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Process Optimization for Spray Coating of Poly (vinyl pyrrolidone)

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Poly(vinyl pyrrolidone) (PVP) is an important synthetic polymer which has a wide variety of applications in the biomedical field because of its film forming properties including adhesion, excellent physiological compatibility, low toxicity, and reasonable solubility in water and most organic solvents [1]. Recently some studies have been published [2, 3] using micromechanical sensors to characterize thin polymer coatings under various conditions. With the final aim to deposit thin PVP film on cantilevers we studied the process optimization of PVP by spray coating on microscope glass slides. Here, we present a study of the parameters determining the quality of the deposited film.

Spray Coating was done in an Exacta Coat Ultrasonic Spraying System (Sonotek, USA) .The main components are illustrated in fig. 1. The tip of the ultrasonic atomizer nozzle was actuated at a frequency of 120 kHz. Nitrogen gas was connected to the inlet of the air focusing shroud. The nitrogen pressure was monitored by a pressure sensor and regulated by a valve. The gas flow and the position of the air focusing shroud allowed the control of the diameter and shape of the spray-coating beam. The movement of the nozzle was controlled by an x-y-z stage. A shadow mask was put on a glass slide before deposition to cover some area from spraying. The masked areas acted as a baseline for characterizing the final coating by a surface profilometer (Veeco Dektak8) from where the thickness and roughness value were calculated as shown schematically in fig. 2. The surface texture was observed with an Optical Microscope (Zeiss).

A 0.5 wt. % solution of PVP in water was prepared and introduced in the central column of the nozzle using a syringe pump. Each slide was coated 10times with a flow rate of 0.1ml/min and nitrogen pressure of 0.03Bar which was kept constant for all the experiments. The parameters varied are speed of the moving nozzle while spraying (nozzle path shown schematically in fig.2), temperature of the substrate and distance between nozzle and substrate. Surface morphology of the films is governed mainly by the rate of drying of the spray on the substrate. The depositions can be broadly classified into a dry state, a wet state and an optimized condition in between. The profilometer scan in fig. 3 and the microscope images in fig.4 show the surface for a distance between the nozzle and the substrate of (a) 100mm, (b) 70mm and (c) 90mm respectively. The further the nozzle is away from the substrate the faster the deposited polymer film dries. Spraying with a distance of 100mm gives rise to the dry state (fig. 3a) with avg. roughness (R_a) 158nm. When the distance between nozzle and substrate decreases to 70mm i.e., at the wet state, R_a reduces to 22nm. The disadvantage of the wet condition is that as the polymer remains wet for a longer time it accumulates at the edge of the deposition to form peaks of few microns in height (fig.3b). The optimized condition (fig.3c) lies in between at a distance of 90mm where we get a compromise between the dry and the wet state where R_a is 76nm but there are no edge peaks as shown before. With an increase in temperature (fig. 5a, b and c) the deposition moves from the wet to dry state were roughness increases due to rapid drying of the sprayed drops. Same dry state is observed for coating with an aqueous solution at 60°C (fig.5c) and when a low boiling solvent like dichloromethane (fig. 5d) is used for deposition at room temperature. The speed of the spraying nozzle influences both the final thickness and roughness of the film. The roughness becomes significant when the nozzle is very fast and the amount of polymer sprayed is not enough to coalesce and form a continuous film.

This study shows the inter-correlation of different parameters for uniform film formation by spray coating. The findings will be used for coating of cantilevers and for studies of material characteristics of thin polymer films used for example drug delivery.

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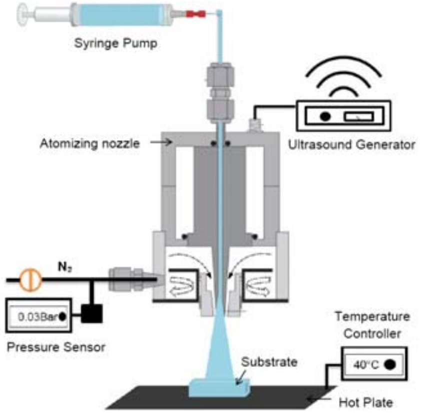

Figure 1. Setup for spray-coating of PVP

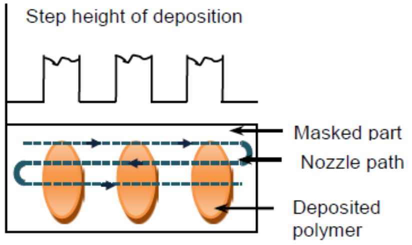

Figure 2. Schematic of shadow mask effect, profilometer scan and spray nozzle path

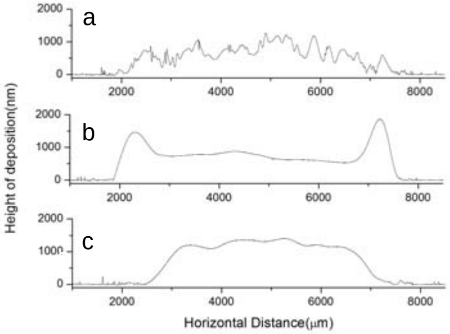

Figure 3. Profilometer scan of the deposition in (a) dry, nozzle- substrate distance 100mm (b) wet, nozzle- substrate distance 70mm and (c) optimized condition with nozzle- substrate distance 90mm

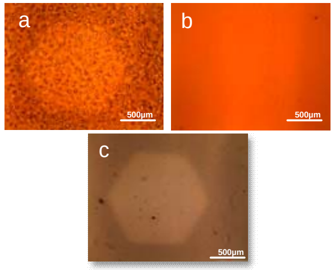

Figure 4.Optical microscope image of the deposition in (a) dry (b) wet and (c) optimized condition

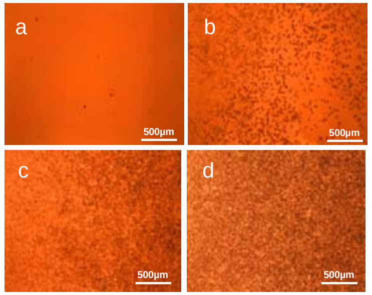

Figure 5.Optical microscope image show temperature effect at (a) 20°C (b) 40°C (c) 60°C and (d) deposition with dichloromethane as solvent