Outgassing Prevention from SLA Printed Materials via Atomic Layer Deposition of Aluminum Oxide

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Program: FY21 R&TD Innovative Spontaneous Concepts

Objectives

The purpose of this work was to evaluate what effect the application of aluminum oxide via atomic layer deposition (ALD-Al2O3) to a polymer surface would have on vacuum outgassing. This process has recently been shown to significantly decrease outgassing from polymers such as acrylonitrile butadiene styrene (ABS), a commonly used polymer in fuse deposition 3D printing. It should be noted that other methods to prevent outgassing do exist, such as coating with vacuum resins. However, when used in fluidic systems, resins can introduce chemical incompatibilities. Additionally, resins have limited application to fine geometries such as those used in microfluidics. However, because an ALD-Al_2O_3 coating is typically on the order of a few hundred nanometers, small features are left intact by the coating process. Additionally, ALD-Al_2O_3 has broad chemical compatibilities.

With this in mind, the objective of this work was to evaluate first whether such coatings would also prevent outgassing from polymers 3D printed via stereolithographic methods (SLA) such as Watershed XC 11122. The second objective of this work was to determine whether this coating would penetrate into complex geometries such as channels used in microfluidic systems.

Background

Additive manufacturing methods have the potential to revolutionize aerospace manufacturing. In particular, additive methods using stereolithography (SLA) enable production of solid-walled, high resolution features which currently is impossible with any other methods at reasonable cost. This is particularly true with regard to liquid chemistry and life detection missions where the manipulation of small volumes of fluid in microfluidic channels must occur. However, the materials used in these techniques pose challenges for flight due to their poor vacuum outgassing performance. This work used Somos® Watershed XC 11122 to produce two sets of geometries. Watershed XC 11122 was chosen because it is commonly used in fluidic systems. The first geometry was printed such that each block contained a 20 cm long serpentine channel with 800 µm diameter connected to inlet and outlet ports. These test pieces were designed to allow us to evaluate the penetration depth of the ALD process into long, narrow diameter, channels. The second geometry was designed to be small disc for outgassing measurement . Each set of geometries consisted of one control (no coating) and two ALD coated pieces, one with 100 nm coating and one with 200 nm thickness coating (figure 1).

Approach and Results

ALD Coating Process - Aluminum Oxide coating was done at MDL lab. Aluminum Oxide was deposited on the surface of the polymer pieces using atomic layer deposition via the reaction of trimethylaluminum $Al(CH_4)_3$ and water (H_2O). The selection of this method allowed the oxide layer to be grown at a temperature below the T_g of the polymer. The control samples were subjected to the same process but without reaction gas, thereby preventing formation of Al_2O_3 coating on them.

Results – Due to low T_g (40 °C) for this polymer, the outgassing test using ASTM E995 was done at 40 °C instead of standard 125 °C. In addition, the minimum recommended mass for sample is 250 g vs. 25 g mass for our samples which was not communicated to JPL until it was too late to make new samples. We believe the inclusivity of the outgassing measurement results is due to these two factors which can be ratified in the future test (table1). Thickness of coating measurement was accomplished by cutting across the channels (figure 2) and using SEM/X-ray spectroscopy. The results of this evaluation showed that while the coating had been deposited, the thickness of the coating was significantly different from expectations. The deposition of ALD-Al_2O_3 coating was non-uniform with a greater than expected thickness on the exterior of the test piece and thinner or no Al_2O_3 on the inner surfaces (figures 3 & 4).

We speculate that insufficient exchange of reaction gasses within the channels is the cause for inconsistency in ALD coating layers. This contributed to an overall poor evacuation of the gasses from the chamber and a poorly controlled Al_2O_3 growth overall. The future work should actively work to expel reaction gasses from within the channels via interfaces to tubing.

Significance/Benefits to JPL and NASA

ALD process using material such as Al_2O_3 can be performed at low temperatures (below the T_g of many polymers) and use to create coatings much thinner than the minimum feature size commonly used in fluidic designs. As a result, this technique will allow the flight use of polymers previously avoided due to their outgassing profiles.

Figures

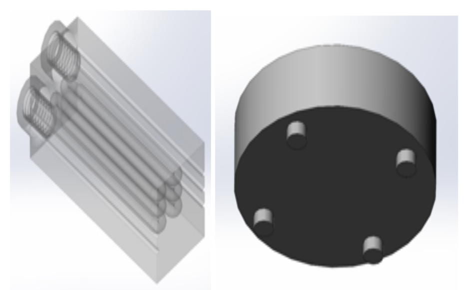

Figure 1. Both samples design for thickness and outgassing measurement.

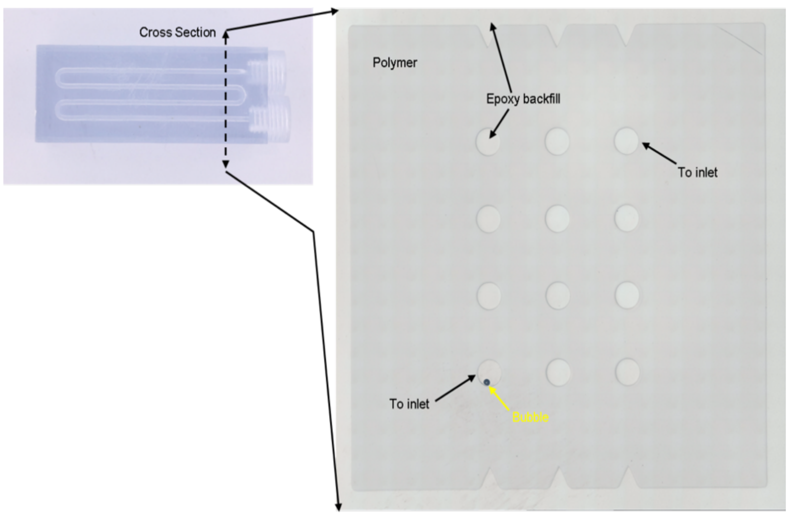

Figure 2. Cross section and view of the test block after the cut showing all 12 segments of the 20 cm channel prior to SEM/X-ray analysis.

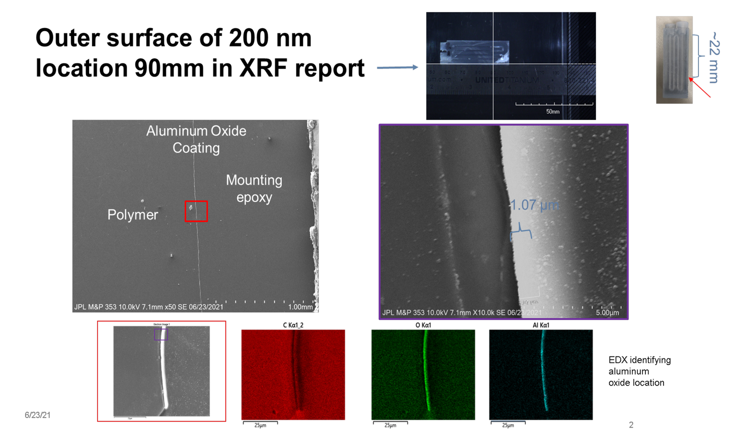

Figure 3. While X-ray spectroscopy did show a layer of Al_2O_3 on the outer surface of the polymer, the thickness was significantly greater than expected at 1.07µm as opposed to the target thickness of 200nm.

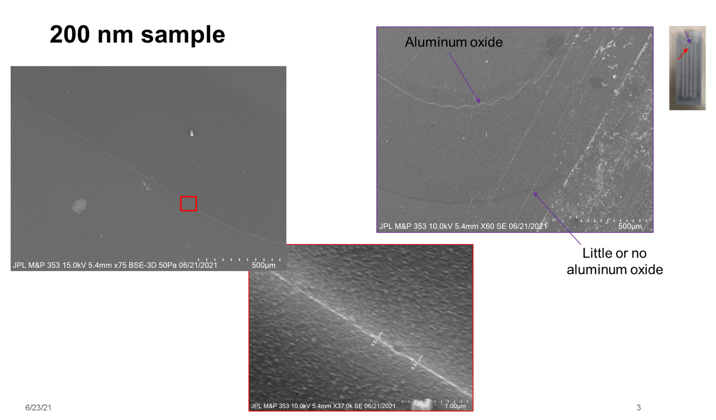

Figure 4. Within the channel it was evident that the 200nm coating was not deposited.

#	Sample ID	TML (%)	CVCM (%)	WVR (%)
1	Control	0.85	0.01	0.47
2	100 µm	0.84	0.01	0.48
3	200 µm	0.79	0.03	0.49

Table 1- Results of outgassing measurement- the results are inconclusive and inconsistent with measurement taken by Goddard on this material (3.25 %TML). This could well be due to the small mass of the samples.

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Clearance Number:
RPC/JPL Task Number: R21233