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A Simple Method to Allow Parylene-C Coatings on Gold Substrates †

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Abstract: Parylene-C, a biocompatible coating material, does not adhere well to metals. This is problematic especially for biomedical devices containing gold structures. In this contribution, we present a simple method to adhere Parylene-C to gold-coated substrates based on thiol-based adhesion promoters. The effectiveness of the adhesion promoters has been demonstrated by pull-tests on autoclaved Parylene-C coated gold-substrates.

Keywords: Parylene coatings; gold substrates; adhesion promoter; biomedical devices; biocompatibility

1. Introduction

Parylene-C is a biocompatible certified material used to protect biomedical devices and electronics from moisture and chemicals [1–3]. The chemical vapor deposition process of Parylene results in thin conformal layers. This process takes place at low pressure of typically 10 Pa and at room temperature. Unfortunately, Parylene does not adhere well to various substrates such as SiO_2, gold, silver, and platinum, materials that are often used in biomedical devices. Adhesion promoters can play an important role to realize functional Parylene-to-substrate coatings. The mostly used and investigated adhesion promoter for Parylene is 3-trimethoxysilylpropyl methacrylate (Figure 1a; silane A174) [4]. The coupling of A174 to substrates containing -OH groups on its surface is realized by hydrogen bonding. The methacrylate group of A174 is the coupling agent to Parylene-C.

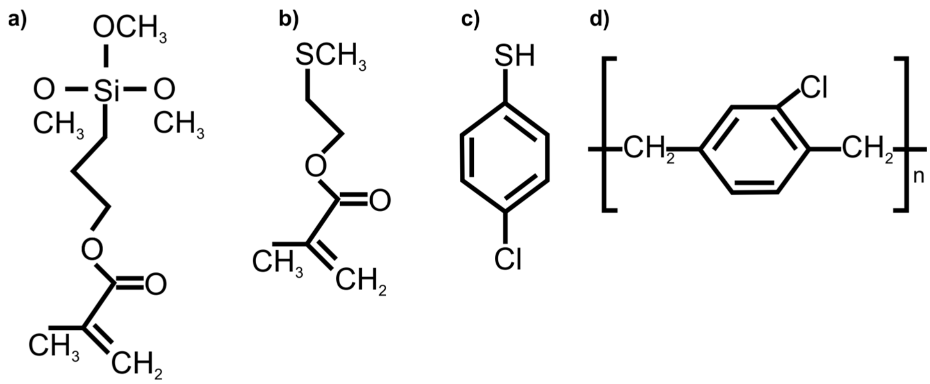

Figure 1. Three adhesion promoters (**a**) Silane A174; (**b**) 2-methylthio ethyl methacrylate; and (**c**) 4-chlorothiophenol; (**d**) Parylene-C.

However, to the best of our knowledge, effective coupling between Parylene-C and gold by silane A174 has not been realized. Zeniieh et al., presented a successful protocol to deposit Parylene-C to gold by applying a custom-built multi-chamber system (a plasma chamber and a Parylene deposition chamber coupled through a load lock) and a plasma polymerization pre-treatment step using highly-flammable trimethylsilane (CAS Nr.: 993-07-7) [5].

Gold, silver, and copper are known to have a strong affinity to thiol (-SH) groups [6]. We have investigated the effectiveness of two thiol based adhesion promoter; 2-methylthio ethyl methacrylate (CAS Nr.: 14216-23-0) and 4-chlorothiophenol (CAS Nr.: 106-54-7) for the deposition of Parylene-C to gold (the molecular structures are depicted in Figure 1b and 1c). 2-methylthio ethyl methacrylate and silane A174 have the same methacrylate group to couple Parylene-C. The methylthio group is responsible for the coupling to gold substrates. Aromatic primers have a higher temperature stability compared to non-aromatic primers. Because a Parylene-C to gold adhesion promoter is desired that withstands an autoclaving decontamination step of at least 121 °C for 15 min, the effectiveness of 4-chlorothiophenol as primer was investigated.

2. Materials and Methods

2.1. Wafer Preparation

Two silicon wafers with a dimeter of 100 mm and a thickness of 525 µm were exposed to a thermal oxidation step to obtain a 500 nm SiO_2 layer on the silicon wafer. A 50 nm titanium layer and a 100 nm gold layer were deposited by physical vapor deposition.

2.2. Adhesion Promoter and Parylene-C Deposition

The gold-coated substrates (Figure 2) were dipped in adhesion promoter solution for two minutes. The adhesion promoter solution consists of 1gram 2-methylthio ethyl methacrylate, or 1gram of 4-chlorothiophenol diluted in 1 L propanol. All chemicals were obtained from Th. Geyer (Renningen, Germany).

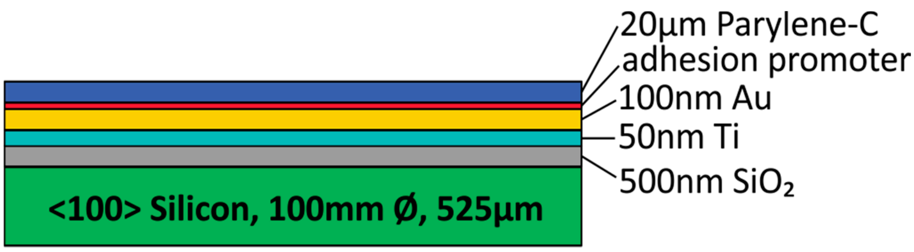

Figure 2. A schematic of a Parylene-C coated gold layer on a Si-substrate.

Subsequently, the wafers were rinsed with pure propanol and dried with nitrogen gas. Parylene-C was deposited with a Labcoater series 300 (PPS Rosenheim, Germany). Fifty grams of Parylene-C dimer vaporized at 122 °C, deposited at a pressure of 10 Pa for 14 h, yielded a 20 µm deposited layer.

2.3. Adhesion Test Method

In order to determine the adhesion strength of the bonding between gold and Parylene-C pull-tests were performed. The wafers were diced in 1cm wide strips (Figure 3) and the adhesion was tested by a manual pull-test (Figure 4). The test method result shows if the bonding strength between Parylene-C and the gold-coated substrate is stronger (breakage of the Parylene-C layer) or weaker (delamination of the Parylene-C layer from the substrate) than the tensile strength of Parylene-C.

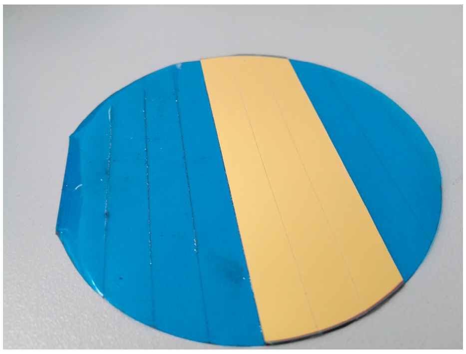

Figure 3. A Parylene-C coated gold substrate diced in 1 cm wide stripes.

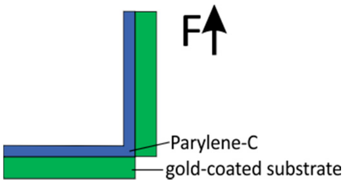

Figure 4. Manual pull-test. The gold-coated substrate was scratched with a glass-scraper and broken. By applying a force, the adhesion of Parylene-C to the gold layer was tested.

3. Results and Discussion

The prepared Parylene-C coated substrates were sterilized in an autoclave (at 121 °C for 15 min). This high humid exposure is a standard method to assure the sterility of medical devices. The results of manual pull tests showed that with both adhesion promoters, an effective Parylene-C to gold connection was achieved. Delamination of the substrates did not occur. Even after autoclaving the coated structures, the effective connection was maintained (Figure 5).

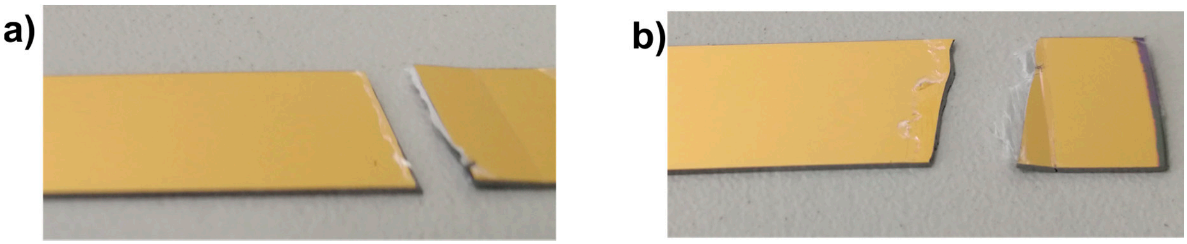

Figure 5. Pull test results of Parylene-C coated gold-substrates, autoclaved at 121 °C for 15 min. (**a**) 2-methylthio ethyl methacrylate; and (**b**) 4-chlorothiophenol. With both adhesion promoters, the 20 μm Parylene-C layer did not detach from the gold-substrate. The corresponding bonding strength was higher than the tensile strength [a preliminary tensile strength of 1.4 N/cm was measured]. This shows that effective Parylene-C to gold connections were achieved.

The 2-methylthio ethyl methacrylate primer has the same methacrylate coupling group to Parylene-C as the primer silane A174 (see Figure 1a,b). For the 4-chlorothiophenol primer, we expect that the chloride bond coupled to the aromatic ring plays an important role in the coupling to Parylene-C (see Figure 1c).

4. Conclusions

Our experiments show the effective connection between Parylene-C and gold by applying 2-methylthio ethyl methacrylate and 4-chlorothiophenol as adhesion promoter. The connections realized by both thiol based adhesion promoters were tested by performing manual pull-tests on autoclaved (121 °C for 15 min) Parylene-C coated substrates. The fast and simple method can easily be implemented for the standard Parylene coating process. It requires a two minute dipping and subsequent drying step before placing the substrates in the Parylene coater. The method enables the growth of Parylene coatings on biomedical devices containing gold structures where biocompatibility must be assured.

Conflicts of Interest: The authors declare no conflict of interest.

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