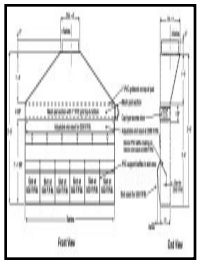

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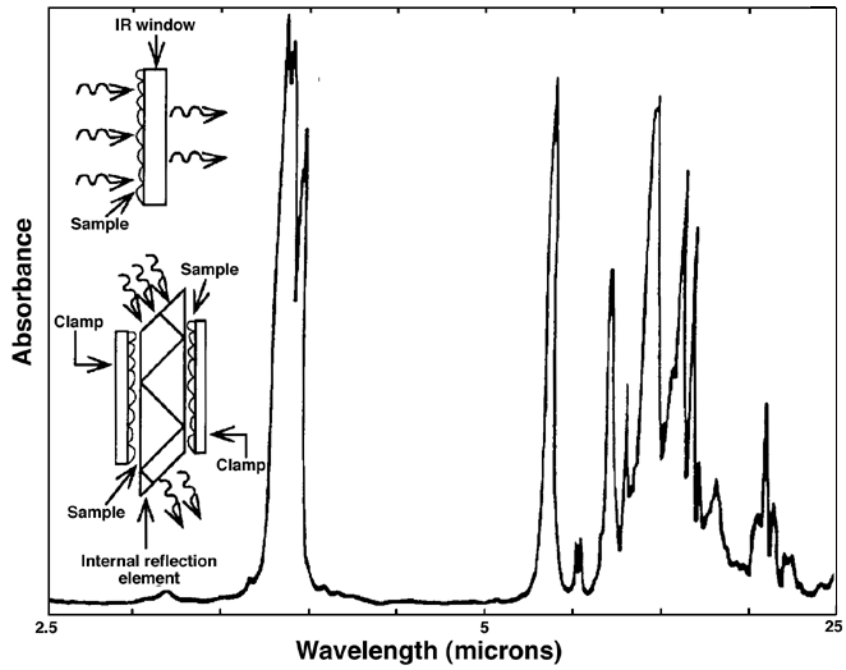
PVD Processes: Infrared Spectroscopy

A polymer is a large molecule formed by bonding together numerous small molecular units, known as monomers. The most common polymeric materials are the organic polymers that are based on carbon-hydrogen (hydrocarbon) monomers, which may or may not contain other atoms, such as nitrogen, oxygen, metals, etc. Polymers can also be formed from other monomer units, such as silicon-hydrogen, boron-hydrogen, etc. In building a polymer, many bonds are formed that have various strengths and separations (bond lengths) between atoms.

Infrared Spectroscopy

Infrared (IR) spectroscopy uses the adsorption of infrared radiation by the molecular bonds to identify the bond types that can absorb energy by vibrating and rotating. The adsorption spectrum is generated by having an infrared spectrum pass through the sample, and then comparing the emerging spectrum to that of a reference beam that has not passed through the sample. In Fourier transform infrared (FT-IR) spectroscopy, the need for a mechanical slit is eliminated by frequency-modulating one beam and using interferometry to choose the IR band. This technique gives higher frequency resolution and a faster analysis time than the dispersive method.

When using a spectrum of adsorption vs. infrared frequency, the type of material can often be identified. If the material cannot be identified directly, then the types of individual bonds can be identified, giving a good indication of the type of polymer material. It can also be used to characterize polymer substrate materials concerning their primary composition, as well as such polymer additives as plasticizers, anti-slip agents, etc. The IR spectra of many materials are cataloged, and a computer search is often used to identify the material.



Infrared spectrum of a phythale plasticizer extracted from a vinyl material.

Sample Collection

Sample collection is an important aspect of IR analysis. Bulk materials can be analyzed, but if they are thick, the sensitivity of the technique suffers. The sample is often prepared as a thin film on the surface of an IR transparent material (window), such as potassium bromide (KBr). The film can be formed by vapor condensation on the surface, dissolving the sample in a solvent, or by solvent extraction from a bulk material, followed by evaporation of the solution on an IR window. The accompanying figure shows an IR spectrum of a phythale plasticizer, extracted from a vinyl material by extraction using acetone. This type of plasticizer is often used in polymers to make them easier to mold, and is a source of out-gassing, out-diffusion and extraction of low-molecular-weight materials by solvents.

Reflection techniques can often be used to analyze surface layers without using solvent extraction. A reflection technique is shown in the figure, where the sample is sandwiched between plates of a material having a high index of refraction in the IR, so as to have a high reflectivity from the surface.

In PVD technology, IR spectroscopy is used in a comparative manner to ensure that the substrate material is consistent. Quite often it is found that

a specific polymer material from one supplier will differ from that of another, in that the amount of low-molecular-weight constituents can differ. This can affect the out-gassing and out-diffusion of material from the bulk during processing.

The low-molecular-weight materials can originate from an additive material or from differing curing of the monomer materials. A procedure to characterize a polymeric material might consist of:

- A "swipe" or solvent clean of the surface of the as-received material, to determine if there is a surface layer of low-molecular-weight species;
- Solvent extraction from the bulk material using a given sample area, solvent, solvent concentration, temperature and time;
- Vacuum heating for a specific time at a specific temperature, followed by solvent extraction to ascertain out-diffusion and surface contamination by low-molecular-weight species;
- Vacuum heating for a specific time and temperature with a cool IR window in front of the surface, to collect volatile species resulting from out-gassing of the bulk material.

These spectra would then form a baseline with which to compare the received material. These same procedures could be used to characterize the polymer surface after surface-preparation processing, such as an oxygen plasma treatment or the application of a basecoat.

In PVD processing, IR spectroscopy can be used to identify such common contaminants as hydrocarbon, silicone and fluorinated pump oils, hand creams, adsorbed hydrocarbons, etc.

System- and process-related contamination can be studied by IR spectroscopy techniques. An IR window, for example, can be placed in front of the roughing port of a deposition system during cycling, and IR analysis will show if there is any backstreaming of the roughing pump oils.

The same can be done in front of the high-vacuum port to detect backstreaming from the high-vacuum pumping system. During processing, a window can be placed out of the line-of-sight of the vaporization source to detect volatile/condensable species that may not be detectable using a residual gas analyzer. *P&SF*

Reference

C. Marcott, "Materials Characterization: Infrared Spectroscopy," *ASM Metals Handbook*, Vol. 10, ninth ed., p. 109, American Society for Metals (1986).

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