a continuously updated internal manual where the individual steps needed to use the ablation laser system are explained.

The ablation laser system was originally designed for ITO ablation. Currently the laser is used for many materials and other not ablation applications. During the development of the Thesis another materials were used and a complete protocol for laser direct write lithography is developed (section 2.2 on page 35).

For laser ablation two main research lines were pursued. The first is mask-less micro-fabrication of electrodes. The second is mask-less fabrication of waveguides.

2.1.3 Electrodes fabrication

One of the main research areas in the CEMDATIC is non display liquid crystal devices. For this complex electrodes are often designed. The design of these electrodes is constantly changing depending on the application and fine-tuning processes. Employing conventional photo-lithographic masks for every short lived design is both costly and time consuming. The

developed laser ablation system for electrodes fabrication allows for new electrodes to be realized in days rather than weeks at a much reduced cost.

The theoretical average power of the laser is 300 mW. The density power (D) can be calculated knowing the diameter of the spot ($\phi = 2 \mu\text{m}$) and the velocity of the movement v according to the equation:

$$D = \frac{P \cdot \phi}{v \cdot A} \quad (2.1)$$

where $A = \pi R^2$.

An ultra-flat glass substrate with coated ITO layer is used for this application. Only prior cleaning is needed. As mentioned before two kinds of ablation can be done: Top ablation (front scribing) and bottom ablation (back scribing) (through the glass).

The optimized parameter for the top ablation is 18% of the power and a velocity of 10 mm/s (54 mW @ 50 kHz $\Rightarrow$ 343.7 J/cm² theoretically. However as shown in Figure 2.1 on page 26, several lenses and mirrors are used in the optics setup make that this estimation is only an approximation).

The optimized parameter for the bottom ablation is 5% of power and a velocity of 10 mm/s (15 mW @ 50 kHz $\Rightarrow$ 95.5 J/cm² theoretical estimation).

There is a linear relation between power and velocity. So it is possible to increase velocity and power in a linear way. However, exceeding 10 mm/s is not recommended since the pulse separation will start to be noticeable (at 10 mm/s and 50 kHz, there are only five pulses per μm).

Figure 2.7: Atomic Force Microscopy (AFM) figures of the laser ablation over ITO. Images show two cuts with a distance between center of 10 μm . Top image show a upper laser ablation. Bottom image show back scribing.

Figures 2.7 show two AFM images. Two cuts with the laser separated 10 μm are seen. The top image shows a front scribing. A lot of power is needed to ensure that all the ITO is eliminated, and short-cuts are avoided. The applied power ablates the underlying glass too. The ablated zone is large and limits a dense pattern. The bottom image show a back scribing, the power is 3 times lower that in the upper laser ablation, the eliminated pattern is close to

a square signal and only the ITO is eliminated. Unless a very short chemical attack is applied afterward, small, nano-sized, agglomerates of ITO are deposited onto the cut.

Figure 2.8: Profiles of the AFM images for the two methods of laser ablation; (Black) front scribing; (Red) back scribing.

Figure 2.8 shows the AFM profiles for the two methods of laser ablation. The reference 0 is on the top part of the ITO layer. The plots show remarkable differences between the methods. In the bottom laser ablate (red line) the width of the ablation is $1.83 \pm 0.05 \mu m$ and the thickness is $0.13 \pm 0.02 \mu m$. The top ablation width measures $8.0 \pm 0.3 \mu m$ and the thickness is $0.6 \pm 0.2 \mu m$.

2.1.4 Waveguides fabrication

This Thesis is focused on micro- and nano- fabrication of waveguides. The first approach was to make waveguides by laser ablation.

Figure 2.9: Principle of a waveguide make by laser ablation.

The idea is to spin-coat and selectively eliminate part of the core layer making the structures needed for the photonic circuits (Figure 2.9). Based on that, the first waveguides were made.

Figure 2.10 shows a waveguide made by laser ablation. The right picture show the waveguide guiding red light. Light was coupled to the waveguide through an optical fiber. Coupling was done by placing a drop of SU-8 onto one of the ends of the previously prepared waveguide. The optical fiber tip was immersed in this drop and the SU-8 droplet was cured keeping the

Figure 2.10: *Left: Transmission microscope image of the end of a multimode SU-8 waveguide coming from the left. The image shows the edges of both the deposited SU-8 and the glass substrate. The two ablation lines limiting the waveguide are visible both in the SU-8 and the glass. A third ablation line eliminated the edge-shaped end of waveguide; Right: Guiding the light. The end of the waveguide, at the end of the SU-8, is clearly visible as a straight line, the glass edge is illuminated by scattered light.*

fiber pointing to the waveguide edge. No attempt was made to optimize light coupling since this topic was beyond the objectives of this work. Nevertheless, the coupling was poor and it showed little light transfer between the optical fiber and the waveguide.

Figure 2.11: *Waveguide made using laser ablation of NOA-81. The waveguide consists in a Y-junction where one waveguide is split into two waveguides which are bent 180°. It is shown the connection of an optical fiber coupled to the waveguide with a NOA-81 drop (the bright point). A lot of light cannot follow the curve and goes straight (losses) but some light still inside of the waveguide and it is guided to the end of the waveguide.*

These SU-8 waveguides were manufactured in several patterns, including curved shapes. Using NOA-81 as photoresist the coupling step was easier (the thickness is 10 times bigger and close to that of the fiber core), but there were still losses in the guiding (Figure 2.11).

The waveguides that were obtained with laser ablation methods may find applications, but they do not have the same quality as other waveguides manufactured in this Thesis. Ablation is used for some parts in the waveguides fabrication but not for the waveguide structure.

2.2 Laser direct write (LDW) lithography for micro-fabrication. (*Developed system*)

In Laser direct write (LDW) lithography, the radiation used to expose the photoresist is focused into a narrow beam, rather than a wide beam exposing a whole mask [71]. The beam writes directly the image onto the photoresist. A similar method, developed by MYCRONIC and Heidelberg Instruments Mikrotechnik, is to scan a programmable reflective photomask, and image it onto the photoresist [72].

For improved image resolution, ultraviolet light, which has a shorter wavelength than visible light, is used to achieve resolution down to around 100 nm. The main optical mask-less lithography systems in use today are developed for generating photo-masks for the semiconductor and LCD industries. Dual photon techniques using a combination of two optical beams of different wavelengths have resulted in 9 nm feature size and 52 nm pitch [73].

2.2.1 LDW protocol

The fabrication protocol is very close to that of laser ablation. Basically, the steps are the same as described in section 2.1.2 on page 27 adding a step of development before the hardening step. It can be summarized as follows

- Substrate preparation (section 2.1.2.1 on page 27).
- Spin-coat with the target material
- Soft-Bake (if necessary)
- Place the substrate in the laser
- LDW (UV exposure)
- Post-Bake (if necessary)
- **Develop** Developing is a process with chemical agent, *the developer*, that principally removes exposed areas of positive photoresists and unexposed areas of negative photoresists. However the time of developing must be carefully controlled, since the susceptibility of the exposed and the unexposed area towards the developer is very similar, and thus over-, and under-developing is often taking place.

The material data-sheet provides the time needed to develop every material. The procedure is simple. Immerse the material in the developer and wait the time. After that, remove the developer.

- Hard bake. (Optional)

The spin-coating parameters are also given by the material data-sheet, as the soft-bake/post-bake/hard-bake conditions.

The LDW parameters can be calculated based on the material data-sheet for the UV-power density emitted by the curing source. For example, the SU-8 need 100 mJ/cm^2 . In the laser setup, when fitted with a 2 mm diaphragm, $2.8 \% @ 300 \text{ kHz}$ power and a velocity of 30 mm/s , was found to be the optimal conditions for curing the SU-8. Thus, under the estimate of an approximately uniform spot-size of $\phi = 2 \text{ }\mu\text{m}$ lead to the conclusion that the transmitted power of the system is about 1.68 mW (See equation (2.1) on page 32).

2.2.2 LDW characterization and results

Different materials were cured using LDW lithography. The best resolution achieved by CEM-DATIC members is $1 \text{ }\mu\text{m}$. Nowadays, the CNC laser only works in a *vector mode* but we are working towards *raster mode*. In the second quarter of 2018 the *raster mode* will be ready hopefully.

Figure 2.12: *SU-8 circles fabricated by LDW lithography method.*

Figure 2.12 shows different circles made by LDW lithography. The target material in this case was SU-8. It is possible to appreciate in the upper part of the circles a widening in every circle. It is due to the start and ending point of the laser curing where the speed of the CNC stage is reduced, and thus material receive excess power.

2.3 Nanoimprint Lithography. (Developed system)

Nanoimprint Lithography NIL was chosen as nano-fabrication method because it is a simple lithographic process with low cost, high throughput and high resolution.

Since commercial NIL instruments are expensive and provide only Photo- **or** Thermoplastic-Nanoimprint Lithography (P-NIL or T-NIL), it was decided to fabricate an instrument that could use both kinds of materials, at a reduced cost.

Figure 2.13: *Nanoimprint machine developed in the CEMDATIC R&D CENTER*

The device had to be **UV transparent** since this is needed for the P-NIL approach as UV is used to cure almost all photo-curable polymers.

Another requirement is **temperature control** which is needed for T-NIL where thermo-curable polymers are used. In all cases thermal control is important in order to avoid, or

exploit, thermal effects such as dilation or speed of curing.

Other important aspect to take into consideration is **pressure control**. This is needed to check the pressure between the negative mold and the material to be copied.

Two different devices were designed to get a negative mold and copies (Detailed information about the devices can be found in appendix C on page 137):

1. **The Mold-maker.-** was designed to make a negative mold of the selected pattern. The machine is composed by three different parts: A wall, a base with rods distributed circularly and a interchangeable central master support where the master pattern is inserted.
2. **The Printer.-** is the machine that makes copies from the negative mold. In the *Printer*, vacuum (pressure) and temperature are controlled. The *Printer* is composed by two parts, one part is the sample holder and the other part is the support of the negative mold. The device is connected to a temperature and pressure control box.

See figure C.4 on page 140 for photos of the devices.

The whole process for NIL procedure is shown in the Figure 2.14. In summary the steps are:

1. Make a negative PDMS mold of the pattern with the *Mold-maker*.
2. Imprint the negative mold onto the spin deposited material on the substrate with the *Printer*.
3. Release the PDMS and the nano copy is done.

Figure 2.14: Illustrative sketch of the steps for the replication of the master pattern onto a UV curable polymer. (Top images) The use of the *Mold-maker* for producing the negative mold. (Bottom images) The use of the *Printer* for creating the final copy.

2.3.1 Manufacturing of negative mold made of Polydimethylsiloxane (PDMS)

The initial process developed by the group has been modified and optimized several times depending on the quality of the resulting samples. About 300 samples of five different materials (NOA-81, OrmoStamp, PMMA, Photoresist, SU-8) have been generated, leading to the procedure shown below. Different parameters like polymer density in the PDMS mold, related to the mold stiffness, has been taken into account and optimized.

2.3.1.1 Procedure for Preparation, Homogenization and Degassing of PDMS

This step aims at getting a negative mold of an appropriate viscosity, able to be cured and to successfully copy the pattern.

- First, mix in a 100 *ml* beaker the two liquid ingredients to make PDMS in a 9:1 w:w proportion, polymer and elastomer respectively.
- Secondly, introduce a magnet in the beaker and agitate the mixture at gently 70 rpm (avoiding resonances and keeping the magnet rotating) during 10 *min* at room temperature to homogenize it. It was proven that skipping this step lead to the elastomer not being uniformly distributed, which resulted in sticky areas in the cured PDMS mold, leading to imperfections rendering the negative mold useless and, in the worst case, contaminate the master pattern and ruin it.
- Finally, degas the liquid PDMS in a vacuum hood using the vacuum pump.

2.3.1.2 Procedure for Deposition of mono-layer release agent: Perfluorodecyltrichlorosilane (FDTS)

This step is needed to facilitate unmolding after PDMS is curing, reducing the risk of breaking the negative mold and/or ruining the master pattern. In the literature [74] there are some ways of doing it, though we adapt the procedure to the following accelerated approach:

- First, the master pattern with the patterned surface pointing upwards is placed in the master support. The *Mold-maker* is introduced in the vacuum hood.
- Then, the open bottle of (FDTS) is placed inside the vacuum hood as well.
- After this, the atmospheric air in the hood is substituted by nitrogen through pumping, and after 30 *s*, the atmosphere will be made of the FDTS and nitrogen. A pressure of 0.8 *mbar* is maintained during 10 *min* resulting in the deposition of a mono-layer of FDTS onto the master.

2.3.1.3 Degassing

Degassing is performed to avoid air bubbles than can lead to defects in the copied pattern.

Figure 2.15: *PDMS out gassing inside vacuum hood.*

- Pour the PDMS into the mono-layer-FDTS-covered *Mold-maker*, start deposition in the center at the patterned master to avoid as many extra bubbles as possible.
- Then, degas by gently applying vacuum. Avoid a vacuum exceeding 0.4 *mbar*, even if some bubbles are still present in the PDMS; since at higher vacuum the air bubbles may flow downwards and dislocate the master pattern holder, tilt the master pattern and as a result ruin the future copies made with the negative mold.

2.3.1.4 PDMS hardening and unmolding

Next thing to do is to harden/cure the negative mold to get a solid negative pattern that can imprint the original positive pattern onto different target materials. To harden the PDMS we only need to wait, but in order to accelerate the process, the temperature can be increased, as described below.

1. Introduce the PDMS-covered *Mold-maker* in the oven at 150 °C during 20 *min*, or leave it 2 days at room temperature. It is very important to follow one of these two procedures, since if the curing time is not long enough, the negative mold will be not cured and will remain sticky, and when separating the PDMS negative mold from the master pattern, the PDMS will contaminate the master pattern and render it useless.
2. When the *Mold-maker* is cold enough to handle detach the negative mold of PDMS

Figure 2.16: *PDMS unmolding process. a) and b) A 1€ coin is used to release the master support from the Mold-maker base. c) The PDMS mold is released from the Mold-maker base using a screwdriver or similar tool. d) The master support is released using a 3-D printed plunger leaving the final mold with the master e). Eventually the master pattern has to be removed gently using a lancet and tweezers.*

from the master pattern, with the help of a screwdriver or a similar tool, a 1€-coin and a special 3D-printed utensil (Figure 2.16).

3. With the help of a lancet separate carefully the master pattern from the PDMS mold.
4. As the last step, before using the negative mold to imprint the copied pattern, the rough edges have to be removed with the cutter; otherwise these edges prevent the pattern to be fully transferred.
5. In order to be able repeat the process, before making another negative mold, the *Mold-maker* has to be cleaned from rests of PDMS, and if the instructions were accurately followed, there should not be any PDMS stuck to the master pattern.

2.3.2 Copying procedure

In this section the copying of the negative PDMS mold is explained.

2.3.2.1 Substrate preparation

The procedure to prepare the substrate is detailed above in section 2.1.2.1 on page 27. It can be summarized as follows:

1. Cut the substrate.
2. Clean the substrate.
3. Treat the substrate by UVO.

2.3.2.2 Deposit material by spin-coating

The spin-coating procedure is explained in section 2.1.2.2 on page 30.

The parameters used in the spin-coating deposition of each material are listed in Table 2.1

Table 2.1: *Spinner parameters*

Material	Weight (mg)	Step 1 parameters	Step 2 parameters
NOA-81	5.5	333 [r.p.m/s], 1000 [r.p.m], 3 s	500 [r.p.m/s], 2500 [r.p.m], 27 s
OrmoStamp	0.8	333 [r.p.m/s], 1000 r.p.m, 3 s	500 r.p.m/s, 2500 r.p.m], 27 s
PMMA	3.9	500 r.p.m/s, 3500 r.p.m, 30 s	-
Photoresist	1.0	4500 r.p.m, 30 s	-
SU-8	1.0	3500 r.p.m, 30 s	-

2.3.2.3 Copying from PDMS mold

In this step the negative pattern is imprinted onto the target polymeric material, reproducing the original pattern. Once the target material is uniformly deposited on the upper surface of the glass, the sample is placed in the central part of the *Printer* and fixed by vacuum. For all tested materials, air removal has to be done by starting at a few *mbar* vacuum increasing it slowly to 110 *mbar*; in this way bubbles between the testing material and the PDMS mold are avoided.

- Remove air from the inner chamber of the *Printer* from zero slowly to 110 *mbar* (making vacuum) until the negative mold imprints the material.

2.3.2.4 Curing of the tested materials

Curing is a necessary step, since just imprinting the negative mold on the polymeric material is not going to make the positive pattern permanent. Hence, while the tested material is in contact with the mold, it has to be cured. Each polymeric material has its own way to be cured:

Figure 2.17: *Curing by UV light (left) and curing by temperature (right).*

- **NOA-81, SU-8 and OrmoStamp:** All of these materials are photo curable, and follow the same protocol:

1. Introduce the *Printer* in the UV light chamber (commercial fingernail curing oven) for 10 *min*.
2. Withdraw the *Printer* from the UV light oven.
3. Let air into the vacuum chamber.
4. Open the *Printer*.
5. Release the cured, patterned polymer-covered-glass sample.

- **Photoresist** : This material is thermo-curable, so the steps to cure it are:

1. Raise the temperature to 90 °C and maintain it for 5 *min* until the materials is cured.
2. Decrease the temperature to 30 °C.
3. Let air into the vacuum chamber.
4. Release the cured, patterned polymer-covered-glass sample.

- **PMMA:** Since this material is thermoplastic:

1. Raise the temperature to 130 °C and maintain it for 1 *min*.
2. Decrease the temperature to 40 °C slowly (just remove the power to the heater).
3. Let air into the vacuum chamber.
4. Release the cured, patterned polymer-covered-glass sample.

The whole process is shown in the Figure 2.14.

2.3.3 Characterization protocols for NIL

Thickness control and a surface characterization were done on every sample. Three different measurements of the thickness and of the surface were done. A profilometry measurement was done with a profilometer in three different regions of the sample. Another three measures of the surface were done with an Atomic Force Microscopy (AFM).

The protocols used to elaborate the measures are explained below in the next sections.

2.3.3.1 Thickness characterization by profilometry

A Dektak 3 Surface Profilometer (Figure 2.18) was used to measure the vertical film thickness and other topographical features, such as film roughness.

A diamond stylus is moved vertically to contact with the sample and then dragged laterally across the sample for a specified distance and specific contact force. The instrument can measure small surface variations in vertical stylus displacements as a function of position.

Figure 2.18: *Dektak 3 Surface Profilometer*

The Dektak profilometer can measure small vertical features, in theory as small as 10 nm and can reach 65 μm of resolution. The height position of the diamond stylus generates an analog signal which is analyzed and displayed. The radius of the diamond stylus is 12.5 μm . The horizontal resolution is controlled by the scan speed [75].

A video camera allows for manual placement of the stylus.

The procedure to measure a sample is simple:

- Turn on the computer and the profilometer.

- Situate the sample on the profilometer base and place it with the help of the camera.
- Measure.

2.3.3.2 Copied surface characterization by atomic force microscopy

The copied surface is characterized by atomic force microscopy AFM. The used AFM was *Agilent Technologies 5500 Scanning Probe Microscope*.

AFM [76] is a microscope that can be used for non-conductive samples and have a lot of application fields like:

- Microelectronic.
- Materials science.
- Tribology.
- Chemistry.
- Biology.

The AFM principle of operation is based on the forces (attraction and repulsion) between the atoms of the tip and the sample (Figure 2.19). It provides a topographic image with a resolution:

- Z resolution $< 1 \text{ nm}$
- XY resolution: $2\text{-}10 \text{ nm}$
- XY scan range: 0.25 nm (single line) - $80 \mu\text{m}$

The movement of the cantilever is detected by using a laser that is reflected by the cantilever. The reflection is detected by four photodetectors. A schematic image of the operation of AFM is shown in Figure 2.20.

The AFM may operate in different modes:

- Contact
- AC Modes:
 - Tapping
 - Non contact.
 - Force modulation

Figure 2.19: Representation of the forces between two atoms [77]

Figure 2.20: *Left: Laser reflection scheme; Right: Photodetector logic to determine the position [78]*

Only the Contact and the Tapping modes were used to measure the samples; These modes are explained in detail.

• Contact mode

In this mode of operation the tip is touching the sample. The AFM tip is attached to the end of a cantilever with a low spring constant. The tip makes gentle contact with the sample, exerting less than $1 \mu N$ force on the sample. The cantilever deflection “follows” the topography of the sample generating a surface topography. The deflection module measures deflections in the nm range.

There are two operation modes in contact mode:

- **Constant height mode.-** where the height of the probe over the sample is kept constant as it scans. For small cantilever deflection ($< 500 nm$) on hard surfaces, the photo detector “error” (*i.e.* deflection) signal is used to generate an image that is sensitive to small changes in topography, though actual topographic information is not obtained [79].
- **Constant force mode.-** where the photo detector “error” signal is used as the input to the feedback circuit which controls the z-height. Keeping the cantilever deflection and thus the force applied constant to the sample constant, the output of the feedback circuit generates the topography image [80].

Constant force mode is more used than constant height mode as it has a larger dynamic z-range. However, the scanning speed is limited by the feedback circuit response. The

resolution is lower than constant height mode, due to inherent noise in the piezoelectric feedback circuit.

To measure in contact mode with the AFM, the following steps in the “PICOVIEW” software are performed:

1. Switch the electronic controller to “Contact mode”.
2. Select in the PICOVIEW software contact mode operation mode.
3. Move the laser to get an intensity close to 1.
4. Calibrate the photodetectors to get a deflection value $[-1,-0.7]$ and LFM (lateral force microscope) value close to 0.
5. Select the area to measure.
6. Choose the span size.
7. Choose the velocity.
8. Measure.

• Tapping measures

In tapping operating mode the cantilever is driven to resonate in an up/down movement. Since the oscillations are made in Z-direction *only* very little lateral force is applied, and thus it is possible to analyze soft samples. Depending on the distance to the sample the tip experiences an interaction potential that goes from long-range attractive to short-range repulsive forces. The tip-sample interactions affect amplitude, phase and resonance frequency of the oscillating cantilever.

A feedback system is employed to maintain the oscillation amplitude at a set point value. The difference (*a.k.a.* the “error signal”) between the amplitude and set point, is used as the input to the feedback system. The output of the feedback loop drives the Z actuator. Thus, three maps can be generated:

- **Amplitude image:** map of the output signal of the feedback loop.
- **Topography image:** map of the voltage applied to the piezoelectric required to keep the oscillation amplitude constant, multiplied by the sensitivity of the piezoelectric in $[nm/V]$
- **Phase image:** map of the phase difference between the driving signal and the detector signal.

In this Thesis only the topography mapped is employed.

The principal advantages of the tapping operation mode are:

- No continuous contact.
- Suitable for soft materials such as biomedical samples.
- No lateral forces applied to the tip.
- Fast and long scan.
- Higher durability of the cantilevers than in contact modes.

To measure in tapping mode with the AFM, the following steps are performed:

1. Switch the electronic controller to “AC mode”.
2. Select the software the ACAFM operation mode.
3. Select the frequency of resonance pressing the button “ACTUNE”.
4. Select a frequency close to the frequency of resonance.
5. Tune the “Drive %” value to get an amplitude [2-2.5][V].
6. Move the laser to get a intensity close to 1.
7. Calibrate the photodetectors position to get a defection value close to 0 and a LFM value close to 0.
8. Select the area to measure.
9. Choose the span size.
10. Choose the velocity.
11. Measure.

Table 2.2: *Contact vs tapping mode*

Contact Mode	Tapping Mode
Advantages	Advantages
High scan speed. Atomic resolution. Scanning of samples with sharp topography.	Lateral force almost eliminated. Higher lateral resolution on most samples. Lower forces so less damage to soft samples or tips.
Disadvantages	Disadvantages
Distortion of the image due to lateral (shear) forces. Strong capillary forces may appearance in ambient condition. Lateral and strong forces reduce the resolution. (may damage tip/sample).	Slower scan speed than in contact mode.

A comparative table can be shown in Table 2.2. In summary, contact mode is used when the sample is hard and tapping mode with the sample is soft.

2.3.3.3 Qualifying procedures for nanoimprint auto-comparation

Qualifying the appropriateness of any given material for nanoimprint depends on the parameters that are considered relevant in any given process. In our case, the main aim was to qualify the manufacturing process and how it applies to different materials. Therefore parameters widely employed for surface quality analysis of micro- and nano-surfaces, such as kurtosis or skewness [81], have limited impact in this context. Within the characterization protocol we have proposed a number of parameters, and have implemented specific procedures for

measuring their magnitudes. The underlying idea is to extract simple figures qualifying the samples. The original idea was to propose a single figure of merit, but this proved unfeasible, since many parameters are derived from numerical data through image processing of an AFM software package. Moreover, the proposed parameters often rely on the specific master (e.g. saw-tooth grating) employed in any given series of measurements. Comparison between these parameters and classical measurements is not trivial and may induce false evaluations of the materials performance.

The parameters obtained from direct measurements are physical dimensions of the grating: the pitch, the blazing angle and the back angle, i.e., the angle opposite to the blazing angle. The remaining parameters proposed in this Thesis, derived from AFM image processing, are: a map of RMS differences from theoretical to actual heights, a histogram of oriented gradients (HOG), the average height, and the average height to pitch ratio. All these parameters are explained in the next section.

Numerical parameters

Support software of AFM units has become a sophisticated tool from which a high volume of numerical and graphical information can be obtained. Many packages, moreover, are open software, thus processing a high number of raw data may ultimately result in a time-efficient, relatively simple task.

Measurements were post-processed using two scripts successively. The first script processes raw AFM data, obtaining the fundamental numerical parameters -pitch, blazing angle and back angle- mentioned above, as well as a BMP image file containing either the surface or a transformation of the same, that will be processed by the second script. The first script is written in Python language, using a virtual Pygwy console of a Gwyddion package [82]. Gwyddion is a software environment specifically designed for data analysis of scanning probe microscopy.

- **Pitch.** A saw-tooth pattern shows an evident spatial frequency that can be checked after the imprinting to ascertain whether the pitch is kept or some shrinking or stretching has occurred during the different processes. Either being an issue or not for the application, the pitch variation and its dispersion for every tested material must be known.

Knowing the expected pattern, 2D Fast Fourier Transform (2D-FFT) was applied on to obtain the spatial frequency. The pitch value is obtained from the inverse distance between the 0th order and the 1st order of the FFT (actually the script takes the distance between the two 1st two orders and divides by two). In practice this is equivalent to average pitch estimations taken from different 1D profiles along the sample width. A material is given the highest quality score in this parameter if it shows the same pitch as the master. The lowest score is given to samples having zero or twice the master pitch. Intermediate values are linearly distributed.

- **Scale maximum.** Scale maximum is not employed as a characterizing parameter because it is not actually the average of pattern maxima, but simply the highest value found in the pattern. The value is directly captured in the script just finding the maximum value. It gives an approximate idea of the image scale and is used for certain general calculations within the code.

- **Angles.** The saw-tooth pattern is characterized by its pitch and angles. The blazing angle $\alpha_{blazing}$ determines the slope of the sawtooth and ultimately the sample topography. The blazing angle and the opposite back angle β are considered essential to estimate the quality of the copy. To evaluate such quality, the angles obtained from the copies are directly compared to the angles taken from the master measurements. Only the linear parts are considered in the calculation, as shown in Figure 2.21. Analogously to the pitch case, the mean master angle is considered the top achievable value. The lowest value correspond to 0 degrees.

Figure 2.21: 2D saw-tooth sketch showing the blazing angle α and the back angle β . Measurements are confined to the linear zone of the slopes (red rectangles), where the angles are supposedly constant.

Image processing parameters

The second script was written and executed on a MatLab interface employing the MatLab Computer Vision Toolbox. It utilizes the output of the Python script to evaluate the second set of parameters, *ie.* those derived from graphical inputs as commented above.

- **Vertical RMS deviations.** Every copy is compared to a perfect saw-tooth pattern having the same pitch and blazing/back angles. For every point of the 2D mesh, the height of the experimental points are compared to the theoretical saw-tooth pattern (Figure 2.22).

Figure 2.22: 3D sketch of a realistic saw-tooth copy compared to its theoretical master. The vertical difference between the mesh points of these two curves (red segment) is used to evaluate RMS.

The root mean square (RMS) deviation is calculated as

$$\sigma^2 = \frac{1}{N \cdot M} \sum_{i=1}^N \sum_{j=1}^M (x_{i,j} - x'_{i,j})^2 \quad (2.2)$$

- **Average height.** The MatLab script also includes a calculation of average height. The aim is not to find maximum heights, but averages of local maximum (Figure 2.23). The purpose is to avoid glitches, scratches and other artifacts in the evaluation of heights.

Figure 2.23: *Plot of an actual saw-tooth copy showing the average height maxima.*

- **HOG parameter.** The histogram of oriented gradients (HOG) is a feature descriptor widely used in image processing. First described a decade ago [83], it is usually employed to detect objects in images, especially pedestrians. Here we propose to apply HOG to quantify the amount of superficial errors of a given topography.

Figure 2.24: *Calculation of HOG descriptor for a given surface. Vectors points to the direction of highest gradient; this should be a parallel set of vectors if the sample were perfect. Deviations from this direction shows the presence of defects. See text for details.*

The underlying concept is relatively simple: in the 2D mesh of the sample under analysis, the maximum change rate (gradient) of different points (pixels) over a mesh is obtained

and their directions are marked with vectors (Figure 2.24). In our case a saw-tooth is being reproduced; were it perfect, the copy should show a set of vectors parallel to the grating pitch over the slope and back angle. However, the vectors show up divergences depending on the material and the manufacturing procedure. The number and magnitude of such imperfections can be related to the quality of the copy.

Figure 2.25: *Evaluation of HOG histograms. The example shows a 500×500 pixels (about $3 \times 3 \mu\text{m}$) fragment of an AFM image of a copy of the blazed grating master. Cells of 6×6 pixels show the local gradient. These are eventually employed to compute the histograms.*

The result is a single multicomponent vector (typically, several thousands elements) called HOG descriptor. The MatLab Computer Vision Toolbox contains a set of functions to calculate all relevant parameters related to HOG, making it straight forward to prepare a script that gathers the required information.

First, the gradients of a chosen point (actually, every pixel) of the surface with respect to its closest neighbors are calculated. In our case, the gradients are calculated from the 8-bit gray-scale image. Pixels are grouped into cells; each cell is associated to either a 9-bin histogram (20° 's per bin) corresponding to mean angles from 10° to 170° or a 18-bin histogram (5° - 175°).

The histogram sums the gradients included in each angle range. For any given gradient, its magnitude is proportionally distributed between the previous and the next bin. *E.g.* in the 9-bin histogram a gradient value of 10 is assigned with 50% to bin 1 and 50% to bin 2, 11 is assigned with 40% to bin 1 and 60% to bin 2. The sense of the vectors is not relevant; 180° wraps around to 0° . Each cell can then be presented by a gradient line (Figure 2.25) indicating the mean gradient direction. Finally the sum of the histograms may be presented as a global histogram (Figure 2.26).

Figure 2.26: Calculation of HOG descriptor. Histogram corresponding to the pixels of one cell. An 18 bin histogram $5-175^\circ$ is used. Gradients of every pixel are accumulated in the columns; the angles wrap around as 180° is equivalent to 0° . A normal Gaussian distribution is fitted to the histogram, and the FWHM Gaussian width is computed.

The histogram distribution is fitted to a normal Gaussian distribution in angles, centered at 90° . This assumption is obviously not valid for a standard images, but is a fair assumption in a blazed grating aligned at 90° . The Gaussian distribution seems appropriate to account for deviations from this preferential direction. The Gaussian FWHM width is employed as a measurement of the gradient dispersion.

- **Height to pitch ratio.** This parameter is useful to analyze whether pitch variations in the samples are due to a volume shrinkage/expansion or just a surface stretching. In the first case, the features are kept but scaled up or down, while in the second case some features may have been affected by the deformations.

2.3.4 Results and discussions

The measure of the thickness was done with the profilometer to know the thickness of the spin layer. Several measures in more than 500 samples were made. The results are summarized in Table 2.3.

Table 2.3: *Summary of results for different thickness of different materials tested in the laboratory*

Material	Thickness [μm]
OrmoStamp	7.85 ± 0.14
SU-8	1.61 ± 0.04
NOA-81	11.5 ± 0.3
Photoresist 1818	1.92 ± 0.05
PMMA	2.51 ± 0.04

The AFM provides the surface information. Figure 2.27 shows the surfaces of the master and one of the copies.

Figure 2.27: *AFM measurement of the master (left) and OrmoStamp copy (right).*

Measurements of the above-described parameters (section 2.3.3.3 on page 48) in 103 samples (copies from a 1800 lines/mm sawtooth grating GR13-1850 from Thorlabs) are shown in Figure 2.28. The six upper graphs are histograms of the different parameters; all the samples have been included, along with several measurements of the master at different positions.

The high number of samples precluded applying the same intermediate PDMS negative mold to all the samples: actually, three PDMS molds were used. Moreover, samples were made in batches on different days; although the experimental procedure was the same, some variables like room temperature or humidity were not controlled. The result clearly shows that for samples prepared with this manufacturing protocol to be comparable to each other, they must be fabricated with the same master mold (there are more variability in the Thorlabs masters than in the copies).

Figure 2.28: Summary of results. Sampled master and all 103 samples are presented.

Despite these difficulties (different master, give different copies), the results can be employed to explore the limits of the 3D-printed nanoimprint unit, along with the polymers that better suit the unit.

- **Pitch** is quite constant and similar in all materials. This is a positive indication of the unit performance and reproducibility; however, it is useless to qualify the different polymers.

- Vertical **RMS deviation** apparently shows a high variability, partly due to the large scale of the graph. Actually the deviation of most samples is within 10 *nm*, which is considered acceptable for the simple home-made unit tested here. It should be noted that RMS is higher in average for solvent-free materials. This result can be explained assuming that solvent somewhat flattens the samples diffusing their roughness. The difference is not large anyhow, except in a few Ormostamp samples of the same batch, whose manufacturing protocol was possibly compromised.
- **Blazing and back angles** clearly show the superior performance of solvent-free materials (NOA-81 and Ormostamp) in reproducing the master. Solvents, as commented above, tend to flatten the fresh samples before curing. This has a dramatic effect on the angles. While solvent-free Ormostamp angles are above 90% and NOA-81 angles are above 80% of the master angle, materials employing solvents barely reach 50% or less of these angles. This issue is extremely important, since the inability of reproducing these angles can be directly correlated to a deficient nanoimprint reproduction of relief features.
- The same result is obtained in the **height** measurement. Again Ormostamp and NOA-81 render a fraction of the master height substantially higher than materials using solvents. The PMMA case is particularly dramatic: the average height is scarcely 15% of the master height.
- The FWHM distribution of **HOG gradients** displays some interesting results. The first noteworthy result is the presence of dissimilar groups of values within the same material. We believe that this is a consequence of the extreme sensitivity of HOG to acquisition parameters. HOG is conceived as a descriptor of specific contents in image processing, Data are taken directly from the gray levels. Note that a number of tested samples show HOG values higher than masters themselves. This needs not be a sign of better performance; the values are just distribution widths. By now, we will consider that a material's HOG is better when showing less dispersion and values closer to master's. With these yardsticks the best material for HOG is S1818. Nevertheless, the number of samples is significantly lower than Ormostamp or NOA-81; a test with a similar number of samples should be carried out before selecting the best material. However the solvent should be help to make the surface more homogeneous and it is helping to have better values than with materials without solvent.

Evaluating the results on an overall scale, it appears that Ormostamp and NOA-81 are the better performers. Ormostamp is specially designed for nanoimprint replication; therefore, its superiority can be easily understood. However, the second position of NOA-81, only slightly behind than Ormostamp, is more surprising. This result can be attributed to the lack of solvent. Indeed, Ormostamp and NOA-81 are the only tested materials that do not require solvent on manufacturing. This subject seems to be crucial for optimum replication, especially in parameters -e.g., height, blazing angle- where the vertical dimension is involved.

A number of parameters have been measured in different materials to ascertain which material performs better in fabrication of nanoimprint replicas. The initial idea was to generate a figure of merit, which would make it possible to unequivocally choose the most apt material for any given application. However, it became clear that such a procedure must be adapted

to the requirements that reveal more relevant in a specific application. This could be done assigning weights to the different parameters, or even including other parameters or discarding any unrelated parameter within the application context, and make little sense in the context of characterizing the novel setup. Values as height, RMS and angle will be useful

One important conclusion derived from the results concerns the reliability of the system. The performance of the nanoimprint unit -notwithstanding its fabrication with a 3D-printer - can be considered satisfactory, providing the correct material is employed. This opens the opportunity of creating sophisticated nanoimprint elements with good reproducibility using a very simple and unexpensive tool.

Chapter 3

MICROSTRUCTURED DEVICES

Two series of devices will be explained below. The devices were design and manufactured with the technologies developed or already available in the R&D center CEMDATIC.

The first devices are the micro-lenses array. We made two difference kind of devices: static and tunable micro-lenses. The static micro-lenses were made using NIL technique. The tunable micro-lenses are made by a LC sandwich and the electrodes were made with two different techniques: conventional lithography and the laser ablation technology developed during the Thesis.

The second devices are the reconfigurable liquid crystal optical vortex generators. They were made using the laser ablation technology. They are composed by 24 pie slices , whose optical retardation can be independently tuned up. A driver was designed and manufactured to control the 24 pie slices individually.

The purpose of the manufacture of these microstructured devices is, on the one hand, to develop devices with an immediate practical application, on the other hand, to develop the necessary technologies to be able to include more complex micro-structures in the photonics integrated circuits that are detailed in the following chapter and on future devices. These technologies will allow, for example, to manufacture efficient coupling and light extraction structures in the waveguides, like prism and gratings, and structures to improve the coupling from optical fibers.

3.1 Micro-lenses arrays

A micro-lens is a lens with a diameter in the $10\ \mu m$ to $1\ mm$ range. Micro lenses are typically made of one uniform material with one plane surface and one curved (convex or concave) surface to refract the light. Other micro-lenses consist of two flat, parallel surfaces. In these the focusing action is obtained by a variation of refractive index across the lens, and thus these lenses are known as graded-index lenses [84].

Micro-lens arrays contain multiple lenses arranged in two dimensions on a supporting substrate.

Single micro-lenses are often used to couple light to optical fibers. Arrays of micro-lens have

several applications, such as collection efficiency for CCD cameras or photovoltaic devices, in Hartmann Shack wavefront analyzers, Light Field Photography or Plenoptic Cameras, cameras that eliminate the need to focus before taking images since the micro-lens arrays allows focusing with software during post-processing.

3.2 Micro-lenses arrays by Nanoimprint lithography (NIL)

In this section all the processes for the pattern transfer with the NIL will be explained.

3.2.1 Nanoimprint lithography for micro-lenses fabrication

The creation of a mold of PDMS with the negative micro-lenses pattern is the first step to the nanoimprint process. The second step is to imprint the pattern on the polymer material and to cure the polymer, so the pattern remains when the mold is removed.

The steps are:

1. Making a negative mold in PDMS (described in section 2.3.1 on page 39).
 - (a) Prepare, homogenize and degas PDMS.
 - (b) Deposit a monolayer of release agent onto master pattern.
 - (c) Situate the master in the *Mold-maker* machine and carefully pour the PDMS on top of the master.
 - (d) Degas the PDMS again, this time *in situ*.
 - (e) Cure the PDMS
 - (f) Release the negative mold from the master.
2. Imprint the negative mold into the desired material using the *Printer* machine (described in section 2.3.2 on page 42).
 - (a) Prepare the substrate.
 - i. Cut the glass.
 - ii. Clean the glass.
 - iii. UVO surface treatment of the glass.
 - (b) Deposit the material by spin coating (see table 2.1 on page 42).
 - (c) Place the spin-coated glass and seal with the negative mold of PDMS in the *Printer*. Apply vacuum.
 - (d) Cure the material.
 - (e) Release the material and the negative master from the *Printer*.

3.2.2 Results of the fabrication of micro-lenses arrays by NIL

Following this procedure, a PDMS mold has been successfully made with a pattern of an array of micro-lenses. Then the pattern in the PDMS is imprinted on NOA-81, resulting in polymer micro-lenses arrays, as shown in the Figure 3.1. Not surprisingly the copies are very good as the resolution of our machine has been checked up to $0.23 \mu m$, and the micro-lenses arrays pattern have a $15 \mu m$ pitch.

Figure 3.1: *Left micro-lenses master mold; Right nanocopy (NOA-81).*

3.3 Modal Liquid Crystal Lenses (MLCL)

Modal Liquid Crystal Lenses (MLCL) are made in standard LC cells using patterned electrodes.

Different electrode patterns and structures have been used in one of the inner faces of the sandwich structure, in order to generate the desired switching profile of the micro-lenses:

- **Hole pattern.-** These lenses have holes in the upper ITO layer, removed by photolithography. The holes generate a different field strength over the surface, and consequently a switching gradient voltage that is reduced towards the center. Thus, in a positive liquid crystal cell, the birefringence increases towards the center gradually, making the cell behave as a lens for polarizations parallel to the switching plane (Figure 3.2).

Figure 3.2: *Hole pattern electrode structure, the simplest approach.*

- **Hole pattern with an extra layer of higher conductivity, such as Al or Mo.-** The principle for this kind of lens is the same, but the added metal layer has two positive effects. 1) being opaque it blocks stray from among lenses, and 2) being better conductor than ITO, no voltage drop is experienced across the array, and thus the micro-lenses in the center of the device switch in the same way as the micro-lenses on the edges of the device (Figure 3.3).

Figure 3.3: *Hole pattern electrode structure with high conductivity layer for uniformity along the array.*

- **Hole pattern with high resistivity layer (PEDOT:PSS).-** The idea is the same than with the simple hole lens, but adding a resistive layer and combining it with a relatively high frequency AC field, a variation in field strength is obtained in the electrodes. A so called line of transmission symmetry is established. The voltage drop over the lens depends on the capacitance of the LC cell primarily defined by the cell thickness, the resistivity of the PEDOT:PSS, and the frequency of the electrical applied field (Figure 3.4).

Figure 3.4: *Hole pattern electrode structure with resistive layer for different field density.*

- **Laser patterned.-** As above a field gradient is generated by inducing variations in the field in a concentric circular manner. The induction of the field will depend on the electrode separation in one plane, the cell thickness and the frequency of the AC field (Figure 3.5).
- **Hole pattern with capacitive bi-layer.-** Electrons in ITO will polarize the resistive layer by induction. An AC field will be applied in the hole pattern with $A = \pi \cdot (rSiO_2)$ high resistivity layer cells. A voltage gradient will be established as a function of field

Figure 3.5: *Laser patterned ITO with different designs to achieve a combination of the previously described effects.*

strength, cell thickness, conductivity of resistive layer, and dielectrical characteristics of the isolating layer (Figure 3.6).

Figure 3.6: *Holed pattern with capacitive bi-layer for different field density.*

3.3.1 LC micro-lenses arrays fabrication by conventional lithography

In our laboratory a 400 W mercury gas lamp is used; the resolution of this technique is around 1 μm . The photoresist used is Microposit 1818, a positive photoresist where only UV-light exposed resin is removed by the developer. This process is shown in Figure 3.7.

In this section photolithography will be used for making electrodes in ITO and for some designs in Al or Mo. In next chapter photolithography will be used to make waveguides and different photonic integrated devices. The procedure is the same but different exposure and developer times are required for some materials.

3.3.1.1 Electrode fabrication (Photolithographic procedure)

Protocol for the substrate preparation is explained in section 2.1.2.1 on page 27 and it can be summarized in the following steps:

1. Cut the glass.
2. Clean the glass.

Figure 3.7: General scheme for standard photolithography protocol with wet etching.

3. Condition the glass by UVO.

Spin coating of photoresist

In this step, the substrate is covered with photoresist (Microposit 1818) by spin coating.

1. Place the substrate into the spinner and fix it with vacuum.
2. Drip photoresist on the center of the substrate.
3. Spin the photoresist covered substrates at 3000 *r.p.m.* for 30 *s.*

Soft bake

Soft baking is used to improve the coated material to the substrate by heating. Some materials have solvent which is eliminated in this step.

- Put coated substrates in the oven at 90 °C for 30 *min.*

Exposure

The exposure is one of the most important steps. The pattern is transferred from the physical mask to the photoresist. Depending on the kind of photoresist the exposed part remain (negative) or is selectively eliminated (positive) by the developer in the subsequent developing step.

1. Put the substrate on the base platform that has perforations for vacuum fixing of the substrate. The fastening to the base may be improved by sealing the holes not covered by the substrate.
2. Attach the desired mask to the metallic frame with using Kapton TESA adhesive tape, avoiding any leak, and place the frame in the lid that covers the the base platform.
3. Align the mask with the substrate.
4. Once they are aligned, fix the relative position of the mask and substrate applying the vacuum.
5. Apply hard contact by creating a vacuum of 15 *mbar* in the sealed environment between the mask and the substrate. This will ensure that light only hits the wanted parts through the mask.
6. Set the exposure time of the mercury lamp to 6 *s* and set the power to 400 *W*. Wait until the UV lamp is ready.
7. Slide the platform underneath the UV lamp and it will be automatically exposed for programmed time and power.
8. Once the UV lamp switches off, withdraw the platform, turn off all the vacuum switches and take out the exposed substrate.

Figure 3.8: Base platform for substrate support(left), metallic platform for mask support(middle) and UV-light exposure (right).

Developing

A *developer* is a chemical agent that removes selectively the exposed areas of the positive photoresist. The developing time is critical, otherwise neither or both (exposed and unexposed area) may be removed.

1. To remove the exposed photoresist: put substrates in the developer and shake it slightly for 30 s, until no more photoresist is removed in the bath.
2. Wash the glass in distilled water and dry it up with nitrogen gun.
3. Check in the microscope that the photo-resist pattern is as expected.

Hard-bake

This bake solidifies the remaining photoresist and makes it resistant the following acid attack (wet etching) used for the selective removal of ITO, Al or Mo from the substrate areas not protected by the resist.

- Put the substrates in the oven at 110 °C for 30 min.

Wet Etching

In etching, a chemical agent removes the uppermost layer of the substrate in the areas that are not protected by photoresist.

- ITO is removed by a mixture of 49% HCl (hydrochloric acid), 1% HNO₃ (nitric acid) and 50% H₂O.
 1. Put the substrates in acid at 45 °C for 35 s. The temperature of the acid is critical. If the time is too long or temperature is too high, the photoresist will not withstand the acid attack and the pattern will be destroyed. If time is too short or the temperature is too low, the layer will not be removed.
 2. Wash the substrate with distilled water and dry with nitrogen gun.
 3. Check the result in the microscope.
- To remove a layer of Al a different acid bath is used 200 cc of HPO₄ (phosphoric acid), 20 cc of CH₃COOH (acetic acid), 15 cc of HNO₃ (nitric acid) and 5 cc of H₂O.
 1. Put the substrates in acid at 50 °C for 20 s.
 2. Wash the substrate with distilled water and dry with nitrogen gun.
 3. Check the result in the microscope.
- For Mo the acid mixture is the same than for the Al, but the etching is made at room temperature.

1. Put the substrates in acid at room temperature for 20 s.
2. Wash the substrate with distilled water and dry with nitrogen gun.
3. Check the result in the microscope.

Photoresist elimination

The last step removes the photoresist from the sample (Figure 3.9).

Figure 3.9: *Microhole pattern with photoresist (left) and cleaned (right). The microholes have $86\ \mu\text{m}$ of diameter and the pattern has $1.5 \times 1.5\ \text{cm}$*

- Put the substrate in acetone until the photoresist is completely eliminated.

3.3.1.2 Deposition of additional layers

In some of the approaches additional layers are deposited before the electrodes fabrication (in the case of conductivity layer) or after (capacitive layers). This section explain how to deposit theses layer.

PEDOT:PSS layer deposition

To deposit a high resistance conductive polymer layer, an aqueous solution of 1.3 wt% PEDOT:PSS is spin coated onto the substrate with holes already made by photolithography.

1. Put the samples in the UVO cleaner for 10 min.
2. Spin an aqueous solution of PEDOT:PSS of 1.3 wt% at 4500 r.p.m. for 30 s.
3. Put the substrate into the oven for 20 min @ $80\ ^\circ\text{C}$ principally to evaporate the water.

Physical vapor deposition (PVD) of additional layers

Some electrode designs require additional layers. Some of them (SiO_2 , Ni and Al) will be deposited by evaporation. The target material is vaporized and then condensed onto the sample. Electron-beam (e-beam) evaporation has been used in this Thesis.

E-beam evaporation is a method to deposit thin layers in a controlled way by e-beam heating of a target. The computer controlled process occurs in a vacuum chamber. The material is loaded into a small container (Figure 3.10) called crucible, specially designed not to react with the target material. Vacuum is typically applied to avoid that O_2 interferes with the evaporated material. The thickness of the coating can vary from few Å to several microns, depending on the evaporation rate and time.

Figure 3.10: *PVD chamber, upper chamber (left) and material container (right).*

Al is evaporated on intact ITO substrates as a high conductive layer (more than ITO). For capacitive approaches, SiO_2 and Ni are deposited as a thin coating on the substrates with hole pattern. The different protocols are explained in the following.

1. Open water-refrigeration system. Power up the e-beam power supply and start the control program. Set the bulk density and Z-factor on the thickness controller that can be checked by the LCD panel. The parameters for evaporated materials and the typical deposited thickness are shown in Table 3.1, as are the typical thickness deposited.
2. Open the chamber. Load material into the crucible and place the samples in the holder. All the surface is covered by Al. For SiO_2 the area where future connections will be made has to be protected with Kapton film, otherwise it becomes non-conductive. For Ni all the area has to be covered except the active area (where holes are located) that is protected by photoresist. Close the chamber.

Table 3.1: *Parameters of evaporated materials*

Evaporated material	Bulk density (g/cm ³)	Z-Factor	Crucible	Thickness (nm)
Aluminium (Al)	2.73	1.080	Alumina	30 @ 1 nm/s
Silicon Dioxide (SiO ₂)	2.20	1.070	Alumina	25 @ 0.1 nm/s
Nickel (Ni)	8.91	0.331	Graphite	60 @ 0.1 nm/s

3. Make sure that the button “check gate” is on in software e-gun menu and that the water is in manual operation. Apply vacuum to the chamber until a vacuum better than 9.0×10^{-5} Torr is reached. Turn on the e-gun to heat the material in the crucible. The e-beam voltage is set to 6000 V. Increase slowly the current to exceed the threshold where evaporation/deposition starts. It is detected by the quartz detector. The material in the crucible starts to shine and evaporates. Typically this happens around 100 mA, but the evaporation rate depends on the quality of vacuum and on the nature of the target material. The higher the current, the higher the deposition rate.
4. Turn off the current when the value is close to the wanted thickness, taking into account that some heat remains just after turning off the e-gun and some evaporation will occur. Then wait for 5 minutes to disperse heat before turning the power and the vacuum off and letting the air come into the chamber.
5. When the vacuum is released, open the chamber and take the coated-substrates out.

3.3.1.3 Procedure for LC cell assembly

Once the electrode is done, the LC cells can be mounted. The steps are explained below:

Alignment layer (PI) by spin coating

To get uniform optical behavior in the active area all molecules in the LC have to be oriented in the same way. The configuration for the micro-lenses is planar, and is achieved by rubbing the alignment layer.

1. Take one patterned electrode substrate and one non patterned and place them into the UVO cleaner with the inner side looking upwards. Expose for 10 minutes to prepare the surface for the next deposition.
2. To make a fine alignment layer, polyimide (PI) has to be stirred in the magnetic stirrer and deposited with a $0.45 \mu\text{m}$ filter until covering the whole surface and then spin coated onto the inner side of both substrates in a ramp from 0 to 4500 r.p.m. in 40 s.
3. Then put glasses in the oven for 1 hour at 200 °C to harden the PI.
4. Mark an arrow along the long axis on the side without PI, and rub the PI coated side following that arrow.

Place Spacers

Before placing the spacers to ensure an uniform thickness of the cell, a nitrogen gun is used to remove any dust. Deposit spacers on the PI layer on one of the two substrates. Spacers are deposited by spin coating if they are $6\ \mu\text{m}$ silica powder (previously suspended in methanol $1\ \text{mg/ml}$) for the simplest cell if a thicker gap is needed place a $0.05\ \text{mm}$ thick pet-spacer.

Mount LC cell

1. Face two substrates with the PI inside the cell in anti-parallel rubbing disposition, taking care that the active area is covered by the two faces, yet leaving some area on the edge for cable mounting.
2. Press the cell with clamps to ensure that thickness between the substrates is uniform (Figure 3.11).

Figure 3.11: *Clamp to ensure the spacers thickness during the gluing.*

3. Apply a little amount of NOA 81 as glue to the already spaced cell using a short wire. The glue will flow inside the cell acquiring the thickness determined by the spacers.
4. Quickly expose the cell to UV light for few seconds to solidify the glue, so it does not flow excessively inside of the cell. Repeat the process on both sides.

Filling and sealing the LC

With the help of a pipette fill the LC while having the cell on a hot plate at $90\ ^\circ\text{C}$ so the LC flows better, and with a lancet beat the cell gently as the LC fills the device by capillary forces to avoid getting trapped air inside, causing defects.

Check the resulting cell through a polarized light emitter and a polarizer.

Figure 3.12: *Filling of cell with a pipette.*

Use a wire to put sticker around the edge of the cell to seal it and put in the UV light if the LC is not polymerizable.

Connect the electrodes

After the cell is mounted, soldering cables to the ITO in the exposed area will allow to test the device. However, copper wires do not adhere well to the ITO, so to make the connections these steps are followed:

1. First remove polyimide in the soldering area using an erasing rubber, and after clean with a wipe dipped in isopropyl alcohol.
2. Then apply a little bit of indium metal with the hot tip of a soldering iron.
3. Cut copper wires and apply some soldering tin using the soldering iron.
4. Then fuse the tin of the wires with the In on the ITO. Now the micro-lenses devices are ready to be tested with an electrical signal.
5. Protect the connections with epoxy.

3.3.1.4 Results

Once the device is made, the response to an electrical field has to be analyzed. The results will be divided according to the different approaches used to manufacture the micro-lenses: lenses made by non-conductive hole pattern, lenses made by non-conductive hole pattern and an additional conductive layer, hole-patterned resistive layer lenses with PEDOT:PSS and

hole-patterned capacitive lenses with evaporated Ni SiO₂. The liquid crystal filled into the micro-lenses was in all cases MLC-13000 (Merck Chemicals).

Micro lenses made by non-conductive hole pattern

The viscous forces tend to align the LC uniformly inside the whole cell. Depending on the position in the lens, these forces will counteract or collaborate with the electrical field. Outside the hole, the electric field lines are perpendicular to the rubbing direction; its intensity would be high enough to fully switch the cell (Figure 3.2). In close proximity to the edge electrical field forces will predominate over the alignment layer forces but as the field reduces towards the center, the viscous forces will tend to be dominated by the alignment direction.

Figure 3.13: *The hole lenses working at 10 kHz at various voltages. The cell thickness was approximately 6 μm , and the diameter 129 μm .*

Thus in the ideal case, the voltage in center will not exceed the threshold voltage, while the cell switch fully at the edges, and a quasi-uniform gradient from edge to center is formed.

The focal length of the cells can be calculated according to equation (1.15), which in the manufactured cells result in a focal length of approximately

$$\frac{64^2 \mu\text{m}^2}{2 \cdot 0.13 \cdot 6 \mu\text{m}} = 2.6 \text{ mm} \quad (3.1)$$

The thickness of the outer cell glasses is 0.7 mm . Therefore, the focal plane of the micro-lens array is approximately 2 mm from the outer surface of the glass cell, This is convenient, e.g for an array located in front of a charge-coupled device (CCD) focusing the incoming light onto the camera pixels.

Based on visual inspection, the performance of the lenses was optimal at approximately 20 V (Figure 3.13), yet showing a retardation color in the center which indicates a slight switching.

Based on the interference colors (*Michel Levy Chart*) the focal length can be estimated using the formula (1.15). Which leads to the following table, where the optical path distance has been determined from the interference color. Only the lenses with a continuous drop over the entire open area have been considered (see table 3.2).

Table 3.2: *Focal length as a function of the applied voltage*

Voltage [V]	Retardation $\Delta n \cdot d$ [nm]	Focal length [mm]
12	800	2.6
16	700	2.9
20	620	3.3
24	570	3.6
28	520	3.9
32	480	4.3
36	440	4.7
40	400	5.0
44	370	5.4
48	350	5.8

In order to improve the response and to increase the flexibility in focal lengths, several variations over the hole-pattern lenses were generated.

Hole-patterned resistive lenses with PEDOT:PSS

PEDOT:PSS is used instead of PI as alignment layer in this cell (Figure 3.4). As already mentioned PEDOT:PSS is conductive, although with a much higher resistivity than ITO or any metal. Various cells with PEDOT:PSS were produced, but the quality of the alignment, and the non-satisfying optical response were inadequate for any real applications (Figure 3.14).

Hole-patterned capacitive lenses with evaporated Ni SiO₂

In order to explore the potential of the capacitive lenses, SiO₂ and Ni were evaporated onto patterned ITO glass to control the voltage across the hole (Figure 3.6). The hole-pattern capacitive lenses were addressed with frequencies as low as 10 kHz , and were still providing a lensing effect as illustrated in Figure 3.15. The quality achieved in these lenses was worst than simple hole-patterned lenses

Figure 3.14: *50 μm thick resistive lenses with a PEDOT:PSS resistive alignment layer. The sample was addressed at with a 1.5 MHz square waveform. Surprisingly the cell showed a much higher threshold voltage than when PI is used for the alignment. The profile of the lenses and the lenticular response were not satisfactory.*

Figure 3.15: *Nickel coated hole patterned ITO creating a capacitive lenses array. The array is addressed by a 10 kHz square waveform with varying amplitude.*

3.3.2 LC micro-lenses arrays fabrication by laser ablation lithography

Laser ablation technique can shape the electrode with microscopic structures, dividing the electrode conductive path into different tracks. The laser ablation technique for shaping the electrode with microscopic structures was employed in order to study the combination of resistive and capacitive lenses.

Five kinds of electrode patterns were made (Figure 3.16).

Figure 3.16: *Laser-made electrode with different patterns resulting in different resistance from edge to center. The track width is $20\ \mu\text{m}$ and the diameter of the lens is $100\ \mu\text{m}$.*

The procedure is explained in the section 2.1 on page 69 and is summary below:

1. Prepare the substrate (Described in section 2.1.2.1 on page 27) .
2. Mount the glass on the XYZ stage using Kapton TESA tape. Make sure that critical parts of the substrate are not covered by the tape.
3. Start the Laser program: Move the cursor along the X and Y axes to the corner of substrate where is designed to set the relative zero position. Adjust Z to focus on the surface with ITO. Make sure that the focus is made on the the correct surface by test scribing in a non-critical area.
4. Upgrade it as default focus. Set the desired power of the laser in the explorer menu. The standard laser settings are 5 A, 50 kHz. The output power employed for the top-scribing for the micro-lenses was 18% (54 mW @ 50 kHz).

5. Import the program code and execute it. The velocity of the movement is normally chosen at 10 mm/s .
6. When the electrode is finished the LC-cell is mounted as described in section 3.3.1.3 on page 69.

The ITO sheet, like all thin film conductors, is characterized by a sheet resistance, measured in Ohms per square ($\Omega/\square$) which typically depends on the thickness and the deposition quality. The ITO employed in this study had a sheet resistance of approximately $100 \Omega/\square$. Thus a 2 mm wide and 10 mm long track has a resistance of 500Ω from end to end.

In order to calculate the effective resistance in the structures, the effective length from edge to bottom must be calculated, and divided by the width of the track. In the tested electrode patterns the track width (W) was kept constant at $20 \mu\text{m}$ (Figure 3.16) while the spiral length (L) from edge to center varied. In the case A, the resistance for one route involving one of the quadrants was calculated, and then divide the resistance by a factor of four, based on the relation between parallel resistors:

$$\frac{1}{R_{tot}} = \frac{1}{R_1} + \frac{1}{R_2} \quad (3.2)$$

$$4R_A = \frac{L_A}{W} \cdot 100 \frac{\Omega}{\square} = \left[\frac{\pi}{2} \cdot \frac{[40+30+20+10]\mu\text{m}}{20\mu\text{m}} + \frac{50\mu\text{m}}{20\mu\text{m}} \right] \cdot 100 \Omega = 1k \Omega \implies R_A = 250 \Omega$$

In case B the resistance among circles is infinite:

$$R_B = \infty \Omega$$

since no connection is present among circles.

In case C there are two resistances in parallel in each circle, but since one is twice the other the calculations are simple:

$$\frac{1}{R_{tot}} = \frac{1}{R_{1/3}} + \frac{1}{R_{2/3}} = \frac{1}{R_{1/3}} + \frac{1}{2 \cdot R_{1/3}} \iff R_{tot} = \frac{2}{3} R_{1/3} \quad (3.3)$$

$$R_C = \left[\frac{2}{3} \cdot \left(\frac{2}{3} \cdot \pi \frac{[40+30+20+10]\mu\text{m}}{20\mu\text{m}} \right) + \frac{50\mu\text{m}}{20\mu\text{m}} \right] 100 \Omega = 860 \Omega$$

In the last two cases there are spirals which can be approximated to four concentric circles plus the radius.

$$R_D = R_E = \left(2\pi \frac{[40+30+20+10]\mu\text{m}}{20\mu\text{m}} + \frac{50\mu\text{m}}{20\mu\text{m}} \right) 100 \Omega = 3.4 k\Omega$$

In all the cases the R_B value is several orders of magnitude too small to induce a voltage drop over the micro-lenses.

The conclusion is that for micro-lenses introducing resistive patterns in the ITO is pointless, however one may consider this approach for larger cells where larger resistances can be achieved.

At the same time, the laser ablation generates tracks which are so close together that the electric field in one electrode induces a field in the neighboring electrode, which is exactly what happens in case B.

The array is addressed by a 10 kHz square waveform with varying amplitude. A lens profile is achieved in a relatively low voltage regime (Figure 3.17).

Figure 3.17: *Laser cut concentric circles*

3.4 Relevant results of micro-lenses arrays

The NIL device works perfectly for micro-structured applications. Errors of a few nm or even tens of nm are irrelevant for micro-structured optics.

Various lenses and lens design have been developed, and a study of their behavior has been done. Calculations show that for micro-lenses resistive tracks in the ITO cannot generate enough resistance for the field to be reduced over the lens area. At the same time, hole-pattern, the capacitive Nickel and capacitive laser ablation lens showed lens profiles, which can be optimized in future devices.

It is also important to notice that devices manufactured with very high thicknesses which when fully switched give rise to focal lengths, which are so short that they are not useful, unless combined in a more complex optical system, or if special care is taken not to work in saturation of the LC cell. However, working with thick cells, gives the wider range of tuning, which facilitates the generation of a perfect lens.

3.5 Reconfigurable liquid crystal optical vortex retarder plate

The simplest optical vortices are characterized by a continuous azimuthal variation of the phase, leading a spiraling, rather than planar, progressing wavefront.

A vortex is described by its topological charge, *i.e.* how many twists the light does in one wavelength, which is the same as how many helices are intertwined, or how many angles in any given cross section have the same phase.

Vortices are characterized, amongst other things, by a singular powerless point in the middle, where all phases are present. At the same time vortices with different topological charge are mutually perpendicular.

Figure 3.18: *SPP scheme [1].*

Vortices have a wide number of applications from solar corona imaging [85], to sub wavelength fluorescent bio-imaging (Stimulated Emission Depletion STED) [86], optical tweezers [87] and optical communications [88].

A novel liquid crystal device for laser beam manipulation based on a multi electrode optical vortex retarder plate (OVRP) or SPP (see section 1.5.2 on page 19 for more detail) is presented below. The device consists of a nematic liquid crystal cell of patterned ITO-coated glass plates.

The device includes a list of novelties:

- The electrode pattern has been generated using mask-less ITO ablation employing a tripled Nd-YAG laser as described in section 2.1 on page 25.
- The resulting OVRP has 24 sections which is, to our knowledge, the highest number of sections reported ever.
- In order to address the 24 sections a novel electronic driver based on PWM has been developed, together with driving software that allows for easy calibration, as well as driving, and reconfiguration of the OVRP.

3.5.1 Fabrication of the optical vortex retarder plate

The device was designed using a CAD program. An active area of $10 \times 10 \text{ mm}$ was engraved on an ITO covered glass substrate measuring $50 \times 50 \text{ mm}$ with electrodes measuring $2 \times 4 \text{ mm}$.

The ablation was done by back-scribing as described above in section 2.1 on page 25. A $9 \text{ }\mu\text{m}$ cell was mounted using PI as alignment layer and MDA-98 as LC. The electrodes were manually soldered and connected to the driver.

3.5.1.1 Laser ablation

The design of the laser ablation permit individual control of each of the pie-slice shaped electrodes (Figure 3.19). The laser ablation has the advantage that only minimal ITO is removed from the central area, where the all the cutting lines meet.

Figure 3.19: *The manufactured design, here drawn in 1:1 scale. The patterned base glass measures $50 \times 50 \text{ mm}^2$, and the single electrode cover glass measures $30 \times 20 \text{ mm}^2$. The active area (purple) measures $10 \times 10 \text{ mm}^2$ limited by the start of the fan-out (blue). The electrodes are on a 4 mm pitch with a duty cycle of 50%. Additional cuts are made between electrodes to reduce the chance of shorts, and markers are included to facilitate assembly (green).*

Various attempts were made in order to use the minimum power needed to generate full isolation between pie slices. In the process a detailed manual for the handling of the laser engraver was generated.

The design was made so that different cutting parameters could be employed in the different sectors. In the active area, it is of out most importance that the finest possible isolating line is generated, while in the rest of the device it is only important that good isolation is achieved.

Eventually the optimal conditions were determined to be 18% power in the active area, and 32% power was used in the rest of the device, at a cutting speed of 10 mm/s).

3.5.1.2 Wiring and Testing

Prior to wiring with cables, the cells were tested for short cuts. A number of devices were manufactured and wired (Figure 3.20).

Figure 3.20: *Examples of two of the devices manufactured and wired.*

The cells were connected to the novel voltage generator and were assessed between crossed polarizers. As appreciated from Figure 3.21, occasional imperfections were present in the devices. In the left sample (Figure 3.21) a line outside the center of the active area can be seen. In the right (Figure 3.21) two imperfections in the active area are detrimental to the device performance, and as a consequence this device had to be discarded (Figure 3.22).

Figure 3.21: *Examples of two of the devices (Y3 and X1) manufactured and wired. On the left a continuously varying signal is applied, on the right a binary pattern.*

Figure 3.22: *The two defects in the active area seen in device X1.*

3.5.1.3 In house, developed LC driver

An Arduino UNO is used as a PC-SPI interface. A 24x12 bit PWM LED driver is used to control 24 analogue switches, each connecting an electrode with an external waveform generator. A full description can be found in appendix D on page 147

Figure 3.23: *Real picture of the developed driver.*

3.5.2 Characterization of the device

The cells were calibrated for different PWM duty cycles by measuring the transmission between crossed polarizers at 45° to the switching plane. Four central zones were chosen in order to evaluate the homogeneity of the cell.

3.5.2.1 Device calibration

In order to eventually use the device as a vortex generator it is imperative to know the voltage levels needed to induce a given phase retardation. For this reason a setup was made with a

Figure 3.24: *The setup used for characterizing the induced phase difference in the devices.*

monochromatic light source (He-Ne laser), which emits at 632.8 nm , and the sample situated between two crossed polarizers (Figure 3.24). The same laser which will be used for the future vortices.

Four arbitrarily regions of the device were chosen to be characterized (Figure 3.25). Note

Figure 3.25: Four arbitrary regions of the device were chosen to be characterized.

that the same tension is applied to whole device for the characterization, this divisions in pie slices become invisible. The illumination is not perfectly uniform, which does not affect data acquisition.

For each of the regions, four sets of data were captured corresponding to each of the four pixels present in the CMOS cameras color filter RGBG. The intensity signal varies with the field applied since the birefringence varies (Figure 3.26)

Figure 3.26: Captured transmission data for one of the green pixels for each of the four regions as a function of the PWM duty cycles. Even if the light transmission is not perfectly uniform over the sample, the birefringence of all sectors is almost the same at any given effective field. It is trivial to assign the points where δ is a known number of π , knowing that $\delta(\infty)=0$.

The phase difference (δ) was fitted assuming pseudo exponential reduction of the birefringence of the cell with the increasing effective applied voltage $\delta = Ae^{-B \cdot dc} + C$ and $I \propto \sin^2 \frac{\delta}{2}$.

A good fit for $\delta \in [\pi; 3\pi]$ was obtained as shown in Figure 3.27

Figure 3.27: Plot of the $\delta - \pi$ and exponential fit (left label), measured normalized intensity and fitted intensity (right label) between π and 3π

3.5.3 Reconfigurable vortex generation

Seven different topological charges [1,2,3,4,6,8 and 12] were tested, focusing the camera image plane onto the optical vortex retarder plate with the crossed polarizers at 45° to the switching plane (Upper images from Figure 3.28).

Figure 3.28: Upper row shows the δ introduced by the sectors of optical vortex retarder plate, middle row shows the laser beam power distribution after the optical vortex retarder plate for the different topological charges [0,1,2,3,4,6,8 and 12]. Bottom row show laser beam simulation through the optical vortex retarder plate for different topological charges using Fourier optics.

One of the crossed polarizers was subsequently removed and the other was aligned with the switching plane of the LC, and the collimated laser beam was shone directly onto the camera CMOS after passing the optical vortex retarder plate. Clear circular power distribution was

obtained with central power singularity varying in size proportional to the topological charge, illustrating the correct functioning of the device (middle images from Figure 3.28).

Simulations were performed to compare actual output with the theory. The simulation

Figure 3.29: Screenshot of the vortex simulator program.

consists of a laser that shines through the OVRP whose the topological charges can be varied. Initially a Gaussian power distribution was employed for the laser beam, but it was found that an intensity variation corresponding to an Airy pattern corresponded better to the measured behavior. This pattern implies that the output of the laser is dominated by the presence of a pinhole. The laser output was thus taken to be a *shifted* FFT of a pin hole transmission

function. A second FFT, *un-shifted* this time, was then applied to this Airy pattern (regenerating the pin-hole power distribution on the OVPR). The introduced phase distribution pattern is then added to the phase, and an inverse *shifted* Inverse fast Fourier transform (IFFT) is performed. The reproduction of the observed pattern is striking, although the justification for the shifting, or non-shifting, of the different transforms is still a subject of study (Figure 3.28).

The simulation allows to change the topological charge in the OVRP (*Topology*), pie slices (*Pie slices*), the calculated space in pixels (*Resolution*) and the virtual pinhole radius (*Virtual Pinhole Size*) (Figure 3.29).

The code is shown in Figure 3.30.

Figure 3.30: Labview code for the vortex generator simulation.

A reconfigurable OVRP with a complete control system has been manufactured and successfully tested. The device represents the leading edge of current development of reconfigurable vortex generators. The device proves not only the viability of manufacturing principles, but is also the first device of a number of derived devices which are being designed in our group, hereunder reconfigurable spiral diffractive lenses.

The produced device has been over engineered, in order to be applicable to the second and third optical communications window. It is the intention to assess its potential in this context.

The device has already formed the basis of an idea, which will lead to several devices which will be basis of future patents.

Chapter 4

PHOTONIC INTEGRATED CIRCUITS

This chapter describes how PICs have been designed, manufactured and characterized. Two techniques are used for devices manufacturing: LDW and conventional photo-lithography. All the devices presented in this chapter are passive (non-emissive). Electro-optically tunable devices containing LC have been prepared as well.

The first device is a power splitter. It is composed by an MMI splitter with an LC on top of the cladding that affects the evanescent wave. The second device is a polarization switch/splitter. The device consists of two parallel waveguides with a plane-parallel LC intersection. This configuration allows to change the optical path of one polarization.

4.1 PIC using conventional photo-lithography (LC-PICs)

This section describes how a PIC can be made using conventional photo-lithography. The objective was to make a PIC and to integrate it with LC to make it electrically tunable.

Structure base on waveguides connected to MMI couplers and directional couplers have been designed, simulated manufactured and characterized. In the case of MMI couplers, the response with temperature and interacting with LC has been studied. Selected materials had been **SU-8 negative photoresist for the core and sputtered SiO₂ for the cladding**.

4.1.1 Device design protocol

In this section a summary of a set of procedures for devices design are presented:

- **Materials selection.** Along the project, we have dealt with a number of materials suitable to be employed as films (core), claddings and/or substrates of our waveguides. Criteria for material selection derive from a number of considerations:

- Optical performance (Transparency and lack of scattering).
 - Mechanical properties.
 - Stability, roughness.
 - Compatibility with other materials and processes.
- **Simulation.** The first step consists in simulating light propagation in the waveguides, taking into account the different materials available for core and cladding. One crucial result of this step is to establish the **dimensions** of the waveguides so that they guide the desired modes.
 - **Design.** Once the simulations yield results on sizes, thicknesses and dimensions of features, the next step is to design the resulting structures in a format that can be eventually processed in the manufacturing steps. We have used AutoDesk® design suite, as its output is accepted by further processing steps.

The steps mentioned above are described in the following sections.

4.1.2 Devices simulation

The first step consists in simulating light propagation through the basic elements taking into account primarily the light wavelength and the materials refractive indices as main variables. Typically red (632,8 nm) or green (543,5 nm) laser sources are used. The specific refractive indices of the chosen materials must be taken into account, (treated appendix A) since they depended on the light wavelength. Table 4.1 summarizes the refractive employed indices of the

Table 4.1: *Refractive indices of SiO₂ and SU-8*

	Refractive index $\lambda=543.5\text{ nm}$	Refractive index $\lambda=632.8\text{ nm}$
Top	1.46025	1.45078
Core	1.6	1.59
Box	1.46025	1.45078

materials. Using 2D mode-solver code running in Matlab® a set of transverse core dimensions are obtained. The height and width of the core must only allow a few number of modes to propagate yet be large enough so for the light to be guided. The best simulation results were obtained for core width of 1 μm and height of 0.5 μm for both wavelengths. Nevertheless other aspect ratios were simulated for future essays.

The simulations were made for a ridge symmetric waveguide on a 3 μm substrate and with a 1.5 μm cladding. Symmetric means that same material was chosen for both, substrate and cladding, but this is not compulsory, in fact many times air is the option selected in uncovered guides.

In Figure 4.1 it can be seen that only two guided modes (first and third) exist while the rest of modes leak most of the power out the core. The same analysis was applied to the magnetic field (Figure 4.2). The magnetic field and electric field are conjugate so odd electric

Figure 4.1: First six transverse electric modes in a ridge waveguide at 632,8 nm. Both axes are expressed in μm .

Figure 4.2: First six transverse magnetic modes in a ridge waveguide at 632.8 nm. Both axes are expressed in μm .

Table 4.2: Simulated MMI length

MMI width [μm]	MMI 1x2 length red light [μm]	MMI 2x2 length red light [μm]	MMI 1x2 length green light [μm]	MMI 2x2 length green light [μm]
8	84	112	97.5	130
10	129.6	173	150.5	200.7
12	185	246.8	215	286.8
16	325	433	378.7	505
20	504	672	588.2	784.4

modes are correspond to even magnetic modes and *vice versa*. Thus the analysis reduces to the first three modes of the waveguide twice rather than six different modes.

Knowing the effective refractive indices for every chosen material at the concerned wave-

lengths, all the dimensions for each device can be mathematically calculated using the formulas already presented in the chapter introduction in section 1.2 on page 8.

To improve the simulations, 3D-physics modeling software COMSOL® was used. As a result it was found that small length adjustments should be made in order to ensure a good coupling in all developed models.

Table 4.3: *Simulated effective coupling length in directional*

Waveguide width [μm]	$d_c = 0.3$ [μm]	$d_c = 0.6$ [μm]	$d_c = 0.3$ [μm]	$d_c = 0.6$ [μm]
0.6	16	61.7	25.4	141.4
1	98	224.7	85.1	569.5
1.5	131	664.6	242.4	1752.6
2	277.6	1458.7	525	3917.6

Figure 4.3: (Top) *1 x 2 MMI simulated in COMSOL®; (Bottom) Simulated directional coupler.*

Not only waveguide were simulated in COMSOL® but also several devices such us MMI and directional couplers (Figure 4.3). The COMSOL® simulations result in several sets of dimensions for each configuration. Simulation models are shown for equal power distribution at the output. Table 4.2 represents simulated dimensions for equal intensity in both outputs of MMI couplers. Dimensions depends on the employed wavelength (632 nm in red, 543 nm in green), MMI width and number of light inputs (one or two).

Table 4.3 represents the simulated dimensions for equal intensity in both outputs of directional couplers, as function of waveguide width and distance between both waveguides.

4.1.3 Mask design

Chips of $2 \times 2 \text{ cm}$ with two different designs were made in the first mask design (Figure 4.4 left). In order to optimize the space over a 5×5 inches mask as much as possible, in the first design mask the total space was divided in four sections, each section, dedicated to a different waveguide width ($0.6 \mu\text{m}$, $1 \mu\text{m}$, $1.5 \mu\text{m}$, $2 \mu\text{m}$). Each of this sections has four chips: two designs, each of them for two wavelengths (632 nm and 543 nm); in total 16 different designs (2 designs $\times$ 2 wavelengths $\times$ 4 waveguides width). In one of the design (Figure 4.5) there are different sizes of MMI couplers and in the other MZIs, ring resonators and directional couplers. Not all these structures have been already characterized.

Figure 4.4: First and second mask design in AutoCAD [®].

In the second mask (Figure 4.4 right), the chip size was reduced from $2 \times 2 \text{ cm}$ to $1 \times 1 \text{ cm}$, increasing the total number of chips from 16 to 52 in a single mask. This time the mask was

Figure 4.5: Detailed view of a single chip on the two mask designs.

intended to be used on a circular silicon wafer so marks were employed at the edge facilitating

the alignment process. Also cross-shape guides were located in every chip corner to improve the cutting. Since the number of chips was large, combining different sort of structures in the same chip was no longer needed, even replication of identical chips was performed.

Script was made to create each device and to setup each position in the chip. In the first design, this program was made in Excel® and in the second mask the script was written in Matlab®.

Special care must be taken with the curvature of the waveguides to avoid light leakage by bending losses (Figure 4.6). Designed curves have a minimum bend radius of $150\ \mu m$

Figure 4.6: *Bending examples of different angles (180° and 30°).*

Finally all polygons were drawn in Autocad® as closed *polylines* as a single solid structure than can be interpreted by a laser beam pattern generator (.gds extension in this case). Both first and second designs were sent to a chromium photo-mask supplier that only partially achieved the desired resolution. Even so, the masks were good enough to start the photolithography process.

4.1.4 Waveguides and couplers manufacturing

4.1.4.1 Manufacturing protocols

Several manufacturing protocols have been employed in this context. In some cases the original protocols have been modified to meet the characteristics for the different waveguides. Two substrates have been employed for the waveguides: ITO coated glass and silicon wafers. The protocols are summarized in Figure 4.7.

The process includes several procedures previously described in detail:

- Substrate preparation (section 2.1.2.1 on page 27).
The glass size was $9 \times 9\ cm$, and silicon wafers were $10\ cm$ in diameter.
- **Sputtering** is a technique used to deposit thin films of a material onto a surface ("substrate"). Firstly a gaseous plasma is generated. Secondly, the ions from this plasma are accelerated into a source material ("target"). The source material is eroded by the

Figure 4.7: *Waveguides manufacturing protocols.*

impacting ions via energy transfer and is ejected in the form of neutral particles - either individual atoms, clusters of atoms or molecules. As these neutral particles are ejected they will travel in a straight line until they come into contact with something - other particles or a nearby surface. If a substrate such as a Si wafer is placed in the path of these ejected particles it will get coated by a thin film of the source material [89].

In the experimental samples, the sputtering is used for 1-10 μm SiO_2 coating, which is too thick for evaporation as described in section 3.3.1.2 on page 68.

The first layer is 3 μm of SiO_2 on top of the substrate Si or ITO covered glass.

- Spin SU-8 on SiO_2 with the parameters can be found in next section.
- Evaporate the solvent on a hot plate (2 *min* @ 95 °C).
- Make the photo-lithographic process (section 3.3.1 on page 63).
The exposure time in Si substrates is 30 *s*. In glass substrates, it is necessary to increase it up to 45 *s*.
- Soft bake on hotplate (2 *min* @ 95 °C).
- Develop (30 *s* in used developer followed by 10 *s* in new developer and followed by 10 *s* in isopropanol).
- Deposit 1.5 μm cladding of SiO_2 by sputtering.
- Cut into dice.

4.1.4.2 SU-8 deposition

As mentioned above, a number of materials have been tested for optimization. SU-8 has been studied for core material on several substrates.

To get the waveguides with thickness of $0.5 \mu\text{m}$, SU-8 is used as photoresist on different substrates with different spin-coating speed and concentration. With each different substrates, the adherence between waveguides and substrate changes so that the thickness is different. The concentration is changed by mixing cyclopentanone (CP) with SU-8 2001. The viscosity of any mixture will differ from the original.

1. **SU-8 on SiO_2 .** Changing the spin-coating speed from 1000 to 3000 *r.p.m.* with pure SU-8 on SiO_2 , the obtained thicknesses are shown in the Figure 4.8. Black points are the

Figure 4.8: *The thickness of waveguides with different spin-coating speed over SiO_2 .*

measured thicknesses data and red points are the average numbers of the data. A red dashed line indicates the tendency that fit with an exponential function. The higher the

Figure 4.9: *Waveguides thickness with different CP % over SiO_2 at 3000 *r.p.m.*.*

spin-coating speed is, the thinner layers we get. However, the thickness of waveguides

at 3000 *r.p.m.* is around $1.1\ \mu\text{m}$, which is too thick for single mode. It is necessary to change the concentration to adjust the viscosity that influences the thickness.

Keeping constant the spin-coating speed at 3000 *r.p.m.*, SU-8 thickness on SiO_2 can be modified by changing the percentage of CP. Figure 4.9 shows the results. As seen in the figure, a $0.5\ \mu\text{m}$ thickness can be obtained using 33% CP.

2. **SU-8 over ITO.** Repeating the same steps as above, using ITO as substrate gives the result shown in Figure 4.10 and Figure 4.11. Through the graph, it can be observed that

Figure 4.10: *The thickness of waveguides with different spin-coating speed on ITO.)*

the thickness does not change significantly with spin-coating speed -even using higher speeds than above. The difference is $0.2\ \mu\text{m}$ from 1000 *r.p.m.* to 7500 *r.p.m.*. The

Figure 4.11: *Waveguides thickness with different CP % on ITO at 3000 *r.p.m.*.*

variation is significant, however, when varying the concentration. A $0.5\ \mu\text{m}$ thickness is obtained when the CP percent is about 40%.

4.1.4.3 Waveguides manufacturing results

The process to obtain waveguides using SU-8 involves several steps that were presented in section 4.1.4 on page 92. In order to achieve the best results, many trials have been made to optimize the method. We will focus on the influence of material, exposure, baking, and developing respectively.

The results of material deposition in section 4.1.4.2 on page 94, show that both the substrate material and the photoresist affect the thickness of waveguides. Thus, to obtain $0.5\ \mu\text{m}$ waveguides, a SiO_2 substrate and a 33% CP SU-8 are used. Besides, the spin-coating speed is fixed at 3000 *r.p.m.*.

The exposure time was optimized as well in several attempts to an exposure power of $4\ \text{mW}/\text{cm}^2$. It is found that the optimal exposure dose depends on the film thickness (thicker films needs higher doses). For a $0.5\ \mu\text{m}$ thickness, 30 *s* on silicon and 45 *s* on glass is a good choice for exposure.

There are two instruments used for baking, oven and hot plate. Both of them can be used to evaporate the solvent and pre-cure the film after the resist has been applied to the substrate. However, it takes much longer time in oven and the effect is not as good. Therefore, hot plate is the preferred option. Baking temperature and time also need to be strictly controlled. Especially for soft bake, if the time is too short, the photoresist is not solid enough easily suffering scratches when handling. Through several attempts, we found 2 minutes at $95\ ^\circ\text{C}$ for soft bake and 1 minute at $95\ ^\circ\text{C}$ for post bake is the best process. A short time interval between exposure and post bake is required otherwise the cured part would not be strong enough and will be easily removed in developing.

For developing, it is best to use the commercial developer for SU-8 photoresist. Other solvent-based developers can also be used. In our project, we use Propylene glycol monomethyl (PGMEA) as developer. Time for developing should be fixed strictly, since too long time will cause some part of waveguides be broken (Figure 4.13 (a)). while too short may result in the photoresist not be fully removed (Figure 4.13 (b)).

Figure 4.12: Pattern through photolithography over SU-8.

(a)

(b)

(c)

Figure 4.13: *Some imperfections example: (a) The waveguide is broken because of too long developing time. (b) Photoresist is not clearly removed because of too short developing time. (c) The waveguide is not straight due to insufficient adhesion to the substrate.*

After several attempts, we optimized the developing time as 40 seconds (30 s in used PGMEA and 10 s in clean PGMEA). In this case, the width of waveguide is as designed and the edge is smooth (Figure 4.12). Another thing we need to pay attention is the adherence between substrate and photoresist. If it is not strong enough, the waveguide would not be straight adopting a wavy shape (Figure 4.13 (c)).

Figure 4.14: *Final chips on glass and silicon substrate. Silicon chip with LCs integration can be observed in the right picture.*

4.1.5 Chip characterization

Once the fabrication process is finished, wafers are cut in small chips (Figure 4.14). Chips that are in best condition are picked, seeing their surface through a microscope and avoiding any imperfection or dirt. They are then characterized one by one in a dark room with the suitable instrumentation.

All the setup in the dark room, is situated on an optical table in order to prevent from vibrations and also to keep the optical elements aligned over time. A base of aluminum was designed to elevate the microscope objective and to align it to the chip ports and the rest of the setup.

Two He-Ne laser of different wavelength light emission were available, 543.5 nm and 632.8 nm.

After the focal point of the laser beam is found on the microscope objective, where the light is most concentrated, the laser spot is aligned with the chip. The movements need to be highly accurate so nanositioners are needed. As the height of the waveguides is

Figure 4.15: *Dark room equipment assembly for the chip characterization*

$0.5\ \mu\text{m}$, slight variations make the difference between light coupling inside to core or not. A Thorlabs® NanoMax 300 stage is used for 3-axis positioning. More information can be found in the Appendix B on page 129.

A digital microscope is a variation of a traditional optical microscope that uses optics and a digital camera to output an image to a monitor, sometimes by means of software running on a computer. A digital microscope often has its own built-in LED light source, and differs from an optical microscope in that there is no provision to observe the sample directly through an eyepiece. We use two digital microscopes in the experimental setup to observe the chips, one for input ports, and the other for output ports. Using the digital microscopes, we can image the light coupling at the input and at the output can be imaged simultaneously.

4.1.5.1 Characterization method

The lasers were utilized as input source of the photonic circuits, pointing the laser beam, previously focused in a microscope objective, to the waveguide taper, which is a spot of about $1\ \mu\text{m}$ diameter. The first step for measuring is to align the light source to the edge of the chip and place the focal point of the microscope objective over its surface. Micropositioners have range and precision enough to accomplish this step. A chip rotation may be needed in such a way the laser beam and the waveguides are parallel. Adjusting the nanopositioner x-axis, the ray is aimed to the input port and using the piezoelectric z-axis the ray is moved from the cladding to the substrate until light impinges the core. It can be observed the ray bouncing inside the taper and passing through the waveguide.

All the characterization system is explained in the appendix B on page 129.

4.1.5.2 Characterization results

Figure 4.16 show a straight waveguide input that is divided into two straight outputs by a MMI 2×2 . Two bright spots can be recognized which means light is successfully guided along

Figure 4.16: *Inputs, outputs and guiding light through a PIC.*

the chip. It can also be observed that light intensity in each output is not equal, as expected, but this fact can be solved by carefully adjusting the nanopositioners to optimize the input spot position. Since this spot is so small, temperature variation can induce the misalignment between the source beam and the input port and hence modify the result seen within a couple

of minutes so a thermally stabilized hood must be considered for the future. There are also some device dimensions, such as the MMI length or the directional couplers gap, that are very sensitive to the thermal variations because its expansion or contraction causes a shift in the output power ratio.

Knowing that temperature [90] can modify the optical properties of the chip, a heat source was applied to its surface to verify this phenomenon. We showed that our 2×2 MMI structures can be tuned by temperature. The chip recovered its initial state have the same temperature is reached.

4.1.6 PIC-LC cell manufacturing

The changes due to temperature motivated us for the next experiment. In this experiment LC is added to achieve the change in light at the output. So, an LC sandwich was made with a PIC and a ITO cover glass. The LC refractive index change when a electric field is applied between the substrate and the cover glass.

The aim of this protocol is to fabricate an LC cell that changes the properties of the waveguides with an external electrical signal. For this purpose, the alignment of LC must be well defined either perpendicular or parallel to the waveguides. In the following we only discuss LC cell manufacturing on Si, although it could as easily have been done on ITO coated glass. Figure 4.17 shows a typical LC cell structure on a silicon base substrate.

Figure 4.17: *Scheme of a LC cell.*

The cell manufacturing in this device is similar to that described in section 3.3.1.3 on page 69.

4.1.6.1 Alignment layers

This process is intended to make a conditioning layer to align LC properly. Alignment layers are made of thin layers of polyimide onto the glass and SiO_2 layers deposited obliquely by thermal evaporation or e-gun deposition onto the SiO_2 cladding. The SiO_2 layers induce orientation of the liquid crystal when the target is tilted with respect to the evaporation source. The optimum angle for deposition was previously obtained in other protocol [91] and is not discussed here.

Procedure for SiO_2 (PVD is explained in section 3.3.1.2 on page 68):

- SiO_2 (50 nm) was evaporated tilting the substrate in order to achieve an angle of incidence of $45^\circ @ 1\text{\AA}/s$.

Procedure for polyimide is explained in section 3.3.1.3 on page 69.

4.1.6.2 Spacers deposition and assembly

The spacers deposition aims to ensure correct and constant distance between two glasses so that LC could be filled. The procedure does not match the description in section 3.3.1.3 on page 70; in this case the spacers are suspended in the NOA-81 gasket sealing the cell to avoid spacers on waveguide itself.

Procedure:

- Use the ultrasonic bath or the magnetic stirrer to mix spacers of suitable size with NOA-81 for several minutes.
- NOA-81 with spacers in suspension is carefully deposited in non-critical areas in as small droplets as possible.
- Assemble two substrates together, paying attention to choose the alignment direction of these two substrates in anti-parallel manner.
- Place it under UV light for a few minutes to cure the adhesive.

4.1.6.3 Filling and sealing

This is the main process in LC cell manufacturing. It is explained in section 3.3.1.3 on page 70

It can be summarized as:

- Place LC into the overlapping area of two substrates from one side of the cell with a pipette. Wait for a moment in order to ensure LC fills the cell completely.
- Seal all four sides of the cell with NOA-81.
- Press the edge to ensure a complete sealing.
- Put it under the UV light for several minutes to cure and make the sealing strong enough.

4.1.6.4 Connections

The last step is the connection of the cables to the substrates. It is explained in section 3.3.1.3 on page 71.

Figure 4.18: *Outputs of a LC-MMI PIC device. Top image shows the ON state. Bottom image shows the OFF state.*

4.1.6.5 PICs+LC characterization results

The characterization has been performed in the same way as in the previous experiments (chip characterization), but applying electric field.

Figure 4.18 show the two outputs of the chip, top image show when the electric field is applied and more power can be shown in the left output. Bottom image show when no electric field is applied and the outputs are approximately 50%-50% .

Figure 4.19: *Relative power of the outputs during ten seconds when the frequency of the electric field is 0.45 Hz.*

Figure 4.19 show the measured power at the outputs of the chip during 10 s. The frequency of the electric field signal is 0.45hz. The power of the output change between 2 states. ON state (applied electric field) 70%-30% and OFF state (no electric field) 53%-47%.

4.2 Polarization manipulation PIC

Polarization manipulating devices are not only important for polarization multiplexing and compensation but also for polarization-transparent PICs[61]. In the next sections we will

present the design and simulation followed by manufacturing and characterization of novel polarization switch manufactured in a polymer platform employing a cost-efficient laser direct-write lithography (DWL). The active element is created by including a cavity filled with a nematic LC. The cavity is crossing two parallel waveguides.

Eventually, the design and simulation of a switching polarization splitter with the same working principle as the polarization switch are presented.

4.2.1 Design and simulation

Figure 4.20: Principle drawing of the polarization switch with the two possible switching states of the LC intersection highlighted. The trench filled with LC forms an angle $\alpha=30^\circ$ with the waveguides.

4.2.1.1 Theoretical design

The device consists of two parallel polymeric waveguides made with Epocore ($n_{core}=1.590$) and Epoclad ($n_{clad}=1.578$) [92] (Microresist Technology) which are interrupted with a trench filled with LC. The trench crosses the waveguides at a specific angle to allow polarization dependent switching depending on the LC orientation (Figure 4.20). Visible light was chosen for that application (630 nm) but the device can also be designed for other wavelength ranges. The LC (MDA-98-1602, Merck KGaA) ($n_e=1.78$ and $n_o=1.52$) was chosen in order to split the light in the two planar polarizations *i.e.* parallel and perpendicular to the substrate.

Figure 4.21: The displacement of a light beam in a plane-parallel slab.

Simulations were done in order to determine optimal dimensions of the LC intersection. As a first approximation, a plane-parallel intersection, a *slab*, was simulated including the losses caused by the refractive index variation between the polymer waveguides and the LC. In accordance with Snell's law, the beam displacement d caused by the insertion of a plane-parallel slab of height h , with refractive index n_{LC} , in a medium with an effective refractive index of mode in the waveguide n_{wg} , which can be calculated from the waveguide dimensions, n_{core} and n_{clad} , at an angle θ_{wg} can be derived as Eq. (4.1)(Figure 4.21). The n_{wg} in this case will be the effective refractive index of the guided mode, which can be calculated using a modesolver. However, in this context it can be approximated to $1.58 \approx n_{clad} \approx n_{core}$, as will be discussed below.

In order to estimate the losses in the device, it is necessary to calculate the magnitude of the reflection at the guide-LC interfaces. At oblique incidence the magnitude of the reflection depends on the polarization of the incident light, *i.e.* whether the light is polarized parallel to the plane of incidence (P-polarization) or perpendicular hereto (S-polarization), or a linear combination of P and S polarizations. It is important to notice at this point that in conventional optics P and S nomenclature is used as pseudonyms for TM and TE, while the convention when designing pics is that TM describes light that is polarized with its electric field perpendicular to the substrate, while TE describes light that is polarized with its electric field parallel to the substrate.

Figure 4.22: Simulated displacement and reflection produced by the plane-parallel slab as a function of trench angle α . Blue dashed line: the displacement (d). Green dashed line: the TM polarized light losses (Γ_{TM}). Red continuous line: the TE polarized light losses (Γ_{TE}).

The loss Γ caused by Fresnel reflections at the surfaces for the two polarizations can be cast as Eq. (4.2,4.3) [93]. The value of the trench width (h) is dictated to $20 \mu\text{m}$ by the fabrication process.

$$d = h \frac{\sin(\theta_{wg} - \theta_{LC})}{\cos(\theta_{LC})}; \theta_{LC} = \sin^{-1} \left(\frac{n_{wg}}{n_{LC}} \sin(\theta_{wg}) \right) \quad (4.1)$$

$$R_{TM} = \left| \frac{n_{wg} \cos(\theta_{wg}) - n_{LC} \cos(\theta_{LC})}{n_{wg} \cos(\theta_{wg}) + n_{LC} \cos(\theta_{LC})} \right|^2 ; \Gamma_{TM} = 1 - (1 - R_{TM})^2 \quad (4.2)$$

$$R_{TE} = \left| \frac{n_{wg} \cos(\theta_{LC}) - n_{LC} \cos(\theta_{wg})}{n_{wg} \cos(\theta_{LC}) + n_{LC} \cos(\theta_{wg})} \right|^2 ; \Gamma_{TE} = 1 - (1 - R_{TE})^2 \quad (4.3)$$

The displacement and reflection coefficients for a simulated thickness of 20 μm and a refractive index of 1.58-1.78 as a function of angle α are shown in Figure 4.22.

A minimum reflection of 2.7% is found at 42°, but a compromise between the losses due to reflection and the displacement of the light has to be made and thus 30° was chosen as the best compromise. At this angle the losses are 5.9% while the displacement is 5.1 μm . At angles smaller than 30° the losses increase exponentially.

The employed preferred alignment of the LCs is in plane with the substrate and perpendicular to the trench, while the field induced alignment is perpendicular to the substrate. (Figure 4.20).

4.2.1.2 Finite difference time domain (FDTD) simulation

To complement the above analytical characterization of the initial design, a Lumerical FDTD Solutions finite element simulation was performed, employing ordinary and extraordinary refractive indices of LCs of $n_o=1.52$ and $n_e=1.78$, respectively. The simulated thickness of the slab, h , was 20 μm , as above. Being optically anisotropic, both the waveguide design and the LC simulation had to be done for the two perpendicular light polarizations in-plane (TE) and out-of-plane (TM) with the substrate, as well as for the two different switching states of the LC: OFF-aligned in the plane of the substrate and ON-aligned in the plane perpendicular to the substrate.

In Figure 4.23 the simulated light progression for both polarizations, in the two switching states of the LC, is shown, for a waveguides separation of 5 μm .

In the switched state, the TM polarization propagates with an effective refractive index n_e since the liquid crystal is aligned with the plane applied perpendicularly to the substrate plane. The displacement can thus be simulated to a high accuracy, employing an exact value for the refractive index, *i.e.* $n_e=1.78$. The TE polarization propagates with n_o and since the $n_o < n_{wg}$, the power in this polarization is refracted away from the output waveguide, upwards in the figure.

In the relaxed state, the TM polarization propagates with n_o and is thus refracted away from the output waveguide. The TE polarization experiences a refractive index n_{eff} , which is between and n_o since the light impinges obliquely to the direction of the LC molecules, see Eq. (4.4). In the case of the design parameters ($90^\circ - \theta = \alpha = 30^\circ$ and alignment as in Figure 4.20) n_{eff} was calculated to be 1.70. Although this value differs from n_e , the numerical simulation shows that also in this case most of the light is coupled out into the output waveguide (see Figure 4.23). Using the same design parameters in the analytical approach, leads to significant variation in displacement, *i.e.* 3.8 μm in the case of the TE polarization in the OFF state and

Figure 4.23: (a) Scheme of the simulated device with approximate aspect ratio. Various simulations were performed, all with a fixed slab height (h) of $20 \mu m$, trench angle α of 30° , waveguides of $3 \times 3 \mu m$, but with separations of the waveguides between 3 and $7 \mu m$; (b) Simulation of the total power and TE, TM field distributions in the central waveguide section for switched and unswitched device with $5 \mu m$ waveguide separation. Notice that the aspect ratio is not conserved in the simulation images.

$5.3 \mu m$ for the TM polarization in the ON state.

$$n_{eff}^2(\theta) = \frac{n_e^2 n_o^2}{n_o^2 \sin^2 \theta + n_e^2 \cos^2 \theta} \quad (4.4)$$

Thus in accordance with this simulation, TM or TE polarization can be transmitted switching the LC ON and OFF.

It is worth while to notice the use of only 2 decimals in the estimation of both n_{wg} , n_o , n_e and n_{eff} , since the displacement (d) of the light beam in the LC, is practically insensitive to any variation in the third decimal of the refractive index, as can be appreciated even in the analytical expression (Figure 4.24). The small deviations, *i.e.* less than $100 nm$ per 0.001 variation in refractive indices, are easily compensated by a taper structure in the waveguide coupling at the trench. Thus the chromatic dispersion of both the waveguide and the LC will only have marginal importance for the final device since the refractive indices of both the LC and the waveguide will tend to vary in the same manner.

Figure 4.24: Simulated displacement and reflection produced by the plane-parallel slab as a function of n_{LC} .

4.2.2 Fabrication

An LC cell is constructed using a transparent ITO coated glass as top electrode with SiO_2 alignment layer and a metalized silicon wafer as bottom electrode. On top of the silicon wafer multiple devices are manufactured in parallel in the polymer layer indicated in Figure 4.25. The cross section of each waveguide is $3 \times 3 \mu\text{m}$ ($n_{TE} \approx n_{TM} \approx 1.585$). In each of the devices a $20 \mu\text{m}$ trench is ablated as indicated by the two red parallel lines. Prior to assembly, the polymer layer is covered by a SiO_2 alignment layer. When filling the assembled cell, all trenches are filled by the LC.

Figure 4.25: The basic structure of the developed devices. The different devices are characterized by a separation, $d \in [3.9 - 5.5] \mu\text{m}$, of the waveguides in the active area. Prior to assembly, the polymer layer is covered by a SiO_2 alignment layer. When filling the assembled cell, all trenches are filled by the LC. The input and output ports are indicated.

Taking into account manufacturing tolerances for experimentally (systematically) verifying the influence of the waveguide separation on the switch operation, a series of devices with varying waveguide separations were designed and manufactured; 20 devices with waveguide separations (d) varying from 3.5 to 5.5 μm were designed.

4.2.2.1 Waveguides manufacturing

The device consists of a pair of parallel polymeric waveguides ($n_{\text{core}}=1.590$) embedded in a polymeric cladding ($n_{\text{clad}}=1.578$). The polymers employed are Epocore/Epoclad [92] (Microresist Technology). An excimer laser (KrF, operating wavelength 248 nm, pulse duration 5-8 ns, pulse energy 570 μJ) was used to make the trench. In the final device the trench was filled with the LC MDA-98-1602 ($n_e=1.78$ and $n_o=1.52$).

Figure 4.26: Steps to fabricate the waveguides

The process flow is shown in Figure 4.26. and can be described as follows; before depositing the optical material, the silicon wafer is rinsed in acetone, a mix of acetone and isopropanol and double distilled water. Subsequently it is dried for 20 min on a hotplate a 150 °C. After cleaning, a plasma-treatment is done to improve the adhesion of cladding to the substrate. Then a 16 μm lower-cladding $n_{\text{clad}} = 1.578$ layer is applied by spin coating, pre-baked on a hotplate and cured with UV light. Hard baking follows the UV curing. Air plasma-treatment was repeated before depositing all layers to improve interlayer adhesion. A 3 μm core layer is deposited by spin coating and pre-baked.

A critical step follows the pre-bake, *i.e.* the Laser Direct-Write Lithography (LDW). The waveguides are directly written with a laser using the system DWL-66fs from Heidelberg Instruments with a continuous UV laser ($\lambda=355\text{nm}$, Genesis CX STM). After LDW the core is post-baked and developed. After the core development hard-baking is repeated. The final upper-cladding layer is deposited using the same steps as the lower-cladding.

Table 4.4 summarizes the optimized process parameters for the fabrication of the waveguides.

Table 4.4: *Optimized process parameters for the waveguides fabrication*

Process Step	Epoclad 50	Epocore 2
Spin coating	5000 <i>r.p.m.</i> 1'	1900 <i>r.p.m.</i> 30"
Soft bake	5'@50 °C, 5'@90 °C, 10'@120 °C	5'@50 °C, 5'@90 °C, 10'@120 °C
UV exposure	1'@10 <i>mW/cm</i> ²	DWL @ 334 <i>mJ/cm</i> ²
Post exposure bake	15'@90 °C	2'@50 °C, 4'@90 °C
Wet development	-	45" in mr-Dev600, 1' rinse IPA
Hard bake	90'@120 °C in oven	90'@120 °C in oven

4.2.2.2 Cavity ablation

Once the waveguides are manufactured, the trench is made directly in the polymer sandwich by ablation. An excimer laser was used to ablate the complete stack of cladding-core-cladding. The excimer laser beam passed through a rectangular mask ($2000\ \mu\text{m} \times 200\ \mu\text{m}$) and the de-magnified image (factor of 10) of the mask was projected onto the polymer coated sample, this technic has been optimized in the past to make angular cuts [94]. With a ablation rate of $0.45\ \mu\text{m}/\text{pulse}$ 70 pulses were applied to make the desired trench in waveguide core.

4.2.2.3 LC assembly

The last step to finish the device is creating the LC cell. In order to establish the preferred orientation in absence of any external field a silicon dioxide (SiO_2) layer was deposited by physical evaporation deposition ($50\ \text{nm}$ with an angle of 45°) as an alignment layer on the polymer layer and trenches and on the top electrode [95]. Spacers of $5\ \mu\text{m}$ were deposited onto the alignment layer of the top surface, so that once assembled the LC could flow freely and fill all the trenches of each of the devices on the chip. A gasket seal is applied leaving a mouth open for the filling of the cell with the LC material. Finally, the cell is filled with LC under vacuum, as described in [1], leading to the final macroscopic structure (Figure 4.25).

4.2.2.4 Structural measurement

SEM was employed to assess the quality of the waveguides, see Figure 4.27.a. These are well defined rectangular shapes. The roughness of the bottom and the top cladding has been measured previously and turned out $2.2\ \text{nm}$ [92].

As a result of unexpected high losses, the quality of the trench cut was evaluated. A trench angle with respect to the waveguide of 32° was measured (Figure 4.27.b), which was 2° rotation with respect to the design. Furthermore, the cross-section of the trench (Figure 4.27.c) revealed that the cut was not a rectangular section perpendicular to the substrate surface, but rather a V-shaped cut. For wider laser-ablated trenches, typically only $5\text{--}10^\circ$ deviation from 90° cuts are expected [94], but in our case the trenches came out with almost 25° deviation

Figure 4.27: a) SEM image of the a waveguide without cladding; b) optical microscope image of a trench. c) optical microscope image of the cross section of a trench.

(114° instead of 90°) due to the desired high aspect ratio and small trenches. The resulting trench is thus thinner and thus is the effective LC slab. At the height of the waveguide the LC slab measures is only $11.3 \mu m$, $8.7 \mu m$ less than the desired value. As a result a smaller displacement is expected and thus the devices with smaller waveguide separations are expected to give the best results. Additional losses were added by the not normal ablation (~ 3 dB for the TM and ~ 1 dB for the TE).

4.2.3 Optical characterization

As mentioned before, several devices were made on the same chip to compensate for manufacturing tolerances. The devices differ in the distance between the waveguides at the position of the LC slab. Devices with separations ranging from $0.9 \mu m$ to $2.5 \mu m$ i.e. this is the waveguide pitch ($[3.9-5.5] \mu m$ between center of the waveguides) were manufactured successfully in steps of $0.1 \mu m$.

Two chips with two sets of the design each were characterized. No noteworthy difference between those four devices were measured.

A polarized laser diode emitting at $630 nm$ coupled to a single-mode fiber reel was used in the characterization of the device. Using a nano-positioner the light was coupled into the waveguide. A fiber rotator was used to manipulate the polarization at the input of the device. The light at the device output is captured using a multi-mode fiber and a photo-detector.

Figure 4.28 shows the relative power transmission versus waveguides separation for the two different input polarizations (TM, TE) both with the LC switched (V_{on}) and unswitched (V_{off}) for the output waveguide.

The device works as a polarization switch. When the applied voltage is off the TE polarization is guided to the output port. When the applied voltage is on the TM mode is guided to the output port. Each of the polarizations have an optimal waveguide separation for maximum transmission to the output port. This is due to the fact that the effective refractive index of the LCs for the TE is smaller than that for the TM ($n_{eff,TE} < n_{eff,TM} \approx n_e$). The geometry means that the unswitched LCs alignment is not perpendicular to the TE mode propagation (see Figure 4.20), however after switching the LCs alignment is almost perpendicular to the TM mode propagation. The slight deviation (less than 3°) is caused by the non-vertical trench sides.

Figure 4.28: *TM/TE intensities at the output port for the two different LC switching states. The measured values allowed for an estimation of the effective refractive indices, $n_{eff_{TM}}$ and $n_{eff_{TE}}$, for the on and the off switching states. These derived values were used in the calculation of the simulated transmission curves. The measured intensities are relative to the power transmitted in a straight waveguide.*

Waveguide separation of $4.9 \mu\text{m}$ provides a measured power transmission of more than 15 % for both polarizations when they are guided to the output port depending on the switching state of the LC. Simulations of the structure taking the trench imperfections into account were performed and the effective refractive indices for $TM_{V_{on}}$ and $TE_{V_{off}}$ were refined so that the simulated maximum transmission waveguide separation corresponded to the measured maximum transmission waveguide separation in the output: $n_{eff}(TM_{V_{on}}) = 1.72$ and $n_{eff}(TE_{V_{off}}) = 1.69$. In the case of $TM_{V_{off}}$ and $TE_{V_{on}}$, a refractive index of n_o has been employed. A reasonable fit between the simulated and the actually measured values was obtained (Figure 4.28).

With a perfectly rectangular trench the simulated loss was around 2 dB (The Theo curves in Figure 4.28 shows the power with the losses included with the actual configuration). The actual loss is ≈ 8 dB, this higher loss is due to the reflections in a not perpendicular surface and the mode expansion in the trench. This could be reduced making a perpendicular cut and including a taper in the *coupling-in-section* of the trench zone.

4.2.4 Proposed advanced devices

Based on the above device another switching polarization splitter is proposed.

By choosing an LC with an ordinary refractive index $n_o = n_{wg} = 1.58$ the same geometry will lead to a switching polarization splitter where the waveguide separations and slab height depend on n_e .

Figure 4.29 shows the simulation of the power distribution for the two polarizations at the two switching states in a device identical to the one above, with the LC in relaxed state aligned

Figure 4.29: Simulation of the designed wide wavelength switchable optical polarization splitter. The total power and TE, TM field distributions in the central waveguide section for switched and unswitched device with $6\ \mu\text{m}$ waveguide separation. The trench size was $20\ \mu\text{m}$ and the LC refractive indices varies between $n_o=1.58$ and $n_e=1.78$.

in plane with the substrate and perpendicular to the alignment in the above (see alignment in Figure 4.20(a)) ($n_e=1.78$, $n_o=1.58$, $\alpha = 30^\circ$ and $h=20\ \mu\text{m}$). The working principle is the same as above, but in this case the two polarizations are guided for both switching states.

Figure 4.30: The fundamental design of a switching polarization splitter using the MDA-98-1602. In this case neither of the two output ports can be aligned with the input port since $n_o < n_{wg}$.

Alternatively, a switching polarization splitter may be manufactured using discontinuous waveguides (Figure 4.30), while maintaining the LC and LC alignment as in the original design. In this case the trench positioning becomes critical since it has to be situated right at the discontinuity of the waveguides.

4.2.5 Relevant results of polarization manipulation PIC

A liquid crystal slab inserted in a tilted trench in the light path of two waveguides has been shown. This simple device has demonstrated several functionalities depending on the tilt angle, the refractive indices of the waveguides, the liquid crystal and the relative position of the output and input waveguides. Some examples are polarizer, optical switch and polarization switch.

These functionalities have been confirmed by simulations with Lumerical FDTD. Quite moderate losses (2 dB) are predicted, making the device potentially useful for practical applications.

Actual devices based on organic waveguides have been demonstrated in the visible region. Extension to NIR wavelengths is straightforward, since no restrictions in the spectral range -other than waveguide limits- are expected. NIR range is better attainable with inorganic waveguides; the system is fully compatible with these waveguides.

Photonic integrated circuits (PICs) have not yet reached the integration levels of electronic integrated circuits. Devices like the presented, making waveguides active, remarkably increase the PIC functionality with moderate increase of complexity. These devices could decisively contribute to the development of more integrated/sophisticated circuits.

Chapter 5

CONCLUSIONS AND FUTURE RESEARCH LINES

5.1 Overall conclusions

This PhD work has been focused on two research areas: phase devices based on liquid crystal technologies and waveguides. A number of novel phase and guide devices have been developed; all of them featuring novel kinds of electrodes, and/or new manufacturing methods for increased resolutions. In the course of the thesis a number of new manufacturing technologies have been developed and implemented in the CEMDATIC R&D center's clean room facilities.

1. Two different protocols were developed for the CNC-Laser:
 - (a) Laser ablation, in which the UV-laser is used to selectively evaporate materials. With this technology electrodes and waveguides have been fabricated.
 - (b) LDW lithography, where the UV laser is used to selectively photo-polymerize pre-polymers. The developed *mask-less* lithography has a theoretical resolution better than $1\ \mu m$.
2. A Nanoimprint Lithography (NIL) in-house made instrument [96] was designed and manufactured for nano-manufacturing. One instrument for P-NIL was manufactured using 3D-printing and another instrument was made in Al to allow for T-NIL. The NIL developed protocol improves the resolution in the CEMDATIC clean room down to $100\ nm$. Also, analytic software was developed for the characterization of the nano-imprinted surfaces. The software compares the copy with the master.
3. Several types of micro-lenses were manufactured, which can be separated into two different kinds classes: Tunable and fixed focal length.
 - (a) Fixed focal length micro-lenses were fabricated by NIL techniques. Several materials were tested. Errors of few nm were detected, with no relevance to micro-lenses.
 - (b) Different approaches for tunable micro-lenses were used. The manufactured laser ablation electrodes do not work because not enough resistance is introduced (there

is ITO in the hole pattern that conducts the electricity). The best results were obtained with the non-conducting hole pattern made by convectional photo-lithography. Micro-lenses with focal lengths as small as 2.6 *mm* were manufactured.

4. A reconfigurable liquid crystal optical vortex retarder plate was simulated, designed and manufactured. A 24 pie-slices reconfigurable optical vortex retarder plate was manufactured using laser ablation for the electrodes. Also a driver was designed and calibrated to apply the correct phase delay in every slice pie. Finally a comparison between the measures results and simulations was made.
5. Several PICs were designed, simulated and fabricated. Two novel devices were developed during this Ph.D. The integration of LCs, opens for the possibility of tuning not only with temperature, but also with an electric field.
 - (a) Several devices were fabricated with conventional photo-lithography. LC was selectively included in the cladding of the PICs. A tunable 2x2 MMI was designed, simulated and manufactured with a tuning range of the exit ports of (70/30)% to (53/47)%.
 - (b) A tunable polarization splitter was simulated, designed and fabricated. The viability of the device has resulted in a patent. The device has at least three different functionalities: polarizer, tunable splitter and switch.

5.2 Future lines

The present work leaves numerous research lines to be explored and questions to be resolved in a near future.

- The CNC-laser may be optimized to approximate the feature to the theoretical value, since the current resolution is twice that size. Fourier optics could be included, which could reduce the feature size further.
- The NIL manufacturing techniques can be applied for PICs fabrication. Thus sub-wavelength feature sizes can be implemented for new applications and couplings.
- Reconfigurable liquid crystal phase devices can be applied to other research areas such as fractal optics. Some of the future research of the group is heading in that direction.
- The PICs is one of the future research pillars in the CEMDATIC R&D center. In combination with LC countless devices and designs can be designed simulated and manufactured, as the logical continuation of this thesis.

5.3 Publications related to this work

5.3.1 International papers

1. Caño-García, M., Geday, M. A., Gil-Valverde, M., Megías Zarco, A., Otón, J. M., & (2018). A 3D-printed device for polymer nanoimprint lithography. *Review of Scientific Instruments*, 89(2), 025106.
2. Gil-Valverde, M., Caño-García, M., Delgado, R., Zuo, T., Otón, J. M., Quintana, X., & Geday, M. A. (2017). Polymer selective laser curing for integrated optical switches. *Photonics Letters of Poland*, 9(1), 32-34.
3. Poudereux, D., Caño-García, M., Alj, D., Caputo, R., Umeton, C., Geday, M. A., ... & Quintana, X. (2017). Recording Polycrystalline structures in photonic crystal fibers. *Photonics Letters of Poland*, 9(1), 5-7.
4. Algorri, J. F., Poudereux, D., García-Cámara, B., Urruchi, V., Caño-García, M., Vergaz, R., ... & Otón, J. M. (2016). Metal nanoparticles-PDMS nanocomposites for tunable optical filters and sensors. *Optical Data Processing and Storage*, 2(1).
5. Poudereux, D., Caño-García, M., Algorri, J. F., García-Cámara, B., Sánchez-Pena, J. M., Quintana, X., ... & Otón, J. M. (2015). Thermally tunable polarization by nanoparticle plasmonic resonance in photonic crystal fibers. *Optics express*, 23(22), 28935-28944.

5.3.2 Patents

1. Caño-García, M., Geday, M. A., Otón, J.M., Quintana, X. Elmogi, A., Mattelin, M., Missine, J., Van Steenverge, G. *Método y dispositivo reconfigurable para el guiado de una señal óptica con dos polarizaciones perpendiculares* (2018). Spanish Patent No. P201830088. (Registered).

5.3.3 Congress contributions

1. Caño-García, M. (2017) Técnicas de fabricación en cámara limpia para la realización de guía ondas poliméricas. At Universidad Carlos III de Madrid, SPAIN 3^a Jornadas de seminarios SINFOTON-CM
2. Caño-García, M., Quintana, X., Gil Valverde, M., Otón, J.M., & Geday, M. A. (2017). A reconfigurable liquid crystal optical vortex retarder plate. At Madrid, SPAIN Reunión de Jóvenes Investigadores SINFOTON: 3^a Feria de Otoño 2017
3. Pereiro, J., Geday, M. A., Caño-García, M., Otón, J.M., & Quintana, X. (2017). Fabricación de un microscopio de fuerza atómica empujando buzzers como actuadores piezo-eléctricos y un cabezal DVD (OPU) como detector óptico. At Madrid, SPAIN Reunión de Jóvenes Investigadores SINFOTON: 2^a Feria de Otoño 2017

4. Gil Valverde, M., Caño-García, M., Geday, M. A., Otón, J.M., & Quintana, X. (2016). A simple device for nanoscale polymer pattern copying. At Madrid, SPAIN *Reunión de Jóvenes Investigadores SINFOTON: 2ª Feria de Otoño 2016*
5. Caño-García, M., Gil Valverde, M., Geday, M. A., Otón, J.M., & Quintana, X. (2016). Liquid crystal modulators on photonic integrated circuits. At Madrid, SPAIN *Reunión de Jóvenes Investigadores SINFOTON: 2ª Feria de Otoño 2016*
6. M., Gil Valverde, Caño-García, M., Geday, M. A., Otón, J.M., & Quintana, X. (2016). Light coupling in microscale polymer photonic circuits. At Erice, ITALY *Ettore Majorana Center for Scientific Culture*
7. Budaszewski, D., Poudereux, D., Caño-García, M., Woliński, T. R., & Otón, J.M. (2016). Photonic crystal fibers infiltrated with metallic nanoparticles dispersed in polydimethylsiloxane. , At: Krynica-Zdrój, POLAND,, *Conference: XXI Conference on Liquid Crystals Chemistry, Physics and Applications CLC 2016*
8. Geday, M. A., Caño-García, M., Escolano, J. M., Otón, E., Otón, J.M., & Quintana, X. (2016). Detection of microbes using flowing lyotropic liquid crystals. At: Krynica-Zdrój, POLAND,, *Conference: XXI Conference on Liquid Crystals Chemistry, Physics and Applications CLC 2016*
9. Poudereux, D., Caño-García, M., Geday, M. A., Quintana, X., & Otón, J.M. (2016) Polarization dependent photonic liquid crystal fiber tunable interferometer. At: Krynica-Zdrój, POLAND,, *Conference: XXI Conference on Liquid Crystals Chemistry, Physics and Applications CLC 2016*
10. Caño-García, M., Poudereux Sánchez, D., Geday, M. A., Otón, J.M., & Quintana, X. (2016). Light coupling and performance of LC modulated photonic integrated circuits. At: Krynica-Zdrój, POLAND,, *Conference: XXI Conference on Liquid Crystals Chemistry, Physics and Applications CLC 2016*
11. Caño-García, M., Ye, F., Gordo, F., Geday, M. A., Otón, J.M., & Quintana, X. (2016). Active photonic integrated circuits modulated by LCs. At: Krynica-Zdrój, POLAND,, *Conference: XXI Conference on Liquid Crystals Chemistry, Physics and Applications CLC 2016*
12. Caño-García, M., Delgado, R., Zuo, T., Geday, M. A., Quintana, X., & Otón, J. M. (2015). Manufacturing arbitrarily-shaped active waveguides with liquid crystal cladding. At Sopot, POLAND, *Conference: Topical Meeting on the Optics of Liquid Crystals, Volume: 16th*

Appendix A

Materials

In this chapter, materials and technologies used in the Thesis are described. Materials are divided in five sections: material employed for machinery construction, intermediate materials employed in nanoimprint processes, materials employed in the creation of waveguides, materials for microstructured devices and other materials usually employed in fabrication of liquid crystal based devices.

A.1 Materials for machine development

Several instruments have been prepared in the lab to contribute to the preparation of devices. Besides these, many tools, handles and pieces for different manufacturing and characterization steps have been created as well. These home-made elements have been mainly realized in a mechanical workshop for metallic tools, by means of a CO₂ laser manufacturing facility for PMMA, Teflon and glass pieces, and with the help of a 3D printer from melted polymers. The materials most frequently employed in fabrication of elements have been:

Acrylonitrile butadiene styrene (ABS)

Acrylonitrile butadiene styrene (ABS) is a well-known amorphous thermoplastic polymer [78]. Its mechanical properties are superior to standard polystyrene, and can be manipulated by extrusion or by injection molding. In this work, ABS has been employed in 3D-printing. The following setup parameters have been used:

- Build-plate: 80°C.
- Nozzle: 245-260 °C.

Polylactic acid (PLA)

Poly(lactic acid) or polylactic acid or polylactide (PLA) is another common polymer, having the peculiar property of being fully biodegradable. It is made of natural products, such as

corn starch, tapioca roots or sugar cane. Like ABS, PLA is commonly used for 3D-printing of fused filament. Its tolerance to high temperatures, however, is much lower. This is often utilized in a technique called *lost PLA casting*, by which a mold made in PLA is encased with a thermal-resistant material and then burned out in an oven. The thermal resistant material forms a negative mold of the previous PLA mold; this can be eventually used to cast pieces of other materials, for example metals. In this work, ABS has also been employed in 3D-printing. The following setup parameters have been used:

- Build-plate: 60°C.
- Nozzle: 190-240 °C.

Aluminium

Most metal pieces created along this work have been made in Aluminum, that results soft enough to be easily mechanizable. Moreover shows excellent thermal and optical properties. Aluminum has been chiefly employed in this Thesis for three different tasks:

- As a mirror, deposited by thermal evaporation.
- As an opaque electrode in microlenses.
- In the workshop to construct metal pieces. For example, the nanoimprint unit developed in this Thesis has been realized in two versions, one in polymer, created in the 3D-printed, and another one in aluminum, prepared in the workshop

A.2 Intermediate materials employed in nanoimprint processes

Polydimethylsiloxane (PDMS)

Polydimethylsiloxane (PDMS) is a polymeric silicone [97], the most extended silicon-based organic polymer. In Photonics, PDMS is particularly interesting for its optical transparency, ranging from near infrared to a fraction of the UV spectrum. For example, it is the preferred material for contact lenses, and finds many uses in optical communications.

PDMS is commonly used as a stamp resin in the procedure of soft lithography, making it one of the most common materials used for flow delivery in microfluidics chips [98]. The process of soft lithography consists of creating an elastic stamp, which enables the transfer of patterns of only a few nanometers in size onto glass, silicon or polymer surfaces. With this type of technique, it is possible to produce devices that can be used in the areas of optical telecommunications or biomedical research. The stamp is produced from the normal techniques of photolithography or electron-beam lithography. The resolution depends on the mask used and can reach 6 nm [99].

In our work, PDMS shall be used as intermediate step to transfer nanosized features from a master to polymer copies, i.e., as a negative mold stamp.

Trichoro(1H,1H,2H,2H-perfluoro-octyl)silane

Perfluorodecyltrichlorosilane (FDTs) material forms self-assembled monolayers that are useful as antiadhesive layers by lowering the surface energy. Wafers used to fabricate PDMS pieces are treated with self-assembled monolayers of FDTs, forming an antiadhesive layer that allows easy release of cured PDMS.

The FDTs monolayer is obtained by moderate vacuum evaporation of the liquid in a vacuum chamber.

A.3 Materials for the creation of waveguides

Materials for waveguides must meet a number of properties both, in mechanical and in the optical side.

The most important property for the fabrication of a waveguide is the refractive index of the materials. Table A.1 show the refractive index of the materials that it possible to find in the R&D center CEMDATIC. All the materials described in this section have being used to fabricate waveguides. The refractive index changes with the frequency of the light.

Table A.1: *Refractive index of different materials @ 588 nm*

Material	Refractive index (n_D)
PDMS	1.394
SiO ₂	1.477
PMMA	1.49
OrmoStamp	1.52
OrmoComp	1.525
OrmoCore / OrmoClad	1.56 - 1.544
NOA-81	1.56
EpoCore / EpoClad	1.61 - 1.587
SU-8	1.62
Microposit 1818	1.65

OrmoCore/OrmoClad

Ormocore and *Ormoclad* are inorganic-organic hybrid polymers for planar optical wave guiding and micro optical applications. The solvent-free ready-to-use formulations can be processed by UV lithography or Photo Nanoimprint Lithography (P-NIL). After processing OrmoCore and OrmoClad show:

- Low optical loss at optical communication IR windows wavelength and also in the visible range.
- Resolution upto sub-50 nm pattern dimensions.

- Chemical and physical stability.
- Excellent mechanical properties.

Ormocore and Ormoclad are typically used for core and clad fabrication of waveguides. They are very similar in chemical composition and can be mixed to obtain intermediate indices.

OrmoPrime

OrmoPrime is an adhesion promoter solution based on organofunctional silanes. It has been designed to enhance the adhesion of hybrid polymer products of Ormo Family to various substrates like silicon, glass and quartz.

It is provided as a ready-to-use solution for spin coating.

SU-8

SU-8 is a negative, epoxy based, near-UV photoresist designed for MEMS and other microelectronic applications. When *SU-8* is exposed to UV light its molecular chains cross-link, causing the *SU-8* to harden. The resulting polymer is highly transparent in the ultraviolet range. This allows for fabrication of films from 0.5 to 200 μ m thickness in a single coating. After exposing and developing, highly cross-linked structure of *SU-8* gives it high stability to chemicals and radiation damage -hence the name "resist". Cured cross-linked *SU-8* shows very low levels of outgassing in vacuum [100, 101]. However it is very difficult to remove, and tends to outgas in unexposed state [102]. Those characteristics make *SU-8* a wonderful material for waveguides manufacturing.

SU-8 is composed of Bisphenol A Novolac epoxy that is solved in an organic solvent (gamma-butyrolactone GBL or cyclopentanone, depending on the formulation) and up to 10 wt% of mixed Triarylsulfonium/hexafluoroantimonate salt as the photoacid generator [103]. With different composition, the viscosity would change and with different spinning speed, the thickness of *SU-8* could be changed as needed (Table A.2)

Table A.2: *SU-8 composition vs viscosity*

SU-8	%Solid	Viscosity (cSt)	Density (g/ml)
2000.5	14.3	2.49	1.070
2002	29.00	7.5	1.123
2005	45.00	45	1.164
2007	52.5	140	1.175
2010	58.00	380	1.187
2015	63.45	1250	1.200

Propylene glycol monomethyl ether (PGMEA)

Propylene glycol monomethyl (PGMEA) is commonly used as photoresist remover in semiconductor manufacturing, solvent for solvent-based coating systems, solvent in silk screen printing

inks. PGMEA is employed as developer for SU-8.

EpoCore/EpoClad

EpoCore/EpoClad is a chemically amplified negative tone photoresist series from Micro Resist Technology GmbH. Their main features are:

- Specifically designed for optical applications in micro system technology.
- Application of EpoCore as a core material in optical wave guiding in combination with EpoClad.
- High transparency to visible light.
- Excellent thermal stability of the resist patterns.
- Development in organic solvent.

In this work, we have used Epocore/Epoclad for the development of an integrated polarization splitter/switch.

A.4 Microstructured devices materials

In this section materials used in the fabrication of micro-structured devices like micro-lenses or optical vortex are explained.

OrmoStamp

OrmoStamp offers glass-like properties after UV curing and was designed for the fabrication of transparent polymer working stamps with excellent pattern fidelity.

Features:

- Transparent for near UV and visible light.
- Thermal and mechanical stability.
- Anti-adhesive properties for low release forces.
- Processing with standard lithography equipment.

OrmoStamp is an inorganic-organic hybrid polymer for the easy fabrication of a transparent working stamp used in NIL as a cost-effective to quartz or electroplated stamp. It can be applied in T-NIL or P-NIL.

Table A.3: *OrmoStamp properties [104]*

Parameter	OrmoStamp
Viscosity	0.4 Pa·s
Thermal stability	Up to 200°C
Refractive index (589nm, cured)	1.516
Replication with PDMS molds	Yes
Solvent free	Yes
Shelf life	8 months

OrmoComp

Ormocomp ® is a P-NIL inorganic-organic hybrid polymer for micro optical applications as microlenses or diffractive optical elements. The solvent-free ready-to-use formulation can be processed by UV molding or UV lithography.

Poly(methyl methacrylate) (PMMA)

Poly(methyl methacrylate) (PMMA), also known as acrylic or acrylic glass as well as by the trade names Plexiglas, Acrylite, Lucite, and Perspex, is a transparent thermoplastic often used in sheet form as a lightweight or shatter-resistant alternative to glass. The same material can be utilized as a casting resin, in inks and coatings, and many other uses. A 5% solution of PMMA on chlorophorm was used [105], because of the high volatility of the solvent (chloroform) used, the films produced here may not be of high quality.

Norland Optical Adhesive 81 (NOA 81)

Norland Optical Adhesive (NOA) 81 is a clear, solvent-free optical adhesive designed to fully cure in only minutes when exposed to ultraviolet light. It is recommended in precision alignment or positioning applications that require a robust and resilient bond. It can be cured in a thin layer between two transparent substrates or applied as a drop to form a bridge between two components. Exposure to UV light quickly cures the adhesive and holds the components in place.

The outstanding characteristic of this adhesive is its extremely fast cure. NOA 81 is sensitive to the entire range of long wavelength UV light from 320 to 380 *nm* with peak sensitivity around 365 nm. Cure time is dependent on light intensity and thickness of the applied drop.

NOA 81 cures from the surface down; longer cures are required for thicker films to allow UV light to penetrate to the full depth. After curing, it has very good adhesion to glass, metals, printed circuit boards and other glass filled plastics. This adhesion improves with age with optimum adhesion reached after room temperature aging for one week. This optimum adhesion can be accelerated by curing at 50°C for 12 hours. The cured adhesives can withstand temperatures from -150°C to 125°C.

For this material, the most important property for waveguides, refractive index, is $n=1.56$

after curing. Other typical properties are shown in Table A.4.

Table A.4: *Physical and optical properties of NOA 81*

Viscosity at 25 °C	300cps
Refractive index of cured polymer	1.56
Elongation at failure	25%
Modulus of elasticity(psi)	200000
Tensile strength(psi)	4000
Hardness -shore D	90

PEDOT:PSS

Poly(3,4-ethylenedioxythiophene) poly(styrene sulfonate) (PEDOT:PSS) is a polymer commonly used in the manufacturing of OLEDs, where it works as a hole injector. It is used as resistivity layer in this project because of higher resistivity than ITO.

It is used like resistive layer in MLCL fabrication.

A.5 Other materials used in device development

ITO coated glasses

All the glasses used in the experimental part are borosilicate glass Glaston glass with supertwist polished quality of 0.7 mm uniform thickness and coated with a 130 nm of Indium tin oxide (ITO) with a resistivity of $100 \Omega/\square$ and a smoothness of $0.1 \mu m/cm$. The glass contains about 10% boric oxide, has good optical and mechanical characteristics, and is resistant to chemical and environmental damage. The ITO coating acts as a contact in the inner side of the glasses that is typically found as an oxygen saturated composition with a formulation of 74% In, 18% O_2 , and 8% Sn by weight. ITO is one of the most widely used transparent conducting oxides because of its electrical conductivity and optical transparency. ITO can be precisely etched into fine patterns [106]. It can also stand temperatures over $200^\circ C$. In addition, ITO does not react with other materials used in the experiment. Their drawbacks are brittle nature and material processing requirements, that make them inadequate for a number of devices applications.

Photoresists

Microposit 1805 and *Microposit 1818* are commercial names of the photoresists mainly used in this project. Microposit photoresists are positive photoresist systems engineered for the microelectronics industry's requirements for IC device fabrication [107].

These photoresists have a very good adhesion to the employed substrates (silicon, glass) and come filtered at $0.2 \mu m$. In our experiments we have used them with a commercial developer (sold as developer for the fabrication of PCBs) from RS-amidata.

This photoresist can be dried and soft hardened in a hot plate or in an oven. Both methods have been used.

Polyimides

Polyimide (PI) is a polymer of imide monomers used to make alignment layers in liquid crystal cell manufacturing. The distribution of the orientation of liquid crystal molecules plays an essential role in the optical properties. So the role of the surface is to ensure a single domain. Without the special treatments of surface, the disinclinations or discontinuities will cause a severe scattering of light.

In our experimental samples, rubbed polyimide is applied to induce an orientation in the bulk of the cell. Besides, rubbing has been a simple and effective way to achieve a preferred orientation.

For planar alignment we have used PIA-2304 Lixon aligner polyimide that provides uniform alignment with a small pretilt in the rubbing direction.

Polyvinyl alcohol (PVA)

Polyvinyl alcohol (PVA) is an atactic material that exhibits crystallinity. It has excellent film forming, emulsifying and adhesive properties. It is also resistant to oil, grease and solvents. It has high tensile strength and flexibility, as well as high oxygen and aroma barrier properties.

PVA has a melting point of 230°C. It decomposes rapidly above 200°C as it can undergo pyrolysis at high temperatures. In the Thesis it is used as a alignment layer because it is very easy to spin and remove.

Silica spacers

In order to keep the cell glass plates and consequently the two opposing electrodes separated uniformly at a fixed distance spacers have to be employed. The spacers consist of silica or plastic micro balls or cylinders with a calibrated diameter [108].

The size of the spacers determines the thickness of the liquid crystal layer (d), and then the desired phase difference. The choice of the spacer is a function of the birefringence (Δn) of the liquid crystal material and the working wavelength (λ).

Depending on the desired thickness, the spacers used have certain sizes. The different sizes used in the laboratory are: 0.8, 0.9, 1.5 ... 20 μm . For higher thickness, polyester film Mylar with standard thickness: 23, 36, 50, 75 ... 350 μm are employed.

Silicon dioxide

Silicon dioxide, also known as silica is one of the most abundant families of materials, existing both as several minerals and being produced synthetically. Applications range from structural materials to microelectronics to components used in the food industry.

In our experiments silicon dioxide is used as cladding for waveguides. It has been deposited by sputtering. Obliquely evaporated silicon dioxide (by electron gun) has been also used as aligning layer for planar and homeotropic alignment.

Liquid crystals

Several liquid crystal materials have been used along the work realized in this Thesis. In the case of waveguided based devices liquid crystals have been chosen by their refractive indices. In lenses and other phase shift devices liquid crystal materials are selected taking into account the birefringence and the cell thickness.

The most used liquid crystals have been MLC-13000 and MDA-98 nematic mixtures from Merck.

MLC-13000

One of the LC used in the micro lenses is a commercial nematic MLC-13000 manufactured by Merck. This material shows an extraordinary and ordinary refractive indices $n_e = 1.6242$ and $n_o = 1.4946$.

MDA-98

The liquid crystal used in optical vortices is nematic LC MDA 98-1602 with ordinary refractive indices $n_o = 1.51$ and extraordinary refractive indices $n_e = 1.78$.

Appendix B

Light coupling system

Coupling light to PICs is a critical task, hence a system was developed to couple light and to characterize the chip. Since some of the waveguides dimensions may be smaller than $1\text{ }\mu\text{m}$ a high degree of precision is required. (See Figure B.1).

Figure B.1: *Stage setup. The sample stage has 3 axis linear micrometer stages and one rotational stage to locate the sample. A 100 $\times$ objective focuses the incoming laser beam onto the input of the chip. A Thorlabs NanoMax XYZ nanometer stage provides the fine positioning of the laser input. The sample holder is thermally controlled.*

The first part of the system is the coupling-in: light from the laser is guided using a fiber to a 5mm diameter collimator and a polarizer to a long distance 100x microscope objective which focuses the light onto the waveguide, as has been highlighted in Figure B.1. The collimator, polarizer and objective are all situated on a commercial 3-axis **nano-positioner** (Figure B.2).

The sample stage is composed by a 3 axes micro-positioner stage and 1 rotational stage (In plane angle). It has 20mm of travel in the 3 axis, and the possibility of rotation. Additionally, it has a thermal control with 0.05°C accuracy. The PIC is held in place by vacuum to the **temperature stage**.

A computer controlled camera is used to capture the output from the chip. The position of the camera is controlled manually with 3 linear and 2 angular stages.

Details of the different parts of the system are explained below.

B.1 Nano-positioner

The height of the waveguides is generally less than a micrometer, so a high precision stage is needed to couple light into the waveguides. The Thorlabs NanoMax was chosen because

Figure B.2: *The input stage* A Thorlabs NanoMax 300 provides 3 axis sub-micrometric manual control (main picture) plus a 3 axis nano-metric piezo controller (top right figure)

it has less than $0.1 \mu m$ accuracy. The NanoMax has $4 mm$ travel in 3 axis with a very high accuracy. With the manual movement the resolution is $0.1 \mu m$, but the stage provides nm precision with a electronic movement using piezo-electric materials that within a maximum travel of $20 \mu m$ has a resolution of $20 nm$.

B.2 Temperature control

Temperature control is very important in the experiments. There are at least 2 main reasons to control the temperature with high precision. First reason is the temperature changes the size of the devices due to thermal dilatation and contraction. The second reason is that the refractive index of the materials is related to temperature. So, a thermal control is attached to the PIC holder.

Figure B.3: *Temperature system. It allows control the temperature in the sample between $0^{\circ}C$ to $50^{\circ}C$.*

The thermal controller consists of 2 parts: the thermal circuit and the electronic controller.

- The thermal circuit has two parts: the Peltier heater/cooler (the Peltier cell) and the temperature interchanger. The Peltier heater/cooler is a ceramic device that has two faces and that allow direct conversion of a current driven by a voltage difference to a difference of temperature between the two faces. Applying a voltage, one face cools down and the opposite face heats up. If you change the polarity of the voltage the opposite happens: The face that before was heating up is now cooling down.

The down face of the Peltier cell is fixed to the temperature interchanger which is kept at room temperature and the opposite face is in direct contact with the sample holder.

A coolant is used to maintain the temperature interchanger and thus the bottom face of the Peltier cell at room temperature. A coolant pump is used to move the coolant through to a radiator to keep the coolant at room temperature.

A PT100 thermometer measures the temperature in the holder. A simple circuit (see Figure B.4) is used to read the temperature in the microcontroller ADC.

- The electronic controller provides the possibility to set the temperature in steps of 0.5°C with a theoretical accuracy of 0.05°C between 0°C to 50°C . Electronics are placed inside of a box with a display and buttons. It was necessary to include a fan for cooling down the temperature inside of the box due to the 8A used to provide power to the Peltier cell. The temperature of the holder is controlled with a PID loop using the PT100 readings, and the power is supply by a PWM (circuit available in Figure B.5). A H-bridge (circuit available in Figure B.6) is used to enable the voltage inversion making it possible to cool down or heat up.

B.2.1 Schematic of the electronic circuits

Figure B.4: *The 5V PT100 controller. OP1 is the LT1006*

Figure B.5: External power pulse width modulator, the NPN is BC546 and M1 is MJ2955

Figure B.6: H-bridge circuit. The NPN Q1 and Q6 are BC546, the NPN Q3 and Q4 are the TIP35C and the PNP Q2 and Q5 are the TIP36C

B.3 Acquisition setup

Following the optical path the last stage is the acquisition stage. It is composed by a 3 axis micro-positioner, and 2 rotational stages and a computer controller Nikon D500 camera. The light that exits the PICs is captured by the camera. The advantage in using a camera is that several outputs are measured simultaneously. However, the power is not absolute.

In Figure B.7 the acquisition setup is shown. The camera CMOS sensor has a 14 bits resolution in 20.9 million pixels behind a RGBG filter, each pixel corresponds to one color and the pixel pitch is $\sim 4.2\mu\text{m}$.

An ad-hoc software was made in order to access each pixel individually avoiding any image compression or camera processing. Prior to be

Figure B.7: Acquisition setup. A Nikon D500 camera is controlled by a computer to capture the optical information. The camera is attached to a micro-positioner with 2 angular and 3 linear degrees of freedom.

Figure B.8: Screenshot of the ad-hoc program. In the live image one can two optical outputs distinguished at the output edge of a PIC. The program plots the power of the ROIs realtime. In blue and red two ROIs are shown, in pink the reflection of the input light.

employed in the setup, the linear response of the CMOS was verified, as was the timing of the shutter speed. The software provides a live image that helps to couple the light as you by setting a number of Regions of Interests (ROI) and plotting the live power of each region. The software controls all the parameters of the camera including exposure time, aperture, and other parameters that are necessary to control the measurement.

Figure B.8 shows a screenshot of the ad-hoc program during a measurement. In the "Capture" tab it is possible to choose any shutter speed between $1/8000$ s and 30s. Another important tab is "Optimize XY" where the power of ROIs is shown and plotted in a graph, but also, a squares with a color are shown. The color of the squares change as the power increases (green color) or decreases (red color). The remaining tabs, allow for control of all camera parameters. The future idea is to substitute the manual controller by motorized controller and to make an automatized light coupling and characterization system. The motors are coming in the first quarter of 2018.

Appendix C

NIL machine

A system has been developed in order to generate a highly reproducible low cost NIL device for low volumes.

The underlying idea is to employ easily manufacturable parts made of low cost materials. The materials may be plastics or metals, such as aluminium. The parts are assembled into two devices: 1) **The Mold-maker** and 2) **The Printer**. A control box includes a PID temperature controller and manually controlled open/close and needle valves to control the pressure, which is monitored using a pressure sensor.

The devices combined are capable of replicating patterns in a target polymer from a master pattern in a two-step process. Firstly, PDMS, a silicone widely used for NIL, is used in the first step making a negative copy of the original master. Secondly, this negative PDMS mold is then transferred onto the target polymer, replicating the original master.

The devices may be made in polymer using a 3D printer. The use of plastics will typically avoids the use of temperature control but it still allows for pressure control. If temperature control is required, the devices have to be made in aluminium or similar metals. In the following, the description of the method will be detailed. Finally, an example of the imprinting will be shown as a proof of performance.

C.1 Device fabrication

The principle behind the method is making a sealed chamber with a flexible membrane as a lid. The inner surface of the membrane is engraved with the negative copy of the desired pattern. The membrane fabrication is fully documented below.

The pattern is transferred onto the uncured target polymer by controlling the pressure inside the chamber. Applying vacuum to the chamber the membrane is forced to bend slightly inwards squeezing the uncured polymer below. The use of vacuum has two advantages. The first, decreasing the pressure inside the chamber the air between the stamp and the target polymer will be eliminated. This is useful since air may inhibit the curing of some UV-curable polymers and the problem of trapped air is avoided. Secondly, it is possible to accurately control the force applied by the membrane onto the polymer. Two parameters may be tuned to control the process: the membrane stiffness and the pressure inside the chamber. The

membrane stiffness must be set during the membrane fabrication. The pressure is controlled by the driver during the imprinting process.

In the following the two devices are described in detail. Sketches of each device can be found below in section C.3 and C.4 on page 142 and 143.

1. The Mold-maker is used to obtain the negative PDMS mold from the original master. Three different parts form the device as shown in Figure C.1. The original master is situated face up in the Master support. Having a separate Master support means that it is relatively simple to change between master patterns, and to employ masters of different dimensions.

The assembled Mold-maker device makes a container into which the PDMS mixture is poured. The outer walls retains the liquid PDMS while it is being cured. The small pillars encircling the central part generate mounting holes in the final PDMS membrane. In Figure C.3 a typical PDMS mold is shown. The mounting holes will be used to fix the PDMS membrane to the Printer.

Figure C.1: 3D model of the Mold-maker. It consists of three principal parts: (A) The outer wall; (B) The Master support, and (C) The base. Besides from easy adaptation to varying master sizes, separate parts ease the demolding process once the PDMS is cured.

2. In the Printer the PDMS membrane with the negative pattern is attached as a lid creating a sealed chamber. Following a similar approach to the previous device, the device has a modular structure (Figure C.2).

In the base there is a vacuum connection used for fixing the substrate with the uncured

Figure C.2: *3D model of the Printer. This device is composed by two parts as well as two O-rings. (A) The ring constitutes the support of the mold and the walls of the structure, includes a hole that allows for the application of vacuum to the chamber. (B) The bottom part, which supports the substrate in which the copy is made. The bottom part contains holes for two heating cartridges, one thermocouple, and a vacuum outlet for the substrate fixing.*

polymer in which the pattern will be imprinted. The three additional holes in the base are for two cartridge heaters and a temperature sensor.

The upper part is used for locating and fixing the PDMS membrane accurately. In the wall there is a hole for applying vacuum to the chamber made by the base, the walls and the mold. O-rings are used in order to improve the seal.

An aluminium version of the devices is shown in Figure C.4 A and B. A 3D printed plastic version of the all the parts making up the Mold-maker and the Printer is shown in Figure C.4 C. An Ultimaker 2+ 3D printer has been used, with carefully chosen

tolerances (determined by trial and error) in order to ensure a proper fit between the different pieces. These tolerances depend on the 3D printer and printer settings.

Figure C.3: *Ready to use PDMS mold. The stamp area contains the negative copy of a grating for imprinting.*

3. The Controller. A schematic of the control box is shown in section C.2 on page 141. An Arduino UNO[109] is employed to monitor the pressure sensor *Honeywell SSC-DANN015PDAA5*. At start-up atmospheric pressure is measured and used as reference for measuring the vacuum. The display shows the difference between the inner and the reference pressure in *mbar*. The temperature control is done using a commercial temperature controller (Omron E5GN), two heaters and a PT100 temperature sensor. The code uploaded onto the microcontroller can be found in section C.5 on page 144.

Figure C.4: (A) *The Mold-maker* and (B) *The Printer* made of aluminium. The latter with a mold attached. (C) *The individual parts for two devices* made in 3D printed PLA. (D) *The Controller box*.

C.2 Schematic of the controller box

Figure C.5: *The Controller box with cover partially removed. The temperature controller is build in the lid. The vacuum system of the box is used to firmly fix the substrate to the printer and to apply vacuum to the printer chamber.*

Figure C.6: *Controller box for temperature and vacuum control of the Printer.*

C.3 Technical drawings of the Printer

Figure C.7: Technical drawing of the Printer base. Measures are in millimetres.

Figure C.8: Technical drawing of the Printer wall. Measures are in millimeters.

C.4 Technical drawings of the Mold maker

Figure C.9: *Technical drawing of the Mold-maker base. Measures are in millimeters.*

Figure C.10: *Technical drawing of the Mold-maker Master-support. Measures are in millimeters.*

Figure C.11: *Technical drawing of the Mold-maker walls. Measures are in millimeters.*

C.5 Driver code

```

1  /*****
2  Controller code for NIL device
3
4  Supplementary Material
5  *****/
6
7  #include <SPI.h>
8  #include <Wire.h>
9  #include <Adafruit_GFX.h>
10 #include <Adafruit_SSD1306.h>
11
12 #define OLED_RESET 4
13 Adafruit_SSD1306 display(OLED_RESET);
14
15 #define NUMFLAKES 10
16 #define XPOS 0
17 #define YPOS 1
18 #define DELTAY 2
19
20
21 #define LOG016_GLCD_HEIGHT 16
22 #define LOG016_GLCD_WIDTH 16
23
24 #define SSD1306_LCDHEIGHT 64
25 #if (SSD1306_LCDHEIGHT != 64)
26 #error("Height incorrect, please fix Adafruit_SSD1306.h!");
27 #endif
28 int analogPin = A0;
29 int val=0;//Value pressure

```

```

30  int val0=0;//Value on startup
31  double suma=0.0; //Calibration offset
32  int ref=1023;//Value ref
33  String texto;
34  double cuenta=0.0;//conversion digital to mbar
35  void setup() {
36      analogReference(EXTERNAL);
37      // Serial.begin(9600);
38
39      // by default, we'll generate the high voltage from the 3.3v line internally! (neat
40      // !)
41      display.begin(SSD1306_SWITCHCAPVCC, 0x3C); // initialize with the I2C addr 0x3D (
42      // for the 128x64)
43      // init done
44
45      // Show image buffer on the display hardware.
46      // Since the buffer is intialized with an Adafruit splashscreen
47      // internally, this will display the splashscreen.
48      display.display();
49      delay(50);
50      // Clear the buffer.
51      display.clearDisplay();
52      val0 = analogRead(analogPin);
53      testscrolltext();
54      delay(100);
55      display.clearDisplay();
56      for (int i=0; i <= 9; i++){
57          val0 = analogRead(analogPin);
58          suma=suma+val0;
59          delay(10);
60      }
61      suma=suma/10;
62      delay(200);
63      display.clearDisplay();
64
65  void loop() {
66      val = analogRead(analogPin); // read the input pin
67      display.setTextSize(2);
68      display.setTextColor(WHITE);
69      display.setCursor(0,0);
70      display.println("Pressure:");
71      display.setTextSize(3);
72      display.setCursor(0,40);
73      cuenta=(val-suma)*(1024/(ref-suma));
74      texto=String((int)cuenta)+"mbar";
75      display.println(texto);
76      display.display();
77      delay(500);
78      display.clearDisplay();
79  }
80
81  //Text scroll Loading
82
83  void testscrolltext(void) {
84      display.setTextSize(2);
85      display.setTextColor(WHITE);
86      display.setCursor(0,0);
87      display.clearDisplay();

```

```
88     display.println("Loading");
89     display.display();
90     delay(1);
91
92     display.startscrollright(0x00, 0x0F);
93     delay(2000);
94     display.stopscroll();
95     delay(500);
96     display.startscrollleft(0x00, 0x0F);
97     delay(2000);
98     display.stopscroll();
99     delay(500);
100    display.startscrollldiagright(0x00, 0x07);
101    delay(2000);
102    display.startscrollldiagleft(0x00, 0x07);
103    delay(2000);
104    display.stopscroll();
105 }
```

Appendix D

Vortex driver

A tunable optical retarder plate was constructed with an LC device with 24 pie-slice shaped electrodes. An in-house driver was developed to control the retardation induced in each pie slice (Figure D.1).

Figure D.1: *Picture of the driver.*

An Arduino [109] microcontroller was used to interface a computer with an *Adafruit 24-Channel 12-bit PWM LED Driver* which employs an SPI communication protocol. The

Adafruit Driver controls 24 separate 12-bit PWM channels. Each channel was used as an input in a analog switch, connecting the pie-slice electrode with an external waveform generator (typically a 6V AC signal @ 10 kHz), or not. Six ADG5412 SPST switches (every chip has 4 input/output) were used. A $\pm 20V$ power supply is needed to power the ADG5412 chips.

Figure D.2: Screen-shot of the software.

The working principle of the driver is forward straight. A signal is sent from the computer to the Arduino. The Arduino converts the signal to the SPI format and sends it to the LED Driver. Finally, the ADG5412 sends the AC power to the pie slice.

In order to control the Arduino an ad-hoc software was developed (Figure D.2). It controls the PWM duty cycle of each pie slice ($1/4096-1/1$), and thus the effective average voltage applied to the LC. Prior to the generation of vortices with different topological charges, the 24 relevant voltages must be determined. Once these levels have been determined (the left array in D.2), the software allows for automated generation of patterns with different *topological charge*, offset.

Figure D.3 shows the driver box. The Arduino UNO at the top of the figure, the Adafruit 24-Channel 12-bit PWM LED Driver in blue (bottom left) and the six ADG5412 SPST switches in green (bottom right).

The driver outlay can be seen in Figure D.4. The connections for one pie slice only (with

Figure D.3: *Picture of the disassembled driver.*

one ADG5412 only) is shown . All the outputs are pulled down with a $10\text{ k}\Omega$ resistance.

The summarized electronic circuit can be seen on Figure D.5 (For one pie slice only).

Figure D.4: *Driver outlay for one pie slice only.*

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