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A Quantitative Study on Key Parameters for the Deposition of Parylene C to Achieve 1–2 µm Coatings

by S. Gary Hirsch and J. Derek Demaree

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14. ABSTRACT
We demonstrate that by controlling three key parameters (i.e., sublimation temperature of the precursory dimer powder, sample temperature, and deposition time), thin layers of Parylene C can be deposited from 45 µm down to approximately 125 nm onto silicon wafer samples. We also show the effects of varying these parameters independently and in combination. Thickness measurements were carried out via mechanical profilometry and Rutherford backscattering spectrometry.

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1. Introduction

Cesium iodide (CsI) is a crystalline material with very high scintillation efficiency. In the presence of ionizing radiation—such as beta or alpha particles from radioactive sources—it converts this radiation into photons with efficiencies as high as 64,000 photons/MeV; thus, making it an important part of many radiation detection systems and radioisotope energy systems (i.e., so-called "nuclear batteries"). In particular, if an alpha-emitting isotope such as americium-241 is placed on top of the crystal, the alpha particles emerging from the radioisotope will penetrate the first 10–25 μm of the scintillator crystal, producing light that can be converted into beneficial power using an underlying photovoltaic cell.[1–3]

However, CsI is slightly hygroscopic, and its surface is susceptible to degradation from atmospheric moisture during service, limiting its efficiency to convert radiation into luminescence. Additionally, if the radioactive material is originally placed on the crystal in the form of an aqueous solution during device assembly, significant degradation can occur as the solution dries on the surface of the scintillator crystal. Protective coatings could be used to create moisture barriers on top of the CsI material; however, this overlayer cannot be so thick that it significantly blocks the incoming alpha particles. An ideal barrier coating for this application would have a thickness of no more than 1 or 2 μm, so that the incoming alpha particles lose only a small fraction of their energy before reaching the scintillator where they can then convert the remaining energy into light during the remainder of their penetration distance.

Parylene is an organic, polymeric material that is often used as an environmental barrier for electronic components and biomaterials. It provides a conformal, pinhole-free—hence, watertight—coating that can be deposited in thicknesses from a few nanometers to hundreds of microns. In this work we investigate changing several key deposition parameters to achieve a Parylene coating of approximately 1–2 μm. To achieve this thickness, we leverage results from previous experiments[1] where a fully functional deposition system was previously used to fill small cracks made from micro indentations in bulk silicon carbide (SiC).

2. Apparatus

The main apparatus used was the same as described in ARL-MR-1005.[4] Figure 1 from that memorandum report is shown below (also Figure 1). In this technical note, however, improvements were made to make the system more stable and obtain consistent results. More heating tapes and temperature sensors were added on some surfaces to more accurately read and control temperatures throughout the

system. These included two thermocouples added to the source to supplement the sensor already integrated with the heating tape (Figure 2). A heating tape and three thermocouples were also added to the conduit between the ultra-high vacuum (UHV) valve and the furnace. This ensured that the temperature of gas flowing through did not cool below the source temperature, inadvertently causing adsorption to the inner walls. Another heating tape was added to the face of the door to ensure that all inside surfaces of the deposition chamber could be kept at the same temperature. A final heating tape was added between the out-flow valve of the chamber and the liquid nitrogen (LN) trap. This was applied to ensure that the valve would not adsorb material, causing the pressure to be unstable during deposition as well as more difficult over many cycles.

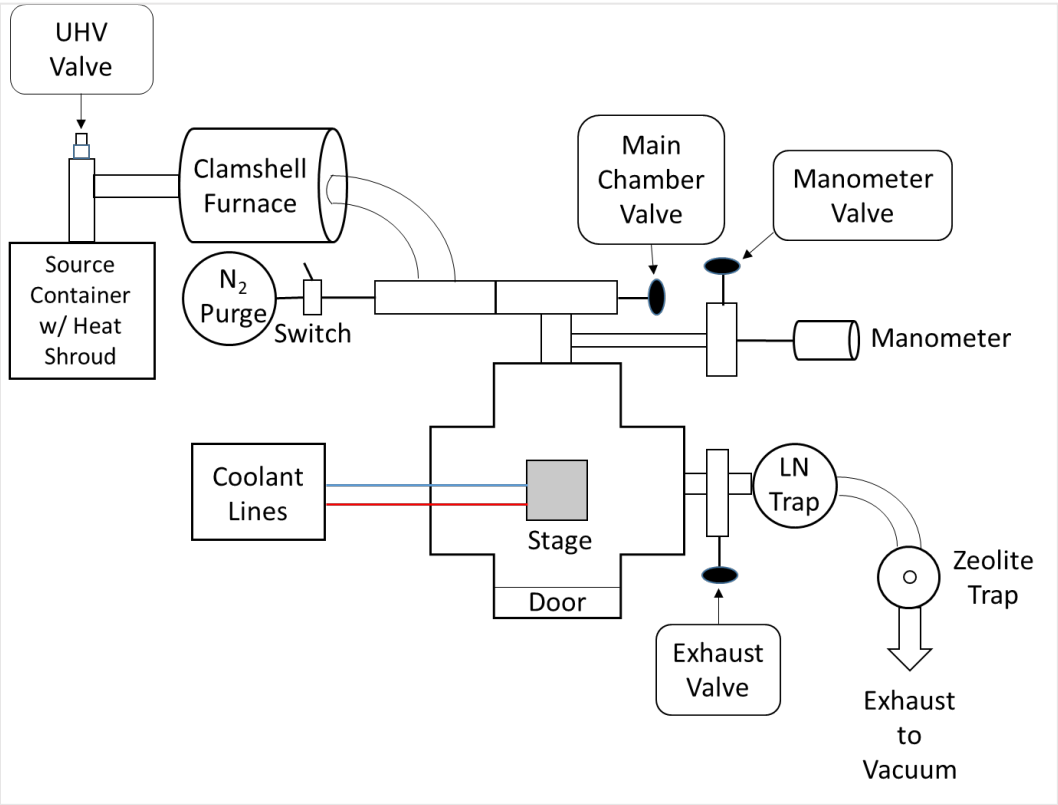

Figure 1. Schematic of deposition chamber.

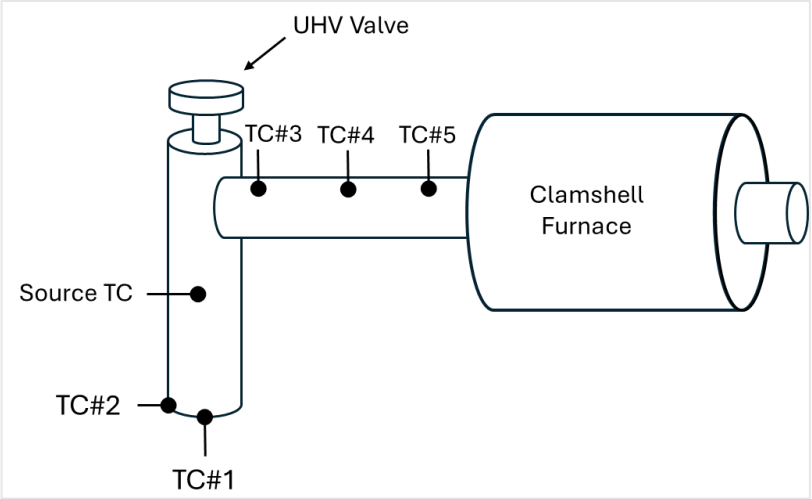

Figure 2. Detailed schematic of upgraded thermal monitoring on source side. (Note: TC = thermocouple).

3. Procedure

Square, silicon wafer samples (approximately 15 mm on a side), were mounted onto the copper stage with silver paint (Pelco no.: 16040). The system was then sealed and pumped down to a base pressure of 50 mT or less before deposition. During the pump-down the heating tapes were turned on to the settings needed to reach desired temperatures. It took approximately 3–4 h for all components of the system to reach their desired temperatures and stabilize. For each run the furnace temperature was set at 650 °C. The temperature of the conduit between the source and furnace was controlled such that the temperature from the source to the furnace was always above the source temperature and increasing into the furnace. The conduit from the furnace to the deposition chamber, the deposition chamber itself, and the exhaust valve leading to the LN trap were heated and maintained at a temperature above 90 °C to keep the adsorption coefficient low.[5–8] Only the water recirculating bath was maintained below the adsorption temperature.

The coating process starts with taking Parylene C dimer, in granular form, and heated to the point of sublimation. The sublimate is then thermally pulled through the furnace where it is "cracked" to form two monomers, which will then self-polymerize as they adsorb onto cooler surfaces.[5–8] The key to making a good pinhole-free coating is using low vapor concentration with a large mean free path. This is achieved by using a low vacuum environment, controlling the ambient temperature of the vapor phase, and controlling the sample temperature, which determines the rate of deposition.[5–8]

4. Results

Four successful depositions were conducted leading to thinner coatings with each subsequent run. Table 1 displays the key parameters used for each trial run and the resulting thicknesses. Initially, a very high temperature—well over the sublimation threshold—was used, which is typical for thicker depositions in the range of tens of microns. With a relatively short deposition time of 15 min the resulting thickness was approximately 45 mm. Next, a significant change of the temperature of the stage was made by increasing the water temperature from 40 to 60 °C. This effectively lowered the adsorption coefficient. The resulting thickness was approximately 15 mm.

For the next deposition run, we decreased the sublimation temperature to 125 °C and increased deposition time slightly to 20 min. In this run, however, two samples were used. One resultant thickness was measured at 4.0 mm and the other at 2.5 mm. This significant difference in thickness between the two was attributed to the design of the system where the entrance to the chamber is 90° to the exhaust. The flow would then be expected to be more concentrated toward the inside of the angle. Hence, a sample closer to this flow will grow thicker than one farther away. Ideally, a better design would have been one where the entrance and exhaust were 180° apart. This would create a more even and laminar flow.

The final run used only one sample affixed to the center of the stage. Three significant changes were made to the deposition parameters. We decreased the sublimation temperature slightly from 125 to 115 °C. This may seem small, but given the nonlinear sublimation rate,[5–8] 10 °C is more significant than it may seem. More critically, we increased the sample temperature by increasing the water temperature to 75 °C; thereby decreasing the adsorption rate significantly. The deposition time was also increased by about a factor of two. The measured thickness for the run was approximately 125–150 nm. The decreased accuracy of this measurement was due to the resolution limit of the mechanical profilometer used.

Table 1. Key parameters and values for each deposition run.

Run	1	2	3	4
Source temp (°C)	165	170	125	115
Water temp (°C)	40	60	60	75
Base pressure (mT)	38.5	38.5	19.7	35.6
Deposition pressure (mT)	+20	+32	+19	+40
Deposition time (min)	15	15	20	45
Thickness (mm)	45	15	2.5/4.0	0.125–0.150

With the 2.5-μm sample coating closest to the desired thickness, Rutherford backscattering spectrometry (RBS) was used as a check on the profilometer measurement. Using a National Electrostatics 2SDH-2 positive ion accelerator, a beam of 2.0-MeV helium ions was directed onto coated and uncoated silicon substrates, and the energy spectrum of the scattered helium particles was measured using a surface barrier detector at a scattering angle of 175°. The thickness of the deposited film was estimated using SIMNRA simulation software,* and—assuming the density of the deposited film was near the nominal value of 1.3 g/cm^3—the deposited film varied from 2.2 to 2.6 μm over the area measured.

5. Conclusions

The source sublimation temperature (i.e., dimer, sample temperature, and deposition time) were the key parameters that affected the film thicknesses. Together, these determine the concentration of monomer available for deposition, the adsorption coefficient of the monomer onto the sample, and how long the sample is exposed to the monomer, respectively. Time is the only linear parameter regarding the rate of deposition. By combining all three parameters, a wide distribution of film thicknesses was achieved from 150 nm to 45 mm—with 2.5 mm being close to the desired final thickness. A wider distribution of thicknesses could easily be obtained by using all three parameters—separately or together. Additionally, Parylene coatings can be made slightly thinner postdeposition by annealing.[9] However, this simply makes the resulting film denser. And while this should not change the ability of the radiation to penetrate through, it will make the film more resilient to water vapor infiltration.

* https://mam.home.ipp.mpg.de/

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List of Symbols, Abbreviations, and Acronyms

CsI	cesium iodide
LN	liquid nitrogen
RBS	Rutherford backscattering spectrometry
SiC	silicon carbide
TC	thermocouple
UHV	ultra-high vacuum

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