VeeMAX III: The Next Generation

By Jenni Briggs, PhD

The research-grade VeeMAX is a staple variable-angle specular reflection/ATR accessory. With its innovative optical design, this accessory has found use across a myriad of fields due to its high performance, ease of use, and ability to maintain a purged environment during analysis and between samples. Some specific applications include depth profiling, electrochemistry, and analysis of monolayers and ultra-thin films, polymer orientation and Brewster's angle.

Through the redesign, the VeeMAX III has retained its core identity, while receiving an overall facelift. The optical design has been maintained (U.S. Patent No. 5,106,196) offering the wide range of incidence angles and flexibility that users have come to expect. In addition, the VeeMAX III continues to use face-angled ATR crystals to maximize throughput allowing for the collection of the highest quality spectra.

Figure 1. VeeMAX III with ATR

From the scientists' viewpoint, performance is the most important criteria when selecting an IR accessory. At a 45° angle of incidence, the new VeeMAX III proudly exhibits a 20% or more increase in specular reflectance energy throughput compared to the VeeMAX II. The enhanced performance is directly realized through PIKE Technologies' manufacturing processes and base materials. To begin with, all parabolic and elliptical mirrors are made from a high-quality aluminum stock, which features a fine recrystallized, uniform grain structure. This allows the mirrors to be cut and polished using single-point diamond turning technology in our manufacturing facilities creating a smoother finish for maximum reflectivity. Surface finishes of 5 nanometers RMS and form to $\lambda/4$ may be obtained using the Ultra-Precision Lathe.

To illustrate the versatility of the VeeMAX III, a couple of applications are presented. All data were collected using an FTIR equipped with a DTGS detector. Data collection time was 1 minute.

Depth Profiling Via ATR

The hallmark feature of the VeeMAX III—variable angle of incidence—makes this accessory ideal for depth profiling. As the angle of incidence nears the critical angle, defined as the inverse sine of the refractive index ratio of the sample to the ATR crystal, the depth of penetration (d_p) increases exponentially. Thus, a wide range of probing depths may be acquired by using a combination of crystal types with different refractive indices and sampling at varying angles of incidence. Penetration depths from a fraction of a micron to over 15 microns may be obtained.

A film used for electronic packaging material was measured using the VeeMAX III with a ZnSe 45° ATR crystal to illustrate depth profiling. From the IR spectrum the film was identified as poly(ethylene terephthalate). Angles of incidence ranged from 43° to 57° resulting in depths of penetration of 2.4 to 1.2 µ, respectively (Figure 1). As the probing depth increases, differences in the absorbance bands throughout the fingerprint region are observed.

The strong absorbance band near 725 cm^{-1}, which may be associated with the out-of-plane deformation of the two carbonyl substitutions found on the aromatic ring[1], is measured as a singlet at d_p = 1.2 µ. As the d_p increases by reducing the angle of incidence, additional absorbance bands materialize between 725 – 730 cm^{-1}.

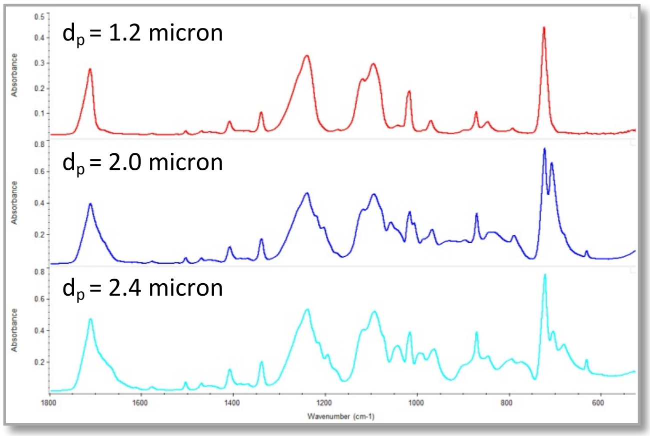

Figure 2. ATR spectra of an electronic packaging film collected at various angles of incidence.

Monolayers and Ultra-Thin Films

Two of the most popular IR sampling techniques to investigate the chemistry of monolayers and ultra-thin films are specular reflectance and ATR. Both may be performed using the VeeMAX III. The angle of incidence for specular reflectance is often 80°, and an MCT detector is recommended. For ATR measurements, a germanium crystal is required, and to obtain the maximum energy the angle of incidence is 59° and 70° for a silicon and gold substrate, respectively. In this configuration, it is possible to collect quality spectra with a DTGS detector. Regardless of the sampling technique, a polarizer set for p-polarization is necessary.

In the case of self-assembled monolayers (SAMs), the objective is to introduce a chemically-defined thin film providing specific chemical and physical properties. Amine-terminated SAMs are of interest because the terminal amine groups may be modified to immobilize biomolecules via a peptide or a phosphoramidite linkage. The development of biosensors is a major driver of these types of research.

Hydrogenated amorphous silicon (a-Si:H) thin film research, another application for the VeeMAX III, is propelled by the development of solar cells, thin film transistors for imaging technologies, and light sensors in imaging scanners. Collected via ATR, a spectrum of a 100 nm thick film of a-Si:H deposited on an n-type silicon wafer is shown in Figure 3. The Si-H stretch absorbance band near 2080 cm^{-1} is one band often used to assess film quality. Measuring the IR spectra of these ultra-thin films may also be used to optimize film processing parameters.

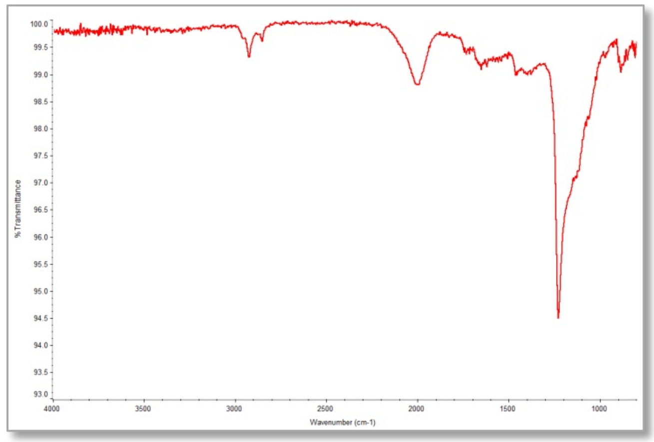

Figure 3. ATR spectrum of hydrogenated amorphous silicon thin film.

The VeeMAX III is designed to be versatile, meeting the demands of an active research lab. Several add-on options are available including automation for precise and reproducible angle settings. The ATR configuration may be fitted with a flow-through attachment, liquids retainer, and heated ATR plates. Please contact us with your specific application needs.

References

1. Holland, B.J., and J.N. Hay. Polymer 43(2002) 1835 – 1847.