APPLICATION NOTE SPECTROSCOPY APPLICATION FOR PARYLENE COATING



METHOD INTRODUCTION TO PARYLENE COATING

Poly (para-xylylene), more commonly known as parylene, is a highly versatile family of polymers used in many conformal coating applications. At present, there are a wide range of parylene variants available on the market – types N, C, D, F(VT-4), and F(AF-4) – each with their own unique thermal, dielectric, and mechanical properties. Despite all these variations, parylene is an extremely simple polymer. Parylene N is the natural (or neutral) form of poly (para-xylylene) is comprised of alternating aromatic rings and methylene groups forming a highly stable polymeric backbone (see figure 1).



FIGURE 1: Schematic representation of a typical polymerization and deposition process for perylene N conformal coatings. Calculated thickness determined using an FFT-based algorithm.

The other parylene variants can be broadly grouped into two main categories – chlorinated or fluorinated. For the chlorinated variants, one (type C) or two (type D) of the hydrogen atoms around the aromatic ring are replaced by chlorine, increasing the maximum operating temperature but decreasing the dielectric strength. Fluorinated parylene, on the other hand, requires the substitution of four hydrogen atoms with fluorine. The two fluorinated variants are differentiated by whether the aromatic rings, F(VT-4), or the methylene groups, F(AF-4), have been fluorinated. It is important to note that Parylene F(AF-4) is often referred to as parylene HT due to its extremely high operating temperature (~350oC) and is the most used fluorinated variant.

STUDY

PARYLENE CONFORMAL COATINGS

The high dielectric strength, low coefficient of friction, biocompatibility, and high operating temperature of parylene all make it attractive for use in the electronics industry. What truly sets parylene apart from other conformal coating technologies is its gas phase polymerization/deposition process. Traditional conformal coatings are applied in the liquid phase by either brushing, dipping, or spraying the components. While suitable for low-end applications liquid phase coatings are always susceptible to variations in coating thickness and localized air pockets resulting in coating defects (figure 2).



Conventional Liquid Coating

Parylene coatings use a three-step vapor deposition polymerization (VDP) process similar (see figure 1) to a traditional chemical vapor deposition (CVD) process. The first step is to vaporize powered di (paraxylylene) at 1500C. Once in the gas phase, the dimer flows into a second tank where it is heated up to the point of pyrolyzation when the dimer splits, forming induvial monomers. The resultant mono (paraxylylene) gas then flows into a third tank where the component of interest is held at 25oC, filling every crack and crevice as the gas adheres to the surface, forming a thin epitaxial polymer coating one molecule at a time. It is essential to point out that since the coating process takes place at room temperature without any additional curing, there are virtually no added stresses and strains on the substrate. Furthermore, since the polymerization takes place without any catalysts or solvents, it is generally viewed as a "green" process.

Gas-phase epitaxial growth produces exceptionally uniform coatings with excellent crevasse penetration, with processing time being the only limiting factor to the coating thickness. For example, the typical coating rate of parylene C is around 3-5 microns per hour, with standard coating thicknesses ranging from as thin as 500 angstroms to over 75 microns. Therefore, parylene thickness is one of the most critical factors that need to be measured to ensure the quality of the coating process. While a wide range of different high-end methodologies can be used to access coating thickness, including scanning electron microscopy (SEM) and white light confocal microscopy, reflectance spectroscopy offers the best balance between size, cost and complexity for widespread quality control applications.

FIGURE 2: Comparison of liquid and parylene conformal coatings

MEASURING COATING THICKNESS

In a previous application note titled "Spectroscopy in Thin Film Fabrication," we explained the effects of film thickness on spectral reflection in detail, so we will only briefly review it in this application note. Some of the light will be reflected and transmitted whenever light passes from one material to another. Therefore, it is perfectly valid to think of a thin film coating as a simple Fabry-Perot resonator when the light reflects off the top and bottom layers constructively or destructively interfere depending on their relative phase difference. In this case, the phase difference is solely determined by the optical path length of the material, which in this case will be equal to the index of refraction of the coating times twice the coating thickness. Therefore, if you know the index of the material, it should be straightforward to determine its thickness. The only caveat is that since the index of refraction is a wavelength-dependent complex function refraction, $n^* (\lambda)=n(\lambda)+ik(\lambda)$, where n is the index of refraction, and k is the absorption coefficient. Fortunately, Avantes has already developed an extensive library of n and k values for most common coating materials, including parylene.



FIGURE 3: Reflectance spectra of a 7-micron parylene C coating, along with the calculated thickness determined using an FFT-based algorithm.

Figure 3 shows a test coupon's reflectance spectra with a 7-micron parylene C coating. Based on the frequency chirp in the reflectance spectrum, the third-party thin film software (created by Boulder Optical Design) was able to determine the exact thickness to be 6.81 +/- 0.08 microns.

This data was collected using the newly released <u>Ava-Reflectometer</u>, currently only available from Avantes USA (see figure 4). This system incorporates Avantes' Avalight-HAL-S-Mini2 halogen light source, a miniature reflection probe, and the AvaSpec-Mini-2048CL into one compact, user-friendly thin film measurement device. The Ava-Reflectometer is a compact lightweight (2.27 Kg) thin-film spectrophotometer with a maximum spectral range from 200 nm to 1100 nm. The Ava-Reflectometer allows for easy measurement of transparent or opaque substrates coated with thin films.

EQUIPMENT OPTIONS

For users interested in a more modular approach, Avantes offers thin-film measure bundles including a fiber-coupled spectrometer such as the <u>AvaSpec-Mini2048CL</u> (figure 5) or <u>AvaSpec-ULS2048CL-EVO</u> (figure 6). A fiber-coupled constant current tungsten or deuterium halogen light sources such as the <u>Avalight-HAL-S-Minis</u> (figure 7) or <u>AvaLight-DHc</u> (figure 8), and fiber optic reflection probe such as the <u>FCR-7UV200-2-ME</u> (figure 9). Avantes also provides the AvaSoft Thin Film software add-on offering two different methods for thin-film calculations – Fast Fourier Transform (FFT) and best-fit optimization algorithms. The FFT method determines the frequency of the interference pattern and using the n and k values can calculate the corresponding thickness. This algorithm is ideal for thicker coatings with relatively high-frequency chirps. On the other hand, spectral matching predicts the best fit based on a series of adjustable fitting parameters. Spectral matching is ideal for thinner coatings with relatively low-frequency spectral chirps and in situations where high-speed monitoring is advantageous.



FINAL THOUGHTS

While parylene coatings were the focus of this application note, it is important to remember that spectral reflection can be used for measuring the thickness of any optically transparent coating. Avantes has long offered a wide range of fiber-coupled miniature spectrometers, light sources, and probes. Combined with their AvaSoft Thin Film software module, it facilitates measurements of single-layer thin films ranging from 10 nm to 100 μ m in thickness with a 1 nm resolution. Now with the addition of the new Ava-Reflectometer, Avantes can now support thin film thickness measurements at any level of the quality control process ranging from offline test coupon analysis to fully automated inline testing. In addition, Avantes' Avaspec instruments are ideally suited to high-speed triggered or continuous measurements such as those required in this application.

Additionally, all of the spectrometers discussed above are also available as OEM modules and can be integrated into multichannel rack mount systems, ideally suited to thin-film process monitoring systems. These units can communicate via USB, Ethernet, and the native digital & analog input/output capabilities of the Avantes AS7010 electronics board provides for a superior interface with other devices. Additionally, the Avantes AvaSpec DLL software development package, with samples programs in Delphi, Visual Basic, C#, C++, LabView, MatLab, and several other programming environments, enable users to develop code for thin-film applications. This software development kit is particularly useful when an instrument will be integrated into an automated sampling system or for analysis of complex multi-layer thin films requiring a customized code.

For more information about the full range of laboratory and OEM spectrometer options available from Avantes, including our light sources, probes, and sample accessories, please feel free to visit the website at www.avantesusa.com or give us a call at +1 (303)-410-8668 where our knowledgeable applications specialists are standing by to help.





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Curious how spectroscopy can help you reveal answers by measuring all kind of materials, in-line, at your production facility, in a lab or even in the field? Please visit our website or contact one of our technical experts, we're happy to help you.

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