Preparation of Contact Lens Materials for Real-Time Protein Adsorption Studies

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Statement of Purpose: Protein buildup onto contact lens type materials can lead to inflammatory reactions within the eye. It is of interest both to the researcher, fundamentally, and to the contact lens industry, practically, how different types of materials will react to proteins. In order to address some of these concerns we developed a procedure for making model contact lens materials out of polydimethylsiloxane (PDMS) polymers assembled onto model silicon oxide surfaces. The idea was to later use these surfaces in quartz crystal microbalance with dissipation (QCM-D) protein adsorption studies. It was necessary to form thin nanometer thick films with uniform roughness and that the films were stable in aqueous buffer solutions. Since this work is ongoing, in addition to discussing the preparation of the materials we will show an example protein adsorption experiment that should approximate what we expect for the PDMS surface.

Methods: Silicon wafers from Silicon Quest International were <100> optically polished on one side and cut into ~1 in squares. The Si substrates were cleaned in 3:1 solution of sulfuric acid and hydrogen peroxide for 10-20 minutes. The substrates were then rinsed with Millipore water and blown dry with N_2 before being used immediately. Anhydrous 2,2,4-trimethylpentane (isooctane) was used as received from Aldrich. Sylgard 184 was used as received from Dow Corning. Solutions of 1 wt% and 10 wt% were prepared as follows:

- 1 wt%: mixed 89.3 mg silicone elastomer with 10.0 mg curing agent for 30-60 s then added 10.063 g isooctane.

- 10 wt%: mixed 0.9589 g silicone elastomer with 99.8 mg curing agent for 30-60 s then added 10.030 g isooctane

Ellipsometry of the bare Si wafers was taken before spin casting so that optical constants of the bare substrate could be extracted for thickness determination. Five separate spots were taken to estimate uniformity.

Solutions were filtered through a 0.45 μm PTFE syringe filter before injecting 4-5 drops onto the cleaned Si wafers. Substrates were spun at 3000 rpm for 30 – 60 s until dry. Another five ellipsometry readings were taken after spin coating. PDMS film thicknesses were extracted from measured ellipsometry angles (Δ and Ψ) for the bare and coated substrates using an isotropic model.

UV-induced cross linking of PDMS was carried out in a UVOCS model T10X10. Thermal curing of PDMS was performed by resting the Si substrate on a hot plate at 170 ^{o}C for one hour.

Images of the surface were taken by atomic force microscopy (AFM) using a digital instruments Nanoscope IIIa in tapping mode with silicon nitride cantilevers. Rough estimation of particle size and distribution on the surface was attempted with the tip and surface alignment camera that has a magnification of 100 X.

Results: Thicknesses of PDMS films prepared using 1 and 10 wt % silicone elastomer plus curing agent in isooctane were determined. It was found that the longer the siloxane agent and cross linker were dissolved in isooctane the thicker the films became. Additionally, preparation conditions were found to influence the number of particles on the surface. Post processing such as annealing and UV light induced cross linking also affected the thickness and presence of particles.

Conclusions: A variety of different preparation conditions were used to make polydimethylsiloxane (PDMS) films on native silicon by spin coating a 10:1 mixture of silicone elastomer : curing agent in isooctane. Film thicknesses and pictures of the surfaces were measured with ellipsometry and AFM, respectively. Thicknesses of the films were on the order of tens of nm and increased depending on the length of time the silicone plus curing agent was allowed to react in isooctane. Increasing the concentration of silicone plus elastomer in solution by a factor of ten increased the PDMS film thickness by a factor of two. Surface particles were found using 1 wt% solutions but not 10 wt% solutions. Rinsing fresh untreated PDMS films caused part of the films to dissolve. UV and temperature curing caused the PDMS films to become resistant to rinsing with water, ethanol and isooctane.

In summary, a procedure was developed to produce 70-80 nm thick, uniform PDMS films free of particles that were resistant to rinsing with water, ethanol and isooctane. These surfaces should be useful for modeling contact lens materials in typical QCM-D experiments measuring protein adsorption.