FDTS as Dewetting Coating for an Electrowetting Controlled Silicon Photonic Switch

Sarah Günther, Shuhao Si, Herbert D'heer, Dries Van Thourhout, and Martin Hoffmann

***Abstract*— The self-assembled monolayer FDTS (1H,1H,2H,2H-Perfluorodecyltrichlorosilane) is presented as suitable dewetting coating material for an electrowetting on dielectrics (EWOD) controlled silicon photonics switch deployed in fiber optic telecommunication systems. The anti-sticking characteristics of Perfluorodecyltrichlorosilane (FDTS) are compared to those of polytetrafluoroethylene (PTFE) as most common dewetting coating for EWOD devices. It is shown that FDTS could outperform other materials such as PTFE when an extremely thin, long-term stable, and uniform layer is required. In the specific case, FDTS is applied in vapor phase as anti-sticking coating to the active optical surface of the integrated silicon photonics switch thus enabling the EWOD driven liquid motion. The suitability of the coating is presented by contact angle measurements and durability tests carried out with the switching liquids. Finally, it is demonstrated by optical measurements that the FDTS coating has a neglectable influence on the optical switching performance.**

***Index Terms*— Optical switches, optofluidics, microswitches, microfluidics, electrowetting, self-assembled monolayer (SAM).**

I. INTRODUCTION

THE material Perfluorodecyltrichlorosilane (FDTS) has been widely applied for anti-sticking coating in Nanoimprint Lithography (NIL) [1]–[5]. The FDTS coating is acknowledged by its processing simplicity that requires uncomplicated facilities at wide-range temperature conditions. The monolayer coating with a thickness of a single molecule has a long-tail consisting of 17 fluorine atoms providing convenient anti-sticking properties. Therefore, these significant properties of FDTS make it advantageous in additional fields as well, such as electrowetting on dielectrics (EWOD) [6] or microfluidics in general [1], [7], [8]. In this letter, the application of the self-assembly silane as dewetting coating in an EWOD controlled silicon photonics switch is demonstrated.

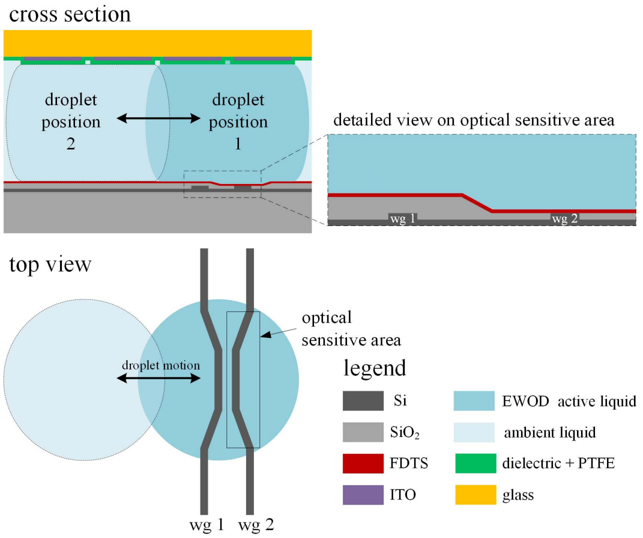

Fig. 1. Schematic diagram illustrating the working principle of the optical switch.

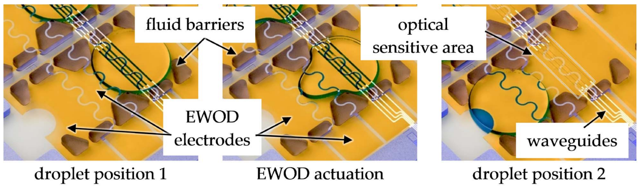

Fig. 2. Rendered 3D images of droplet motion during EWOD actuation.

A schematic illustration of the working principle of the EWOD driven silicon photonics switch is given in Fig. 1. The switch consists of an adiabatic coupler where the oxide cladding above one of the waveguides is removed. Switching is realized by exposing this waveguide to liquids with contrastive refractive indices. The respective liquid serves as a cladding material and hence by exchanging the liquids the effective index of the exposed waveguide changes. The liquid motion over the coupler is driven by EWOD. All details on the EWOD system design, material selection and the liquid switching process are reported in [9]. To enable the switching, two fluids with a specific contrast in refractive index are exchanged over the coupler. By moving the fluids, the coupler switches between two states, cross state and bar state. The active EWOD component that responds to the electric field and is moved is a polar fluid. The ambient liquid whereas is non-polar and does not respond to electrowetting but is displaced due to the motion of the polar liquid. In Fig. 2, a sequence

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of images illustrates the switching process. In Fig. 2 (left), the droplet is parked in position 1, a position which covers the optical sensitive switching area. Fig. 2 (middle) shows the droplet transition from position 1 to position 2 by sequential activation of the EWOD electrodes. The optical switching area is covered by the ambient liquid and the switching process is completed as shown in Fig. 2 (right). Due to the fluidic barriers including a constriction between the two droplet positions, each position is physically stable which leads to a non-volatile optical switch. For the first demonstrator, the droplet diameter is 900 μm, the length and width of a single EWOD cell is 2300 μm × 1310 μm. Details on the optical switching concept and its characteristics are reported in [10]–[13].

II. MATERIAL AND METHODS

A. Dewetting Coating

Depending on the surface of the optical switch and the used liquid combination, a certain Young contact angle θ_Y forms at the interface between the liquid and the surface of the coupler. The contact angle can be used as an indicator for the wetting behavior of the liquid. A contact angle below 90° corresponds to a high wettability [14], i.e. the liquid wets the surface and movement is impeded. In order to enable droplet movement, it is necessary to achieve a low wettability (contact angle higher than 90°) for the active polar liquid on top of the coupler which can only be achieved by applying a dewetting coating on the optical surface. The coating material has to fulfill the anti-sticking requirement for the EWOD system as well as its optical specifications. The intensity of the optical mode of the light propagating through a waveguide decreases exponentially and for high index-contrast waveguides light is largely confined in the waveguide core. Thus, the thickness of the dewetting layer is mandatorily minimized as it greatly impacts the efficiency in the optical sensitive area. The coating should also possess a long-term stability and temperature sustainability to endure the droplet manipulation at ambient conditions.

The most commonly acknowledged dewetting layer used in electrowetting devices is Polytetrafluoroethylene (PTFE, commonly referred to the trade name Teflon AF). That material has excellent dewetting properties and can be deposited through several approaches, e.g. dip-coating, spin-coating, chemical vapor deposition or even sputtering [15]–[20]. However, certain characteristics of PTFE make it unsuitable for the application of the optical switch: The minimum coating thickness of a closed PTFE film is relatively large. In general, layers from 100 nm to 1000 nm nm thickness are used for microfluidic applications. We reduced the minimum stable film thickness of a PTFE dip coating process to 12 nm. At this condition, the PTFE coating still affects the optical characteristics of the optical switch extensively, which will be shown later. Further thinning by plasma etching led to a massive loss of the dewetting characteristics. Additionally, PTFE forms only physical bonds to the surface where it is applied to, rather than covalent bonding. The PTFE polymer is very stable and has a low surface energy resulting in a bad adhesion to the surface that it is applied to. Furthermore, the most common deposition methods are dip- or spin coating delivering satisfying results for planar substrates but insufficient uniformity and edge-coverage for surfaces with 3D-structures like fluid channels or etched grooves. Thus, the topology and shape of substrates that can be effectively coated are limited. By spin- or dip coating, uniform wetting of high aspect ratio structures such like micro-fluidic channels as well as sub-micro structures of high resolution and density is barely possible when material is coated in liquid phase [2]. Alternative deposition technologies, which deliver highly uniform coatings on structured samples, like chemical vapor deposition or sputtering are feasible yet require dedicated equipment.

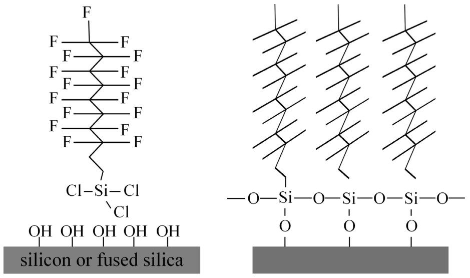


Fig. 3. Illustration of FDTS molecule and schematic sketch of the self-assembly monolayer formation process.

FDTS has shown several advantages over Teflon as anti-sticking coating material for the optical coupling surface. It can be deposited in vapor phase which leads to an optimal uniformity and an excellent edge coverage. The silane, i.e. the reactive head, bonds covalently to silicon and silicon dioxide which are key materials in silicon photonic devices. This leads to a high chemical and mechanical stability. The long tail of the molecular including 17 fluorine atoms ensures the dewetting properties of the surface. The FDTS deposition results in a self-assembled monolayer (SAM) with a thickness of the single molecule having a length of ∼1.4 nm [1]. Fig. 3 gives a schematic illustration of the FDTS molecule and the SAM formation process. Experimentally, the FDTS is deposited in vapor phase at 165 °C for at least 2 hours. Another major advantage of the vapor phase deposition is the feasibility to apply the coating after the switching device is assembled. The switch consists of an optical and an EWOD substrate, whereby the EWOD substrate contains openings for liquid filling with a diameter of approximately 350 μm. The mounting of both substrates is carried out using UV-curing glue. However, anti-sticking coatings prevent the bonding of adhesive materials such as glue or resin to the substrate hence hampering the assembly. This issue can be circumvented by coating after mounting. In case of the optical switch, vapor deposition of an assembled sample through the liquid filling openings is possible and was carried out successfully.

B. Switching Liquids

A suitable liquid combination fulfilling the complex requirements of both the optical and the EWOD domain of the optical coupler has been presented in [9]. From the optical point of view, the liquids preferably have a high contrast in refractive indices of about 0.16 or larger and low absorption

TABLE I

LIQUID SELECTION FOR THE FIBER OPTIC SWITCH

nonpolar ambient liquid	diphenyl sulfide (DPS) triphenyl sulfide (TPS)
polar electrowetting active liquid	ethylene glycol (EG) hydroxy propylene carbonate (HPC)

TABLE II

MEASURED CONTACT ANGLES USING THE YOUNG-LAPLACE METHOD

Liquid		DPS	TPS	EG	HPC	H20
CA in °	Air\FDTS	85	as DPS	97	99	116
	Air\PTFE	84	as DPS	99	103	116

in the telecommunication wavelength range from 1260 nm to 1650 nm. For the EWOD actuation, it is necessary to have a high polarity and high permittivity for the active liquid, while the ambient liquid should be nonpolar and have a low permittivity. The dynamic viscosities and the difference in density of the liquids are critical for the EWOD dynamics. The liquids have to retain their properties within the temperature range for ambient conditions as well. These optical, electrical and environmental demands require a novel selection of liquids listed in Table I that have never been employed in optical or EWOD systems before. For the dewetting coating, the pair of the liquids shall meet the requirement that the contact angle for the active polar liquid should be higher than 90° whereas the contact angle should be below 90° for the ambient one. Mechanical and chemical long-term stability of the coating under exposure of the liquids should also be guaranteed.

III. RESULTS AND DISCUSSION

The FDTS monolayer reduces surface energy due to its long fluorinated tail. The Youngs equation correlates the surface energy and the contact angle θ_Y of liquid-gas interface:

$$\gamma_{SG} = \gamma_{SL} + \gamma_{LG} \cos \theta_Y \tag{1}$$

where S, L, and G refer to the solid, liquid and gas phases, respectively, and γ refers to the interfacial tensions of corresponding interfaces. Youngs equation indicates that the reduction of surface energy can be determined by a contact angle measurement. Contact angle measurements of each liquid on top of a silicon surface in air were performed using a Krüss DSA10 drop shape analysis system following the Young-Laplace equation [12]. The contact angles of each liquid on a PTFE coated substrate were also measured as a reference. The results of the contact angle measurements can be found in Table II. The results show that the FDTS coating exhibits an equivalent dewetting performance as PTFE.

EWOD actuation on FDTS coated switches was carried out successfully whereby a switching speed ranging from 3–5 s was achieved at a voltage of 35 V. Actuation on PTFE coated samples led to a comparable switching speed whilst droplet movement without coating was impeded.

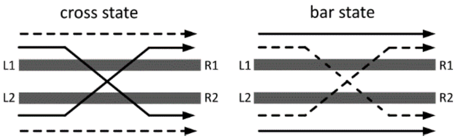
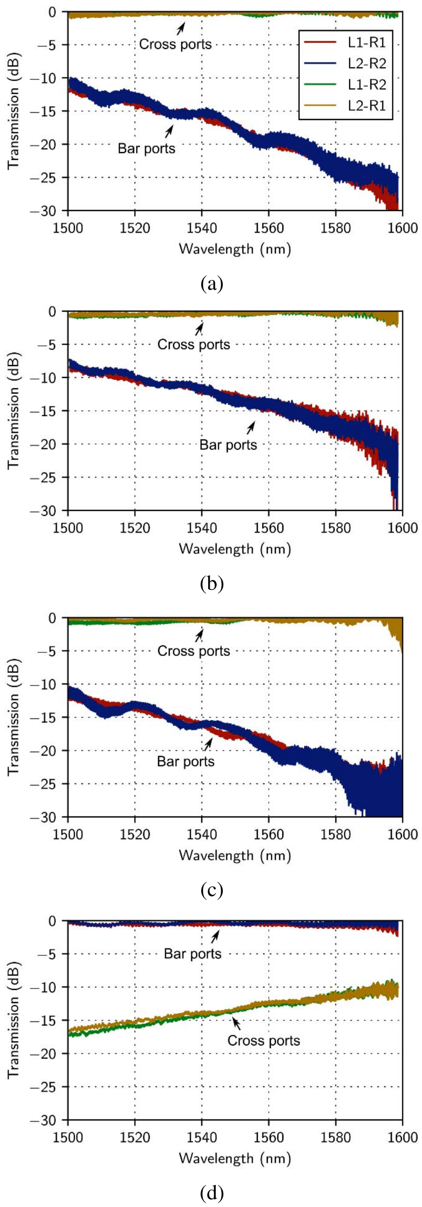


Fig. 4. Possible light paths in a 2 × 2 optical switch in cross and bar state. Transmission is represented by solid lines and cross talk by dashed lines.

The stability and insolubility of FDTS coating in contact with the liquids have been approved by experiment over a period of 60 days. An optical switch has been assembled and filled with the liquids HPC and TPS. EWOD actuation was tested initially and after 30 and 60 days, respectively. Droplet motion could be observed over the full test period, indicating that the FDTS layer on the silicon dioxide surface of the optical chip is stable and has not degraded. Therefore, the FDTS is approved for its stability and insolubility in the selected liquids for the application of the active optical switch.

Finally, the optical performance of an optical switch device with FDTS coating has been evaluated. Optical switches with a length of 1.4 mm are fabricated on silicon-on-insulator (SOI) substrates as described in [10] on which then a dewetting coating is applied. The switches are characterized with the liquids HPC and TPS by measuring the transmission of light propagating through the switch on substrates that have 1) no coating, 2) a PTFE coating with a thickness of approximately 12 nm and 3) a monolayer coating of FDTS. In each configuration, the cross and bar state transmission is measured for the four possible paths in which light can propagate in the 2 × 2 optical switch, shown schematically in Fig. 4. In cross state, transmission is desired between the cross ports L1-R2 and L2-R1 while a low cross talk is desired between the bar ports L1-R1 and L2-R2. The coating should have a minimal effect on the transmission of the switch. This is verified by measuring the transmission of a coupler before and after application of the coating. Fig. 5 shows the measurements of two switches in cross state before coating and after coating with FDTS respectively ~12 nm PTFE. The wavelength range of the measured transmission is limited by the wavelength range of the used Santec laser and the bandwidth of the used fiber grating couplers that couple light into the optical structures. The transmission of the switch is normalized to the transmission of a reference waveguide. For the FDTS coating (see Fig. 5(a) and 5(b)) a high cross port transmission and a low bar port transmission is obtained before and after application of the coating. For a coupler with a PTFE coating, the measurements show inverted results compared to the transmission before the application of the coating (see Fig. 5(c) and 5(d)). With the PTFE coating the cross state is suppressed and most of the light travels between the bar ports instead, i.e. the bar port transmission is high while the cross port transmission is low. Hence, the use of PTFE as a dewetting coating in such active optical switches substantially degrades the optical performance of the switch in its cross state. The optical switch with a monolayer coating of an FDTS, however, demonstrates that the transmission spectra in the cross state are similar to switches without coating. No degradation in switch performance is observed in bar state with PTFE or FDTS.



(a)

(b)

(c)

(d)

Fig. 5. Measured transmission of an optical coupler in cross state (a) before and (b) after applying the FDTS coating and of a coupler on a different substrate (c) before and (d) after applying the PTFE coating.

IV. CONCLUSIONS AND OUTLOOK

The FDTS monolayer was found to be an applicable coating for the EWOD controlled active optical switch. This letter presented its suitability regarding the optical compatibility, EWOD actuation and long-term stability. As anti-sticking layer applied to the active optical surface, it enables the EWOD driven liquid motion over the coupler and transmission measurements validate that the effect on the optical performance is insignificant in contrast to PTFE. Long-term stability of the coating in contact with the liquids HPC and TPS has been proved experimentally. The thickness of the layer corresponds to the length of the molecule which is about 1nm. Contact angle measurements show that the dewetting properties of the FDTS are comparable to PTFE. However, the FDTS vapor phase deposition exhibits significant advantages as it leads to a substantial thinner film that forms stable chemical bonds to the Si/SiO$_2$ surfaces of the optical coupler whereas the thinnest possible dip coated PTFE layer has a thickness of 12 nm. Furthermore, the substrate materials for FDTS application are not limited to Si or SiO$_2$ as the silane can bond covalently to any hydroxilated surface.

REFERENCES

[1] R. Maboudian, W. R. Ashurst, and C. Carraro, “Self-assembled monolayers as anti-stiction coatings for MEMS: Characteristics and recent developments,” *Sens. Actuators A, Phys.*, no. 82, nos. 1–3, pp. 219–223, 2000. [Online]. Available: https://doi.org/10.1016/S0924-4247(99)00337-4

[2] M. Beck *et al.*, “Improving stamps for 10 nm level wafer scale nanoimprint lithography,” *Microelectron. Eng.*, vols. 61–62, pp. 441–448, Jul. 2002.

[3] H. Schift, S. Saxer, S. Park, C. Padeste, U. Pieles, and J. Gobrecht, “Controlled co-evaporation of silanes for nanoimprint stamps,” *Nanotechnology*, vol. 16, no. 5, pp. S171–S175, 2005.

[4] C.-W. Wu, Y.-K. Shen, S.-Y. Chuang, and C. S. Wei, “Anti-adhesive effects of diverse self-assembled monolayers in nanoimprint lithography,” *Sens. Actuators A, Phys.*, vol. 139, nos. 1–2, pp. 145–151, 2007.

[5] S. Si, L. Dittrich, and M. Hoffmann, “The NanoTuFe—Fabrication of large area periodic nanopatterns with tunable feature sizes at low cost,” *Microelectron. Eng.*, vol. 180, pp. 71–80, Aug. 2017.

[6] H. Gu, M. H. G. Duits, and F. Mugele, “A hybrid microfluidic chip with electrowetting functionality using ultraviolet (UV)-curable polymer,” *Lab Chip*, vol. 10, no. 12, pp. 1550–1556, 2010.

[7] B. J. Adzima and S. S. Velankar, “Pressure drops for droplet flows in microfluidic channels,” *J. Micromech. Microeng.*, vol. 16, no. 8, pp. 1504–1510, 2006.

[8] K. Handique, D. T. Burke, C. H. Mastrangelo, and M. A. Burns, “Nanoliter liquid metering in microchannels using hydrophobic patterns,” *Anal. Chem.*, vol. 72, no. 17, pp. 4100–4109, 2000.

[9] S. Günther *et al.*, “EWOD system designed for optical switching,” in *Proc. 30th IEEE Int. Conf. Micro Electro Mech. Syst. (MEMS)*, Las Vegas, NV, USA, Jan. 2017, pp. 1329–1332.

[10] H. D’heer *et al.*, “Nonvolatile liquid controlled adiabatic silicon photonics switch,” *J. Lightw. Technol.*, vol. 35, no. 14, pp. 2948–2954, Jul. 15, 2017.

[11] H. D’heer, C. L. Arce, J. Watté, K. Huybrechts, R. Baets, and D. Van Thourhout, Eds., “Broadband and non-volatile liquid controlled silicon photonics switch,” in *Conf. Lasers Electro-Opt. (CLEO) Tech. Dig.*, San Jose, CA, USA, Jun. 2016, Paper SM3G.6.

[12] H. D’heer *et al.*, “A 16 × 16 non-volatile silicon photonic switch circuit,” *IEEE Photon. Technol. Lett.*, vol. 30, no. 13, pp. 1258–1261, Jul. 1, 2018.

[13] H. D’heer, K. Saurav, W. Xie, C. L. Arce, J. Watté, and D. Van Thourhout, “Vertical liquid controlled adiabatic waveguide coupler,” *Opt. Express*, vol. 26, no. 16, pp. 19877–19884, 2018.

[14] Y. Yuan and T. R. Lee, “Contact angle and wetting properties,” in *Surface Science Techniques*, vol. 51, G. Bracco and B. Holst, Eds. Berlin, Germany: Springer, 2013, pp. 3–34.

[15] H. Moon, S. K. Cho, R. L. Garrell, and C.-J. Kim, “Low voltage electrowetting-on-dielectric,” *J. Appl. Phys.*, vol. 92, no. 7, pp. 4080–4087, 2002.

[16] I. Barbulovic-Nad, H. Yang, P. S. Park, and A. R. Wheeler, “Digital microfluidics for cell-based assays,” *Lab Chip*, vol. 8, no. 4, pp. 519–526, 2008.

[17] B. Koo and C.-J. Kim, “Evaluation of repeated electrowetting on three different fluoropolymer top coatings,” *J. Micromech. Microeng.*, vol. 23, no. 6, p. 067002, 2013.

[18] S. Terrab, A. M. Watson, C. Roath, J. T. Gopinath, and V. M. Bright, “Adaptive electrowetting lens-prism element,” *Opt. Express*, vol. 23, no. 20, pp. 25838–25845, 2015.

[19] R. Sharangpani, R. Singh, M. Drews, and K. Ivey, “Chemical vapor deposition and characterization of amorphous teflon fluoropolymerthin films,” *J. Electron. Mater.*, vol. 26, no. 4, pp. 402–409, 1997. [Online]. Available: https://doi.org/10.1007/s11664-997-0110-z

[20] H. Biederman, “RF sputtering of polymers and its potential application,” *Vacuum*, vol. 59, nos. 2–3, pp. 594–599, 2000.