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# Shaping and characterization of an integrated optic on polymer by simple 3D printing

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## ABSTRACT

Integrated optics has been intensively developed the last decades for the production of components for telecommunications or sensors in metrology. The basic element constitutes the waveguide which transmits the information by guiding the light. The manufacturing processes take place in clean rooms with controlled atmosphere (pressure, temperature, dust) and with generally various deposition and coating machines. The production of these photonics devices remains relatively expensive since it is carried out in a clean room by series of manipulations which requires the intervention of a specialized engineer into a specific environment. More recently, techniques for manufacturing different mechanical parts of mostly research scientific devices have been developed using commercial 3D printers without the need of clean room environment. With such commercial machines, plastic materials are heated and then shaped by controlling their flow through a nozzle. This work concerns the study of the feasibility of realizing and shaping a kind of integrated optics by a simple 3D printer in a normal environment. It aims to understand and develop the fabrication of an integrated planar optics with polymer waveguides using a 3D printer and G-code programming. The coating processes and the G-codes of the 3D printer have been developed in order to produce the thermoplastic polyurethane (TPU) guiding structures onto a silicon/silica wafer. Finally, the characterization of this TPU organic (measurements of surface or energy tension, Raman analysis, ellipsometry measurements) and the optical injection made possible to validate this concept in terms of production and approach. The results concerning the shaping of various rib waveguides and obtaining a propagation of the light in organic thermoplastic polyurethane planar guides with fiber tapers by this simple and low-cost printing processes are positive. This work further proves that the development of integrated optics on thermoplastic polyurethane by 3D printing is possible; this opens the way to the realization of other optical patterns based on the 3D printer principle.

**Keywords:** 3D printer, G-code, polymer, Thermoplastic Polyurethane (TPU), integrated optics, waveguides, optical characterizations

## 1. INTRODUCTION

All of the integrated photonic<sup>1,2</sup> components for optical telecommunications<sup>3</sup>, sensors and metrology<sup>4-10</sup>, the medical and optical industries, artificial intelligence are generally produced in controlled environments such as deposition reactors and clean rooms for successive manufacturing processes. Thus, no matter which substrates and materials are used in microtechnology, their masking and photolithography, shaping and deposition processes take place in a controlled atmosphere and environment (humidity level, temperature, pressure, particles and dust). For example, organic materials in the form of resins<sup>9</sup> spread out by spin coating (various phases of time, speed, acceleration) are flashed (UV) and developed in clean room. Among their working principle (modulations, filtering, multiplexing, ...) and types of photonic components,

various structures have become very classic, such as Mach-Zehnder interferometers<sup>3,4</sup>, micro-resonators<sup>5-10</sup>. However the original basic element is unquestionably the waveguide<sup>1,2,11</sup> (rectangular and rib, planar, buried, fiber...). The latter makes it possible to guide the light and to propagate the optical wave.

At the same time in recent years, 3D printer technology has emerged; such 3D printers allow the production of macroscopic 3D parts or pieces (millimeter or fraction of millimeter) in different materials including plastics. Some of them can work in an uncontrolled environment and even in the open air with a single cover. This new approach is interesting and would essentially reduce production costs and also operating costs compared to clean rooms.

The purpose of this work is to estimate the potential to manufacture the basic elements of waveguide types (ribs, multi-modes) in polymers (thermoplastic polyurethane, TPU) by the simple use of a 3D printer. Section 2 presents the pre-production of Si/SiO<sub>2</sub> wafers added to the 3D RAISE printer technology used, its mechanics (nozzle plus organic TPU material) as well as the G-code programming to control the design and the targeted coating of the thermoplastic polyurethane (TPU) rib. Section 3 is dedicated to the physico-chemical characterization of the TPU polymer by Raman analyses,  $\gamma$  surface tension or energy measurements and ellipsometric measurements (optical indices, real and imaginary parts). Section 4 presents and concludes on the first results of optical guidance and propagation in TPU rib waveguides produced in the open air at derisory manufacturing costs.

## 2. MIX OF TECHNOLOGIES FOR THE REALIZATION OF TPU WAVEGUIDES BY SIMPLE 3D PRINTER, G-CODE PROGRAMMING

### 2.1 Getting started with the 3D printer

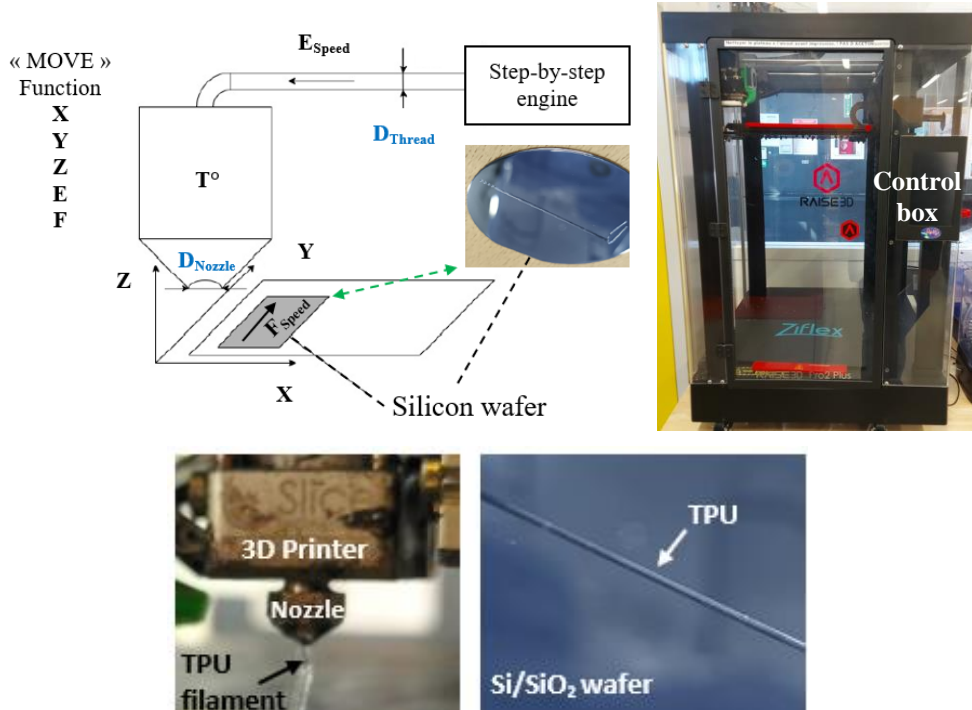


Figure 1. Diagram and photography of the RAISE 3D ‘Pro2Plus’ printer with G-coded parameters (XYZEF); Photography and zoom of the nozzle ; TPU (Thermoplastic Polyurethane) polymer coating by heating and material flow control, deposition onto Si/SiO<sub>2</sub> wafer.

Figure 1 shows the complete RAISE 3D printer. In terms of the environment, polymers are purchased in coils of rolled-up wires. The cost of these materials is extremely low. The thread in organic material (TPU for us) is attached to the entrance of a motorized head which unrolls the wire and heats it up. The device is finished by a metal nozzle with a  $D_{nozzle}$  diameter of 400  $\mu\text{m}$  (see photograph, bottom left). In such simplest operation, the Thermoplastic Polyurethane (TPU) polymer will be then ejected and shaped like a wire or fiber with a section close to 400  $\mu\text{m}$ . A digital control box (on the right of the

printer machine) enables to control and display various parameters such as the heating temperature. The temperature of the 3D printer nozzle is set at 230°C, the optimum temperature for obtaining homogeneous quality filaments. On the motorized plate (movement in z axis, figure 1) it can be placed a support, a wafer, or simply position the start of printing (X, Y, Z coordinates).

## 2.2 G-code programming, polymer shaping control

In order to fully control the 3D printer more precisely with a set of parameters such as the length of the filament to be extruded (E), the temperature (T), the 3D positioning of the nozzle (X,Y,Z), the speeds of movements of the nozzle (F), it is necessary to launch a G-code program on the printer (USB socket on the control box). There are several ways to prepare a G-code for a printer. The first is to use a slicer like 'Slic3r', 'Skeinforge' or 'Cura'. These programs take a Computer Aided Design (CAD) model and cut it into elementary layers that make it up. Slicers are the best way to go from a 3D model to a printable G-code file, but the user sacrifices some flexibility when using them. Another option for generating the G-code is to use a lower level library like 'Mecode'. Libraries like 'Mecode' give you precise control over the tool path, and so are useful if you have a complex print that is not suitable for native slicing. The last option is just to write the G-code yourself. This may be the best choice if one just needs to run a few test lines during the calibration of the printer.

```

T0
X;Choisir la buse 0
G21
X;D finir l'unit en millim tres
G90
X;Coordonn es d fini en position absolue
M82
X;Extrudeur en mode absolue
G28
X;Retour l'origine

X;G1=operation bouger F=vitesse mm/min XYZ=position absolue E=extrusion de fil durant
op ration programm e

G1 F1200 Z005
X;aller a la coordonn e Z005 a la vitesse de 1200mm/min
G1 F1200 X213 Y010 Z005
X;aller a la coordonn e X213 Y010 Z005 a la vitesse de 1200mm/min

M221 T0 S100
X;D finir le d bit de la buse 0 100%

M104 T0 S230
X;D finir la temp rature de la buse 230
M109 T0 S230
X;D finir la temp rature de la buse 230 et attendre
M140 S70
X;D finir la temp rature du plateau 70
M190 S70
X;D finir la temp rature du plateau 70 et attendre

G1 F120 Z000.6
X;aller a la coordonn e Z000.6 a la vitesse de 120mm/min
G1 F120 X213 Y060 Z000.6 E009.5
X;aller a la coordonn e X213 Y060 Z000.6 a la vitesse de 120mm/min et extrusion de
9.5mm de fil
G1 F2400 X213 Y060 Z025
X;aller a la coordonn e X213 Y060 Z025 a la vitesse de 2400mm/min
G1 F2400 X50 Y50 Z025
X;aller a la coordonn e X50 Y50 Z025 a la vitesse de 2400mm/min

G28 X0 Y0 Z0
X;retour l'origine

```

Figure 2. G-code for the realization of multimode rib waveguides in TPU polymer by 3D printer.

A G-code RepRap is a list of fields that are separated by white spaces or line breaks. A field can be interpreted as a command, parameter, or for any other special purpose. It consists of a letter directly followed by a number or can be just

a standalone letter (Flag). The letter gives information about the meaning of the field. Numbers can be integers or fractional numbers depending on the context. Figure 2 represents the G-code written to trace by TPU extrusion rib waveguides of widths of 400  $\mu\text{m}$  onto silicon/silica wafer; see also Figure 1 (photograph bottom right) of the TPU guide deposited on wafer.

The goal was to produce and deposit a waveguide of the same dimension as the  $D_{nozzle}$  diameter of the nozzle, therefore the extrusion speed  $E$  of the TPU wire was tuned to the displacement speed  $F$  of the nozzle taking into account the  $D_{thread}$  diameter of the TPU thread at the origin, see Figure 1.

$$E = \left(\frac{D_{nozzle}}{D_{thread}}\right)^2 \cdot F, \text{ with } F \equiv \sqrt{X^2+Y^2+Z^2} \quad (1)$$

The previous G-code with the relevant values determined by eq. (1) made it possible to shape the rib waveguides by TPU extrusion and 3D printing (see Figure 1, photography bottom right).

### 3. CHARACTERIZATIONS AND PHYSICO-CHEMICAL MEASUREMENTS OF THE TPU CORE MATERIAL

Thermoplastic polyurethane (TPU) includes alternating rigid and soft blocks. Flexible segments consist of reaction products between polyols (HO-R-OH) and diisocyanates (OCN-R-NCO), while rigid segments are the reaction of diisocyanates with small molecular chain extenders (HO-R-OH). The flexible segments provide elastomeric properties while rigid segments lead to polyurethanes groups (R-O-(C=O)-(N-H)-R) with various physicochemical properties. TPU is an elastomeric thermoplastic<sup>12</sup> which, when heated (230°C) by the printer nozzle will flow under gravity and then coat to create waveguides. Therefore it is very interesting to investigate its shape properties and to measure its  $\gamma$  surface tension<sup>13,14</sup>. First, the hanging drop method was used to determine the surface tension of the heated TPU forming a drop shape. By imaging the profile of the drop, it is actually possible to estimate this surface tension<sup>13,14</sup>.

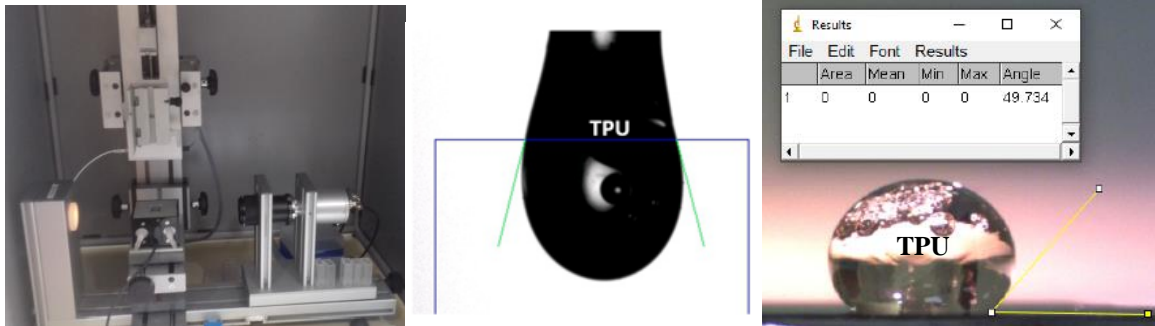


Figure 3. Machine for measuring  $\gamma$  surface tension by imaging : 'hanging drop method'. Measurements of  $\gamma_{TPU}$  superficial tension by pendant drop ( $\gamma_{TPU} \sim 30\text{-}34 \text{ mN.m}^{-1}$ ). Characterization and triple point method, behavior of molten TPU onto the Si/SiO<sub>2</sub> wafer : ( $\gamma_{SiO_2/TPU} = 49.4 - 30 \cdot \cos(180 - 49.7) = 68.8 \text{ mN.m}^{-1}$ ).

Figure 3 represents the machine for photographing the drop of TPU. For our drop profile we obtained a value of the surface tension of about  $\gamma_{TPU} \sim 30\text{-}34 \text{ mN.m}^{-1}$ . In a second step, we were interested to determine the  $\gamma$  surface energy of the TPU under deposition condition onto the Si/SiO<sub>2</sub> wafer serving as a support for a planar integrated optics; the silica has an optical lower cladding role with a lower optical index than the TPU guiding core. This was done by the measurement of the  $\theta$  angle at the triple point (Vapor Liquid Solid) by heating the TPU (that become liquid L) directly onto the solid (S) or wafer covered with SiO<sub>2</sub>, see Figure 3. Contact angle measurements were easily performed by establishing the angle at the base of a liquid drop lying on a SiO<sub>2</sub> flat solid surface<sup>15,16</sup>. Using the given value of the  $\theta$  angle (49.4°) taken by the molten TPU droplet, and using the equivalence of Newton's projected  $\gamma_{SL} = \gamma_S - \gamma_L \cdot \cos \theta$  second law, we get  $\gamma_{SiO_2/TPU} = 49.4 - 30 \cdot \cos(180 - 49.7) = 68.8 \text{ mN.m}^{-1}$ . This surface tension value provides information on the mechanical behavior of the soft molten TPU material which will interact on the Si/SiO<sub>2</sub> wafer.

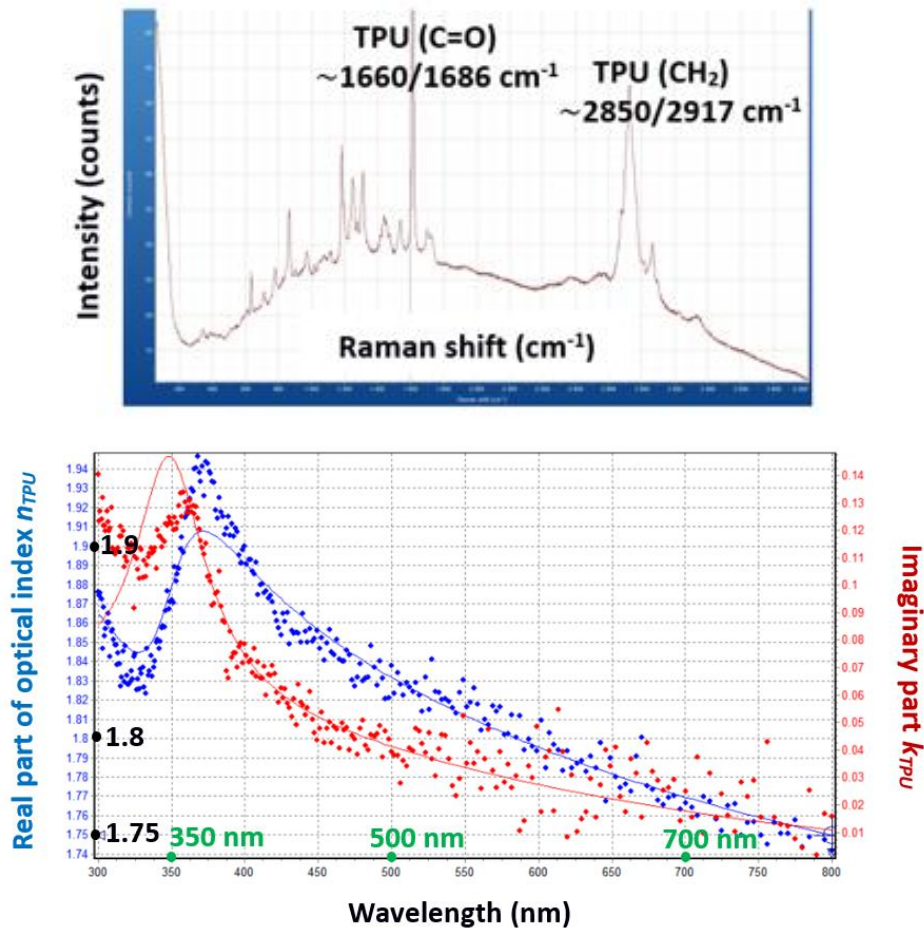


Figure 4. Raman analysis and ellipsometric measurements, optical index of TPU polymer and dispersion curve ( $n$ -real then  $k$ -imaginary part).

The high quality control of the material (properties, geometries, surface aspects and so on) was performed by imaging and analysis methods: optical microscopy, Raman micro-spectroscopy and ellipsometry (Figure 4). The LabRAM HR800 Raman Spectrometer (from Horiba Scientific company, Jobin-Yvon) is a high resolution spectrometer coupled with a confocal microscope, several laser sources (633 nm He-Ne), (785 nm Toptica) and (532 nm Coherent) and nano-positioners. The coupling with a confocal microscope enables to image a sample in 2D with a spatial resolution of the order of the spot size (0.9  $\mu\text{m}$ ) in the focal plane and a spectral resolution of typically  $1 \text{ cm}^{-1}$  per pixel. The laser excitation power was lower than 0.1 mW, which excludes heating effects onto the TPU photonics waveguides. The Raman analysis confirms, with two particular peaks in terms of chemical bonds, the signature of the TPU. Finally, very important for integrated photonics applications are the measurements of the real (propagation) and imaginary (absorption) parts of the optical indices. These were carried out by a Horiba Uvisel 2 ellipsometer. Figure 4 shows the dispersions curves (experimental and simulation) of the optical indices of the TPU proving that such material, with an optical index greater than silica (1.45 at 700 nm), can serve as optical guide or core for the envisaged structures: optical indices of TPU material close to 1.75, 1.8 and 1.9 at wavelengths of 700 nm, 500 nm and 350 nm respectively were measured. The associated simulation is obtained with a Tauc-Lorentz model and a simple Lorentzian.

#### 4. INTEGRATED OPTICS AND PROPAGATION, CONCLUSION

The deposition of the TPU by 3D printer follows the prefabrication of the support based on a silicon wafer with a thermal silica layer obtained by the wet oxidation method (Table 1).

Table 1. Pre-plasma procedure and thin layer processes for the fabrication of thermal SiO<sub>2</sub>.

Thermal Silica onto Silicon	
Step (SiO <sub>2</sub> , AET Technologies)	Parameters
Thermal oxidation	4h min at 1175°C, successively
Humid pyrolytic oxidation	O <sub>2</sub> (2 l/min) + N <sub>2</sub> (2 l/min) 5 min
	O <sub>2</sub> (2 l/min) 10 min
	H <sub>2</sub> (1.8 l/min) + O <sub>2</sub> (1 l/min) 4 h
	N <sub>2</sub> (2 l/min) 20 min

The wafer needs to be cleaned with the RCA method (developed by 'Radio Company of America'), to avoid all kinds of impurities. Then the plate is put in a quartz furnace with a torch. H<sub>2</sub> and O<sub>2</sub> gases are added to the flame in order to ensure the combustion and the oxidation. A step lasting typically 4 hours in an oven at 1175°C can form a typically 1 μm layer of silica on the wafer surface, creating then the silica lower cladding. After oxidation, a mandatory annealing at 700°C-800°C strengthens the single crystal structure. Such a thickness value secures a highly stable and homogeneous refractive index for the proposed organic TPU waveguides, so as to reduce any losses of optical radiation from the propagation modes.

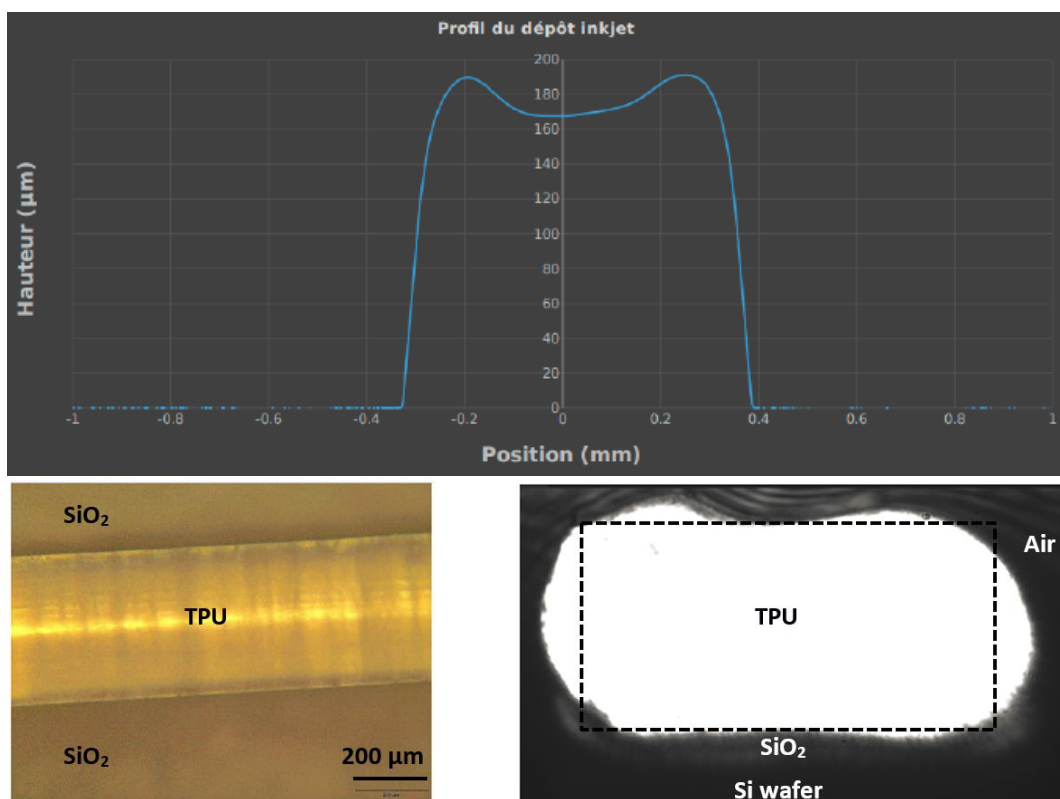


Figure 5. Profilometric measurements of the 'rib' guide. Microscopy and optical imaging of the thermoplastic polyurethane (TPU) planar ribbon guide (upper view) ; injection and cross-sectional view of the propagation light into the waveguide (wavelength  $\lambda = 800\text{nm}$ ). Air and silica act as upper and lower optical cladding respectively.

Following the production of the TPU rib waveguide by direct coating on the Si/SiO<sub>2</sub> wafer directly in an ambient environment, that is non-clean room (Figure 1, bottom right) with the help of the G-code (Figure 2), the cleavage is obtained in a conventional manner for optical injection (superluminescent diode at  $\lambda=800$  nm). Cross-sectional measurements of the tape guide profile show a slight bulge of the TPU material during coating through the 400  $\mu\text{m}$  orifice nozzle (Figure 5, top). Deposition of the TPU guides by 3D printer reveals good mechanical strength after cooling of the Si/SiO<sub>2</sub>/TPU structure. Figure 5 shows also a top view of the bright tape guide (bottom, left) with the optical intensity detected by a single camera at the outlet guide (bottom, right).

The optical properties (index, transparency) of this TPU<sup>17</sup> by such a simple manufacturing method enabled to obtain excellent light propagation over several centimeters. These results by using a simple 3D printer can pave the way for the production of certain low-cost optical components and connections without the use of a clean room.

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## REFERENCES

- [1] Adams, M.J., [Introduction to Optical Waveguides], John Wiley & Sons, New-York, (1981).
- [2] Snyder, A.W. and Love, J.D., [Optical Waveguide Theory], 2<sup>d</sup> Ed. Kluwer Academic Publishers, (2000).
- [3] Donval, A., Toussaere, E., Hierle, R. and Zyss, J., “Polarization insensitive electrooptic polymer modulator”, *J. Appl. Phys.* 87, 3258–3262, (2000).
- [4] Pelletier, N., Bêche, B., Tahani, N., Zyss, J., Camberlein, L. and Gaviot, E., “SU-8 waveguiding interferometric micro-sensor for gage pressure measurement”, *Sensors and Actuators A* 135, 179–184, (2007).
- [5] Chauvin, D., Bell, J., Leray, I., Ledoux-Rak, I. and Nguyen, C.T., “Label-free optifluidic sensor based on polymeric microresonator for the detection of cadmium ions in tap water”, *Sensors Actuators B* 280, 77-85 (2019).
- [6] Castro Beltran, R., Huby, N., Vié, V., Lhermite, H., Camberlein, L., Gaviot, E. and Bêche, B., “A laterally coupled UV210 polymer racetrack micro-resonator for thermal tunability and glucose sensing capability”, *Adv. Dev. Mat.* 1, 80-87, (2015).
- [7] Li, Q., Vié, V., Lhermite, H., Gaviot, E., Moréac, A., Morineau, D., Bourliou, C., Dupont, D., Beaufils, S. and Bêche, B., “Polymer resonators sensors for detection of sphingolipid gel/fluidphase transition and melting temperature measurement”, *Sensors Actuators Phys. A* 263, 707-717 (2017).
- [8] Castro Beltran, R., Garnier, L., St Jalmes, A., Lhermite, H., Gicquel, E., Cormerais, H., Fameau, A.L. and Bêche, B., “Microphotonics for monitoring the supramolecular thermoresponsive behavior of fatty acid surfactant solutions”, *Opt. Comm.* 468, 125773.1-7 (2020).
- [9] Garnier, L., Lhermite, H., Vié, V., Pin, O., Liddel, Q., Cormerais, H., Gaviot, E. and Bêche, B., “Monitoring the evaporation of a sessile water droplet by means of integrated resonator”, *IoP J. Phys. D: Appl. Phys.* 53, 125107.1-125107.10 (2020).
- [10] Garnier, L., Gastebois, J., Lhermite, H., Vié, V., St Jalmes, A., Cormerais, H., Gaviot, E. and Bêche, B., “On the detection of nanoparticle cloud migration by a resonant photonic surface signal towards sedimentation velocity measurements”, *Results In Optics*, 12, 100430.1-13, (2023).
- [11] Bêche, B., Jimenez, A., Courbin, L., Camberlein, L., Artzner, F. and Gaviot, E., “Functional silica nano-connections based on fluidic approach for integrated photonics”, *Electron. Lett.* 46, 356–358, (2010).
- [12] Brochard-Wyart, F. and Guéré, D., [Gouttes, bulles, perles et ondes], Ed. Belin, (2005).
- [13] Lucas, L-D., “Tension superficielle”, *Techniques de l’Ingénieur*, M67, 1-7 (1984).
- [14] Eustathopoulos, N., Ricci, E. and Drevet, B., “Tension superficielle”, *Techniques de l’Ingénieur*, M67, 1-13 (1999).
- [15] Yonger, M., ‘Dynamique du mouillage pseudo-partiel de la silice par des fondus de polymère’, Thèse de Doctorat en Physico-Chimie des Polymères, École doctorale Physique et chimie des matériaux - Paris, (2016).
- [16] Zebda, A., Sabbah, H., Ababou-Girard, S., Solal, F. and Godet, C., ‘Surface energy and hybridization studies of amorphous carbon surface’, 254, 4980-4991 (2008).
- [17] <https://www.atome3d.com/products/esun-etpu-95a-clear-1-75-mm-1-kg?variant=34483461423255>