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Investigation of the long-term adhesion and barrier properties of a PDMS-Parylene stack with PECVD ceramic interlayers for the conformal encapsulation of neural implants

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Abstract—In this paper, we investigate the long-term adhesion strength and barrier property of our recently proposed encapsulation stack that includes PDMS-Parylene C and PECVD interlayers (SiO₂ and SiC) for adhesion improvement.

To evaluate the adhesion strength of our proposed stack, the sample preparation consisted in depositing approximately 25 nm of SiC and 25 nm of SiO₂ on half wafers, previously coated with Parylene C. Next, 50 μm PDMS was spin-coated on top. Finally, the samples were detached from the Si wafer and soaked in a PBS solution at 67 °C to accelerate the aging process. Two samples were also implanted, subcutaneously, on the left and right subscapular regions of a rat. The optical inspection and peel tests performed after two months confirmed our preliminary findings and showed a significant improvement of the adhesion in our proposed encapsulation stack compared to the case of PDMS on Parylene C alone. In addition, the X-ray photoelectron spectroscopy (XPS) analysis at the interface between SiC and Parylene C showed different peaks for the interface compared to the reference spectra, which could be an indication of a chemical bond. Finally, water vapor transmission rate (WVTR) tests were performed to investigate the barrier property of our proposed encapsulation stack against water vapor transmission. The results demonstrated that the proposed stack acts as a significantly (two orders of magnitude) higher barrier against moisture compared to only Parylene C and PDMS encapsulation layers.

The proposed method yields a fully transparent encapsulation stack over a broad wavelength spectrum that can be used for the

conformal encapsulation of flexible devices and thus, making them compatible with techniques such as optical imaging and optogenetics.

Keywords—Conformal encapsulation; PDMS; Parylene C; adhesion; moisture barrier; accelerated soak test

I. INTRODUCTION

From all biocompatible polymers, Parylene (poly-paraxylylene (PPX)) C is considered to be a good candidate for insulating high-density conductive tracks in many implantable medical devices due to its ionic barrier property and the fact that it can be conformally deposited using chemical vapor deposition (CVD) processes [1-3]. The stiffness of Parylene C (~3.2GPa) is often beneficial to protect thin-film metal tracks from the longitudinal stress and strain applied during implantation. However, recent research showed damage in Parylene C itself, due to *in vivo* exposure [4-5]. Also, degradation of the Parylene encapsulation layer itself was reported caused by oxidation and chlorine abstraction of Parylene C surface after an *in vivo* experiment [6]. Polydimethylsiloxane (PDMS), on the other hand, does not provide the required mechanical stability for the thin film metal tracks but creates a more natural interface to the tissue due to its softer nature. Therefore, the combination of Parylene C and PDMS is advantageous to achieve the required flexibility,

reduce tissue damage, protect thin-film tracks against breakage, and prevent degradation of the Parylene C *in vivo*. However, to ensure long-term stability of the implantable devices during chronic *in vivo* experiments, a strong adhesion of PDMS to Parylene C is of paramount importance. To achieve this goal, we have previously proposed a method of improving the adhesion between the two materials, by creating chemical bonds using intermediate ceramic layers (silicon carbide (SiC) and silicon dioxide (SiO₂)) (Fig. 1) [7].

PDMS is known to have a strong adhesion to SiO₂ due to the presence of hydroxyl groups at the interface between the two materials. Due to its wide use in micro-electro-mechanical systems (MEMS), Si bond formation between SiO₂ and SiC has also been studied well [8]. However, the interface created between SiC and Parylene C needs further investigation.

In [9], the authors have reported the presence of covalent bonds between PDMS and Parylene C after investigating the selective bonding of the two materials under nitrogen and oxygen plasma treatments. Techniques such as X-ray photoelectron spectroscopy (XPS) and transmission electron microscopy energy-dispersive X-ray spectroscopy (TEM-EDS) were used for the interface analysis. It was claimed that these 3D soft and flexible structures could be used in implantable biomedical applications, however, no long-term investigation was reported on the adhesion strength. Implantable devices operate in harsh environments, and their reliability should, therefore, be considered and evaluated in non-standard dedicated representative tests. The long-term stability of the conformal coating is mainly affected by (a) the adhesion between all interfaces, as well as by (b) the water vapor and ionic barrier properties that the respective coatings can offer. Therefore, in this work, we present a thorough evaluation of the aforementioned properties under conditions that better match the environment that the implant will encounter when inside the body.

The paper is organized as follows: Section II describes the methodology used for the experiments, while Sections III and IV present and discuss the results obtained. Finally, conclusions are drawn in Section V.

II. METHODS

A. Sample Fabrication

To evaluate the proposed concept, relevant test structures were fabricated as illustrated in Fig. 2. To ease the release of the final structures from the Si wafers, a de-adhesive material from Sigma-Aldrich (Trichloro (1*H*,1*H*,2*H*,2*H*-perfluorooctyl) silane was first coated on a clean silicon wafer in a vacuum environment created inside a desiccator. Next, a 5 μm Parylene layer was deposited using an SCS PDS 2010 Parylene coater that employs a chemical vapor deposition (CVD) technique at room temperature. Following the Parylene C deposition, the proposed SiC-SiO₂ stack was deposited at 180 $^{\circ}\text{C}$ using plasma-enhanced chemical vapor deposition (PECVD) processes

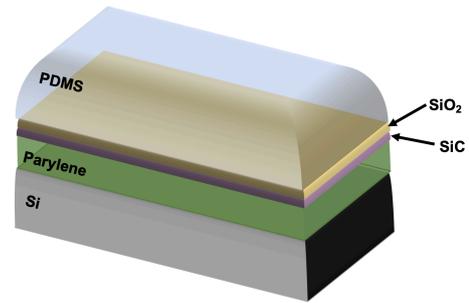


Fig. 1. The proposed encapsulation stack including ceramic layers as adhesion improvement layers.

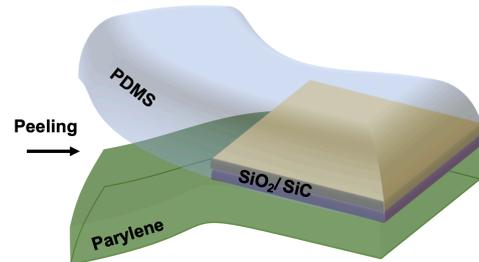


Fig. 2. Schematic representation of layer stack for test samples. Samples were only partially covered with the ceramic layers, to allow for easy testing.

(Elettrovava Amor) in the same chamber. Approximately 25 nm of SiC were deposited on Parylene using 1.6 sccm SiH₄, 3.7 sccm CH₄, and 200 sccm H₂ at 6 W power and 2 mbar pressure. Next, approximately 25 nm of SiO₂ were deposited using the following deposition parameters: 1 sccm SiH₄, 20 sccm CO₂, 1.4 mbar pressure, and 20 W power. Finally, PDMS (Dow Corning Sylgard 184) was mixed with a curing agent at a 10:1 ratio, spin-coated on top of the ceramic layers at 1250 rpm, and cured at 75 $^{\circ}\text{C}$ for 3 hours. This resulted in a 50 μm thick layer.

To evaluate the influence of the ceramic layers on the adhesion between PDMS and Parylene C, the proposed layers were deposited only on half of the wafer (using metal masks to cover the other half during deposition).

B. Characterization

1) Accelerated aging

To evaluate the long-term performance of our encapsulation stack samples were exposed to elevated temperature to accelerate chemical reactions. To simulate oxidizing and ionic conditions inside the human body samples were soaked in a 1X phosphate-buffered saline (PBS) solution. The 10-degree rule states that increasing the temperature by about 10 $^{\circ}\text{C}$ roughly doubles the rate of polymer reactions [10]. Therefore, maintaining a polymer at 67 $^{\circ}\text{C}$ for 2 months is equivalent to aging it for 16 months at 37 $^{\circ}\text{C}$. After two months, the samples were optically inspected, and peel tests were performed to evaluate the adhesion strength.

2) *In vivo* experiments

To study the longer-term adhesion strength of our proposed encapsulation stack, *in vivo* experiments were performed for two months. The results of the experiments could, potentially, provide a more realistic evaluation compared to the aforementioned accelerated aging study because the samples are subjected to an environment similar to what an implantable device would encounter inside the body.

The samples presented in Fig. 2 were disinfected with isopropyl alcohol and rinsed with distilled water before implantation. Next, two samples were implanted subcutaneously, on the left and right subscapular regions of a rat, and the wounds were later closed by using two sutures and a vet bond. The samples were optically inspected after explantation, followed by a peel test to evaluate the adhesion.

3) XPS

XPS was used to study the SiC-Parylene C interface. Two samples comprising 10 μm Parylene C on a Si wafer and 0.5 μm SiC on a Si wafer, were considered as reference samples. A model sample (test sample) customized for XPS analysis was prepared by depositing less than 25 nm of SiC on Parylene C. The XPS analysis was carried out in vacuum (10^{-9} mbar) using a PHI-TFA XPS spectrometer (Physical Electronic Inc.), equipped with an Al-monochromatic X-ray source. The analysed area was 0.7 mm in diameter and the analysis depth was approximately 10 nm. The survey spectra were collected from 0 to 1000 eV. Next, high-resolution multiplex scans of the measured peaks were recorded using a pass energy of 23.5 eV with a step size of 0.1 eV at a take-off angle of 45° with respect to the sample surface. The collected spectra were analysed using Multipak v8.0 (Physical Electronics Inc.).

To reach the interface and remove any potential carbon contaminants, the surface was rastered using a 2 keV Ar ion beam over an area of $4 \times 4 \text{ mm}^2$. The emission current was 20 mA, and the Ar pressure was 10 mPa. Subsequent XPS measurements were conducted after 1, 4, 8, and 15 minutes of sputtering. SiC and Parylene C can be recognised and distinguished by detecting the silicon (Si) and chlorine (Cl) peaks, respectively.

4) Water Vapor Transmission Rate (WVTR) test

Apart from improving the adhesion substantially, ceramic layers also act as a barrier layer against moisture. To compare the barrier property of different materials against water vapor, a WVTR test was performed using Permatran-W 3/33 from MOCON Inc. The encapsulation film was placed between two chambers, one filled with water vapor and the other one with nitrogen carrier gas. The amount of water vapor permeation from one chamber to the other, through the encapsulation layer, was measured with the units of $\text{g m}^{-2} \text{d}^{-1}$.

The evaluation was performed for different flexible samples, including Parylene C alone, PDMS-Parylene C bi-layer stacks, PDMS-SiO₂-Parylene C stacks, as well as PDMS-SiO₂-SiC-Parylene C stacks. The samples were transferred to the WVTR test tool using an Al foil as shown in Fig. 3 (a). Then, the Al foil was removed (Fig. 3 (b)) and the encapsulation film separated a chamber with 100 % relative humidity (RH) at 38 °C from the dry nitrogen flow, which was analysed for moisture content and flow rate.

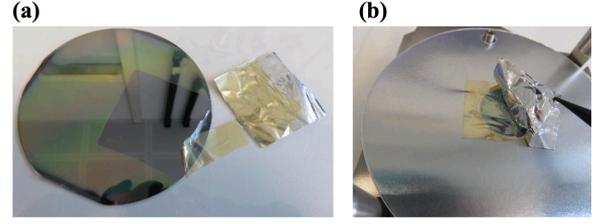


Fig. 3. Method used to (a) transfer the flexible encapsulation layer first to an Al foil and (b) install it in the tool.

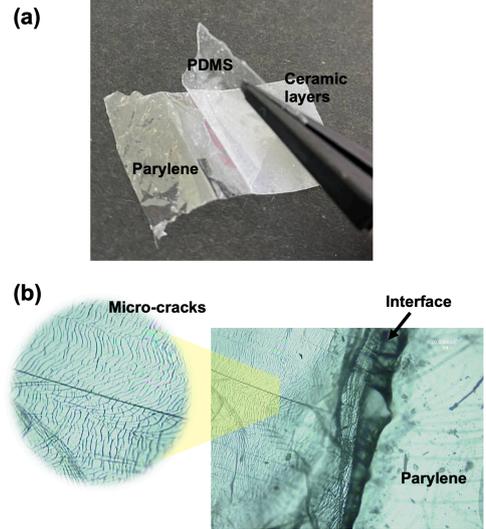


Fig. 4. (a) Delamination of PDMS from parylene after two months soak test, (b) Micro-cracks appearing on the sample caused by the force applied during peel test after two months soak test at 67 °C.

5) Optical transmittance measurement

The optical transparency of the encapsulation layer is of paramount importance for techniques that play a significant role in modern neuroscientific studies, including optogenetics and *in vivo* optical imaging methods (e.g., calcium imaging or fluorescence imaging). Therefore, a quantitative evaluation of the optical transparency of our proposed multilayer encapsulation stack was conducted using a Perkin Elmer Lambda 950 UV/Vis (PerkinElmer, Waltham, Massachusetts). The wavelength used for the measurement ranged from 300 nm to 1200 nm.

III. RESULTS

1) Accelerated aging

The peel test, performed after the two-month-long accelerated aging experiment, revealed no delamination for the samples having ceramic interlayers. As shown in Fig. 4 (a), PDMS was peeled off from Parylene C only over the region with no ceramic layers. As depicted in the optical microscopy image shown in Fig.4 (b), the presence of micro-cracks on the ceramic layers is related to the force applied during the peel test. These observations are in accordance with our previous findings in which a strong adhesion for the proposed encapsulation layer was demonstrated and prove that even a two-month soak test at 67 °C does not lead to a deterioration of the adhesion strength.

2) *In vivo* experiments

Fig. 5 (a) illustrates the optical microscopy results from the explanted samples after a two-month implantation period. Delamination of PDMS from Parylene C was observed in the region without ceramic layers and no delamination was observed for the area with PDMS-SiO₂-SiC-Parylene C multilayers. However, it seems that there is some discoloration suggesting some changes in the stack with interlayers. The peel tests were performed manually for the explanted samples. As shown in Fig. 5 (b), the PDMS layer could easily be peeled off from the areas with no ceramic interlayers. However, once the ceramic interlayers were reached, peeling was not possible anymore, and the PDMS layer was torn at the edge of this region. The cracks that appeared on the area with ceramic layers were due to the force applied during the peel test.

3) XPS

The survey spectrum of the test sample, shown in Fig. 6 (a), indicates the presence of O, C, Cl, and Si elements in 53.1%, 34.4%, 1.1%, and 11.4% atomic concentrations. The high-resolution XPS spectra for the SiC and Parylene C reference samples, shown in Fig. 6 (b, c), indicate a Si 2p_{3/2} peak at 99.4 eV and a Cl 2p_{3/2} peak at 202 eV (dash-line). Moreover, a C1s peak at 285eV and an O1s peak at ~533eV for both samples were observed. The high-resolution spectra for the test sample are shown for Si2p_{3/2}, Si2s, C1s, O1s, and Cl2p in Fig. 6 (d, e, f, g, h). The presence of the chlorine peak is strongly related to the

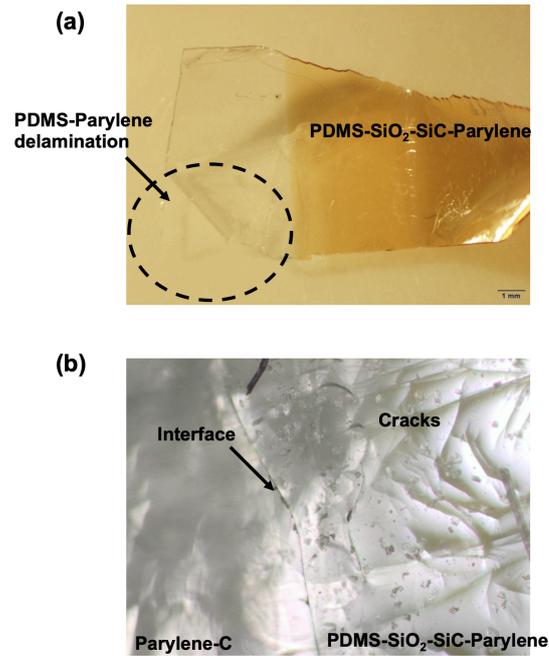


Fig. 5. Optical image of the sample after a two-month implantation, (a) PDMS delamination from Parylene-C on the region without ceramic layers, (b) sample after the peel test

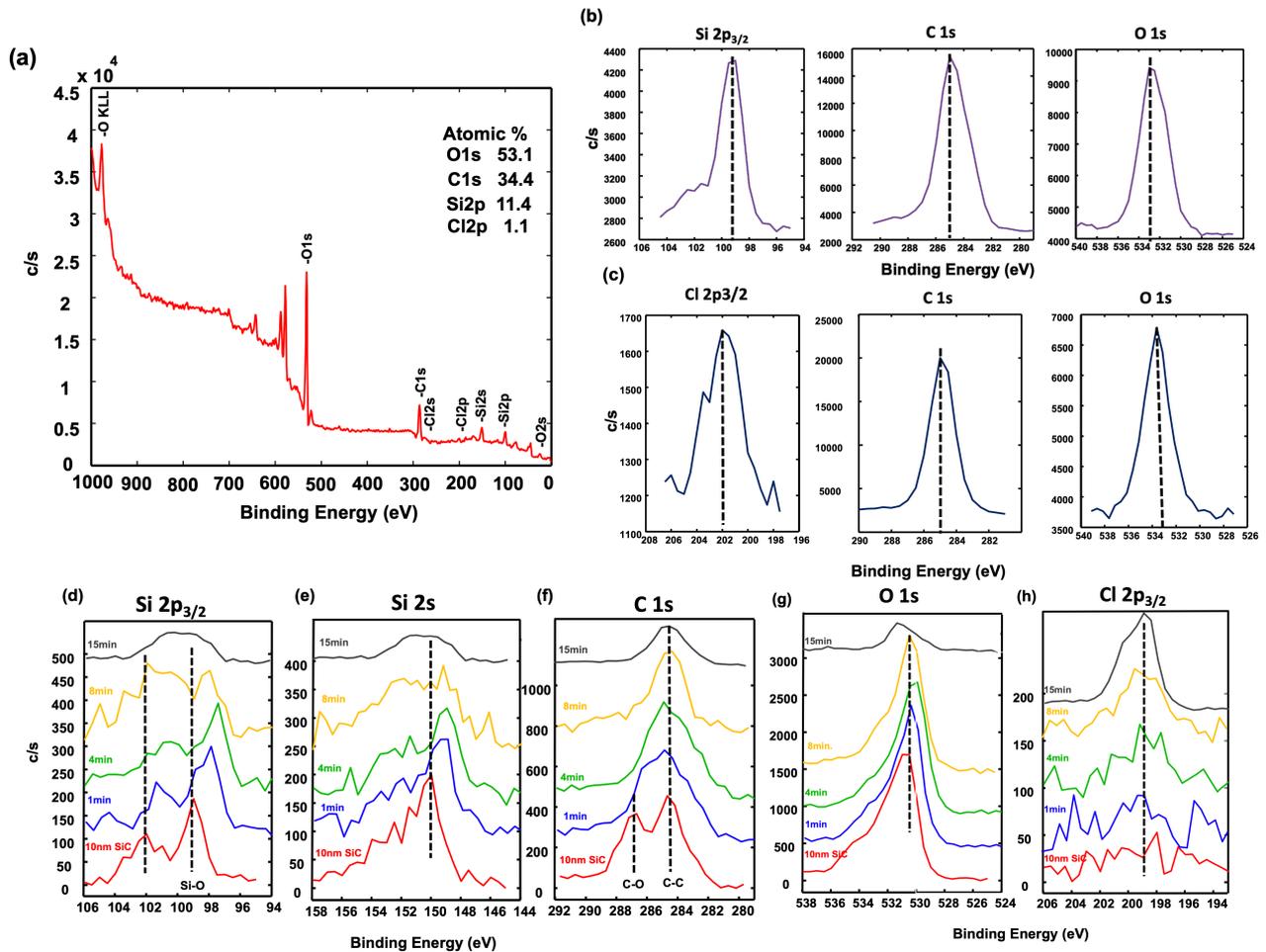


Fig. 6. XPS results (a) Survey spectra of the test sample, (b) Si2p, C1s, and O1s peaks for SiC reference sample, (c) Cl2p, C1s, and O1s peaks for Parylene C sample, (d) Si2p, (e) Si2s, (f) C1s, (g) O1s, and (h) Cl2p high resolution spectra for the test sample.

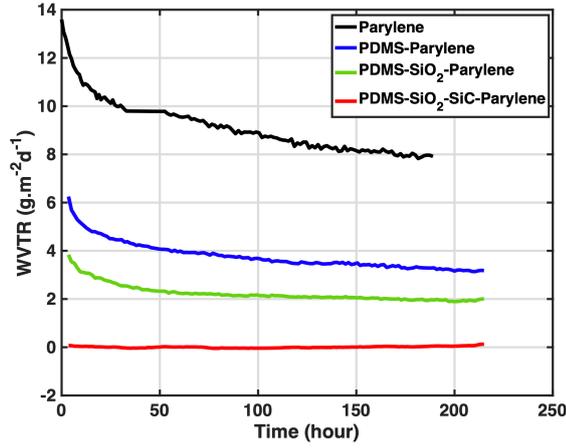


Fig. 7. WVTR result comparing different encapsulation stacks.

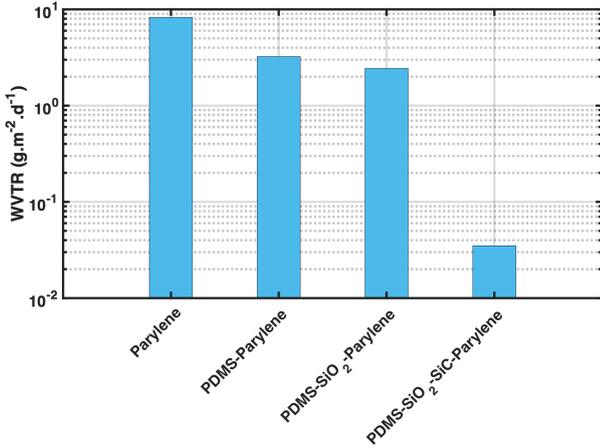


Fig. 8. WVTR results comparing different encapsulation stacks after 100 hours stabilization.

presence of Parylene C. After sputtering for 1, 4, and 8 minutes, an increase in this peak was observed. A strong Cl peak appeared after 15 minutes of sputtering, which originates from Parylene-C. High-resolution spectra for O1s and Cl2p for both the reference and test samples showed the same results. In the Cl1s spectrum, both the reference and test samples contained a peak at 285 eV arisen from the C-C bond, as shown in Fig. 6 (f). At the interface, a new peak at 286.5eV appeared, indicating either a C-O or a C-Cl bond formation. However, after longer sputtering times, this peak was not visible anymore. The high-resolution spectra of Si 2p_{3/2} shown in Fig. 6 (d) also indicate the presence of another peak close to the Si-O peak, which could be a characteristic of a Si-based bond at the interface [11]. This peak, however, is still present after sputtering, indicating that it is not related to contamination. Compared to the reference, the different peaks observed on the test sample could be an indication that different chemical bonds are being formed between the two materials under investigation. However, more characterization is necessary to be able to define the nature as well as properties of such chemical bonds.

4) WVTR test

The conducted WVTR tests showed a clear improvement of the barrier property when ceramic layers are present at the

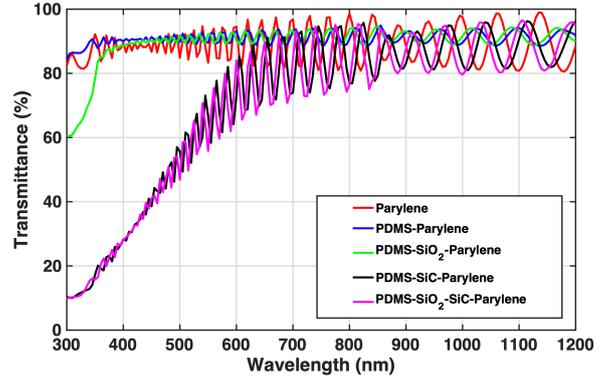


Fig. 9. Optical transmittance measurement for different encapsulation stacks.

interface between PDMS and Parylene C. The WVTR was calculated based on measurements conducted for 100 hours after which a steady state was considered to be established (Fig. 7). Comparing the results obtained for Parylene C alone versus the PDMS-Parylene C stack, it can be concluded that the combination of materials leads to a lower WVTR. As indicated in Fig. 8, adding barrier layers to the encapsulation stack can lead to an increase in the barrier efficiency by orders of magnitude. However, since the value is close to the upper limit of the investigation equipment, it might be difficult to accurately determine the WVTR for better barriers.

5) Optical transmittance measurement

The optical transmittance measurements shown in Fig. 9, for Parylene C alone, the PDMS-Parylene C stack, and the PDMS-SiO₂-Parylene C stack, show more than 80% transmittance over a broad wavelength spectrum, from 300 to 1200 nm, and specifically above 85% for the 470nm (blue light) wavelength that is mainly used in optogenetics applications. Adding SiC to the encapsulation stack shows that the optical transmittance for wavelengths higher than 600 nm is greater than 70%, which is acceptable for optical imaging. However, the optical transmittance drops to 48% for a 470 nm wavelength. This could be further improved by depositing thinner SiC layers.

IV. DISCUSSION

To improve the adhesion of PDMS to Parylene C, SiC and SiO₂ were used as intermediate adhesion layers. A strong adhesion among the different layers that future active implantable medical devices will be comprised of is of paramount importance, as this ensures the long-term stability of the device when implanted inside the harsh environment of the human body. To this end, adhesion evaluation has to be done under similar conditions. One approach consists of soaking the samples under test at 67 °C, which accelerates the aging process and could cause an early failure of the adhesion. This temperature was also chosen to make the result comparable to other works in the literature. However, [10], [12] suggest that the accelerated temperature should not be so high to initiate physical or chemical processes that are unlikely to be involved in normal aging. Therefore, changes in the structure of the material cannot be excluded at this temperature. Nevertheless,

no delamination was observed when performing the experiments for two months. Although the PBS solution commonly used for these types of tests can resemble the environment that an implant will see in the human body, it still lacks the complexity of the targeted *in vivo* scenario. Therefore, it is possible that the failure mechanisms usually observed during the *in vivo* experiments are neither accurately nor fully captured by such tests [6]. However, during the *in vivo* experiments presented here, neither delamination nor peeling was observed for our proposed encapsulation stack after explantation. However, it should be noted that a longer *in vivo* experiment might reveal different results as it seems after this two-month study there is some discoloration in the explanted samples with ceramic interlayers suggesting some changes in these interlayers.

These results are an indication of the strong adhesion between the different layers proposed in our encapsulation stack. The XPS results also confirmed these findings by indicating different peaks at the interface between SiC and Parylene C compared to reference materials. However, more analysis is needed to further understand the nature of this bond.

As presented in a previous section of this paper, it is expected that the ceramic layers can also act as a barrier layer against moisture, which is essential, especially when a good adhesion cannot be achieved. To better understand the resulting barrier properties of the proposed stack, a water vapor transmission rate test was employed. The results have shown that by stacking two polymer layers, the WVTR is reduced. However, adding ceramic interlayers between the two polymers leads to a significant (two orders of magnitude) improvement in the moisture barrier property of the encapsulation layer.

Conformal encapsulation has the potential to solve important miniaturization challenges in the field of active neural interfaces and become the enabling factor for the realization of mm-sized implants for bioelectronic medicine [13]. Alternative encapsulation approaches based on a combination of polymers and ceramic multilayers, or even polymers alone, have been recently proposed in literature [8], [14-16]. Our proposed method allows for a gradual adjustment of the mechanical properties of the encapsulation, from a relatively rigid (Parylene C) layer to a softer one (PDMS), with properties similar to those of soft tissues, while yielding a fully transparent encapsulation stack over a broad wavelength spectrum. Therefore, the proposed solution can be used for the conformal protection of a variety of flexible devices and even be combined with transparent conductors [17], making the device suitable for optical imaging such as calcium imaging and optogenetic applications.

V. CONCLUSION

In this paper, we investigated the long-term effect of thin ceramic interlayers (SiO₂, SiC) used to improve the PDMS-to-Parylene C adhesion for the encapsulation of implantable devices. The results show that the adhesion of PDMS to Parylene C after using intermediate SiO₂ and SiC layers is significantly improved, as no delamination was observed after two months of accelerated aging, for which the samples were

soaked at 67 °C. Similarly, this multilayer stack adhesion was not compromised even after *in vivo* validation, where the samples were subcutaneously implanted for two months. The improvement in adhesion strength was also confirmed by XPS analysis which showed different peaks at the interface of SiC-Parylene C compared to reference samples.

The proposed ceramic interlayers have a dual function, acting at the same time as a barrier layer against water permeation. The WVTR test results have shown an improvement of about two orders of magnitude for the full stack compared to only using polymers without any additional ceramic layers.

As a future work, more emphasis will be put on further investigating the adhesion strength under the effect of bias voltages at elevated temperatures. It is expected that such experiments may reveal failure mechanisms that cannot be observed when passive tests are performed [18].

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