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Parylene-C as High Performance Encapsulation Material for Implantable Sensors

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Abstract

This work presents high performance multilayer coatings for a wide range of applications and substrate materials including medical implants and implantable bio-transducers. This was done by combining the glow discharge polymerization and the chemical vapor deposition of Parylene-C processes in a special designed cluster-closed coating system. An outstanding performance of the resulting multilayer in terms of adhesion to substrate and barrier properties was observed by performing the wet adhesion test in normal saline (0.9% NaCl).

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1. Introduction

The use of polymeric materials in biomedical field such as medical implants and implantable bio-transducers is becoming a trend [1]. Not only as a substrate material, but also as encapsulation; polymers are a very attractive material due to their unique properties [2,3]. Starting from its biocompatibility [4], mechanical, chemical, electrical properties and its friendly process [5], Parylene is gaining bigger share in the medical market technology in the last few years.

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Although it shows very unique advantages, Parylene suffers from severe limitations mainly summarized in its poor adhesion to substrate (metals, ceramics and polymers) and barrier properties [6].

In order to tackle these limitations, a dip-coating silane-based adhesion promoter (A-174) was introduced by the industry [7]. Some drawbacks accompany the A-174 adhesion promoter such as the low performance on some surfaces such as metals.

Glow discharge is a known methodology for its capability to modify the surfaces. Depending on the type of the gas used during the process the modification is ranging between the simple cleaning of the surface to the generation of very thin layer (nm range) of polymer [8]. The resulting surfaces are characterized by their modified surface energy, and by the radical content in the form of dangling bond or as trapped radicals in the generated polymer matrix [8]. In addition to the previously stated benefits of glow discharge, this process is very clean and it is known for its suitability to a wide range of materials [8].

Modifying the surface energy of the target substrate to make it suitable for Parylene and at the same time create and/or increase the amount of available radicals on the surface offers an excellent alternative to increase the adhesion and improve the barrier properties of the deposited Parylene films [6,9].

This work shows that the combination of both processes (plasma polymerization and chemical vapor deposition of Parylene) brings tremendous advantages in terms of Parylene adhesion to various types of substrate materials including metals.

2. Experimental procedures

2.1. Deposition processes

The combination of both deposition processes (glow discharge and Parylene) was done in a specially designed and implemented system. The closed-cluster system offers several advantages in terms of lowering the cross contamination between the different processes, the cleanness of the interfaces and its processing flexibility; more details about the system are covered somewhere else [10].

Magnetron enhanced glow discharge at 15 kHz of TMS (Trimethylsilane) was used as surface and interface engineering methodology to make the surface of the target substrate more suitable for Parylene film which was deposited in a consequent step. The concept and its advantages is discussed and covered elsewhere [8,10,11]. For the glow discharge process different combination of gases are used.

A Parylene deposition process was done directly after the glow discharge process by moving the samples between the processing chambers under high vacuum which guarantees a very clean interface and a high reactivity of the modified surface. Parylene deposition followed a standard process (Gorham's process) [5] where the used parameters are the same as in [10]. Table 1 summarizes the used process recipes and their parameters.

Table 1. Deposition processes steps and the parameters of used plasma recipes, Ar: Argon, TMS: Trimethylsilane, O₂: Oxygen

| Recipe index | Coating process sequence | Plasma activation (Ar / O ₂) | | | Plasma polymerization (TMS) | | |
|--------------|---|--|-------|--------|-----------------------------|-------|--------|
| | | Flow rate | Power | Time | Flow rate | Power | Time |
| | | [sccm] | [W] | [min.] | [sccm] | [W] | [min.] |
| 1 | Ar activation then TMS polymerization then Parylene deposition | 5 | 120 | 5 | 3 | 80 | 10 |
| 2 | O ₂ activation then Parylene deposition | 3 | 120 | 5 | | | |
| 3 | Parylene deposition only | | | | | | |

2.2. Substrates and coating characterization

Four groups of samples were tested; each one contains three samples. The first one consists of platinum (Pt) evaporated on glass coated with recipe 1 (see Table1). The second one consists of polyimide foils (Dupont Pyralux AP) coated with recipe 1; the foils are partially masked by a tape so that Parylene encapsulates only half of each foil area while the other half is free of Parylene. The third one is also Pt on glass, treated only by oxygen followed by Parylene deposition without TMS deposition (recipe 2 in Table 1). The last group is a control one (Pt on glass), coated only with Parylene without any plasma process.

All samples are boiled in normal saline (0.9% NaCl) for one hour and at the end of the test they are monitored under microscope (Nikon OPTIPHOT 200).

3. Results and Discussion

3.1. The wet adhesion of Parylene films

The boiled samples are examined under microscope to observe any delamination in the films and/or any accumulation of water underneath at the interface between the coating and the sample. Depending on the processing recipe the results ranged from total removal to healthy films (without any defect) as summarized in table 2.

Table 2. Parylene film status after wet adhesion test

| Recipe index | Group of samples | Film status after 60 min of boiling in normal saline |
|--------------|------------------|--|
| 1 | 1, 2 | Fully intact |
| 2 | 3 | Partially removed |
| 3 | 4 | Fully removed |

Figure 1 shows a sample of group 1, 2, and 3 after boiling test.

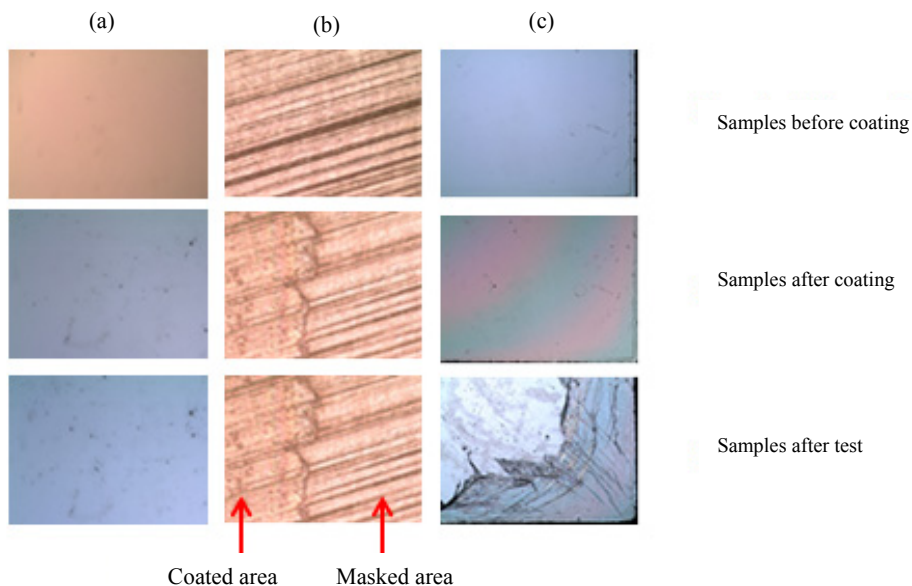


Fig. 1. The wet adhesion test of the multilayer combinations (a) Pt evaporated on glass and, (b) Polyimide foil processed by recipe 1, (c) Pt evaporated on glass processed by recipe 2

3.2. Discussion:

The drastic improvement in adhesion of Parylene to substrate by using a plasma polymer nano-film (TMS in this case) as adhesion promoter originated from two mechanisms. First is the similarity in surface energy between the modified surface of the sample and the deposited Parylene film. This similarity was examined by measuring the contact angle of water on top of the surface. A modified surface by TMS plasma polymer has a water contact angle of (120°) [8] while the water contact angle of Parylene is (93°) [10].

The second is the huge amount of radicals and /or free radicals that characterize both the plasma deposited nano-films and Parylene films [8, 11]. This leads to a radical-radical interaction between both layers and increase the stability and the strength of the adhesion.

4. Conclusion

The wet adhesion of Parylene-C films on top of metallic and polymeric substrates was investigated. Several multilayer combinations were tested. Superior adhesion performance was achieved by using plasma nano-film as adhesion promoter.

The improvement of Parylene adhesion is originated from the similarity of surface energy between the modified surface and Parylene film. Also the radical-radical bonding, between the plasma polymer and Parylene, plays a role in strengthening and stabilizing the adhesion.

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References

- [1] J. R. Davis (Ed), Handbook of Materials for Medical Devices, ASM International, Ohio, 2003.
- [2] T. Stieglitz, M. Schuetter, K. P. Koch, Implantable biomedical microsystems for neural prostheses, Eng. Med. Biol. Mag. IEEE 24 (2005), 58-65.
- [3] C. Hassler, T. Boretius, T. Stieglitz, Polymers for neural implants, J. Polym. Sci., Part B: Polym. Phys. 49 (2011), 18-33.
- [4] C. P. Tan, H. G. Craighead, Surface Engineering and Patterning Using Parylene for Biological Applications, Materials 3 (2010), 1803-1832.
- [5] W. F. Gorham, A New, General Synthetic Method for the Preparation of Linear Poly-p-xylylenes, J. Polym. Sci., Part A-1: Polym. Chem. 4 (1966), 3027-3039.
- [6] H. Yasuda, Q. S. Yu, M. Chen, Interfacial factors in corrosion protection: an EIS study of model systems, Prog. Org. Coat. 41 (2001), 273-279.
- [7] D.D. Stewart (Union Carbide Corporation), Process for adhering poly-p-xylylene to substrates using silane primers and articles obtained thereby, US 3600216, 1971.
- [8] H. Yasuda, Luminous Chemical Vapor Deposition and Interface Engineering Surfactant Science Series, A. T. Hubbard (Ed), Marcel Dekker, NY, USA 2005.
- [9] R. K. Sadhir, W. J. James, H. K. Yasuda, A. K. Sharma, The adhesion of glow-discharge polymers, Silastic and Parylene to implantable platinum electrodes: results of tensile pull tests after exposure to isotonic sodium chloride, Biomaterials 2 (1981), 239-243.
- [10] D. Zeniech, A. Bajwa, L. Ledernez, G. Urban, Effect of Plasma Treatments and Plasma-Polymerized Films on the Adhesion of Parylene-C to Substrates, Plasma Processes Polym. 10 (2013), 1081-1089.
- [11] H. Yasuda, Magnetron-AF Plasma Polymerization, Plasma Process. Polym. 5 (2008), 215-227.