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FTIR Spectroscopy in the Materials Laboratory

Background

Everyone is familiar with various kinds of electromagnetic radiation. The spectrum of visible light has been long observed in nature in the form of the rainbow. In 1666 Sir Isaac Newton demonstrated that radiation from the sun could be split into the component colors of the rainbow using a glass prism. However, the visible region is only a small portion of the total electromagnetic spectrum. Other detection systems reveal radiation beyond the visible regions of the spectrum, which are classified as gamma rays, x-rays, ultraviolet rays, infrared, microwave, and radio waves.

The radiation from these different regions of the spectrum interact with matter in many different ways which can be summarized as follows:

(See column 2 top)

Gamma Rays - Atomic Nuclei Excited
X-Rays - Inner electron transitions
Ultraviolet - Outer electron transitions
Infrared - Molecular vibrations
Microwave - Molecular rotations
Radio wave - Nuclear Magnetic resonances

This article will focus on the interactions of infrared radiation with matter and the applications this finds in the material testing laboratory.

As noted above, infrared radiation interacts with matter to cause molecular vibrations. To get some kind of a mental image of this it is helpful to think of atoms as round masses and the chemical bonds which connect atoms as springs. When infrared radiation interacts with this system of springs and masses the springs are set in vibrational motion at specific frequencies depending on the "stiffness" of the spring. Infrared radiation passing through a sample will therefore have certain wavelengths absorbed depending on the particular vibrational modes of the sample. An example of this is shown below.

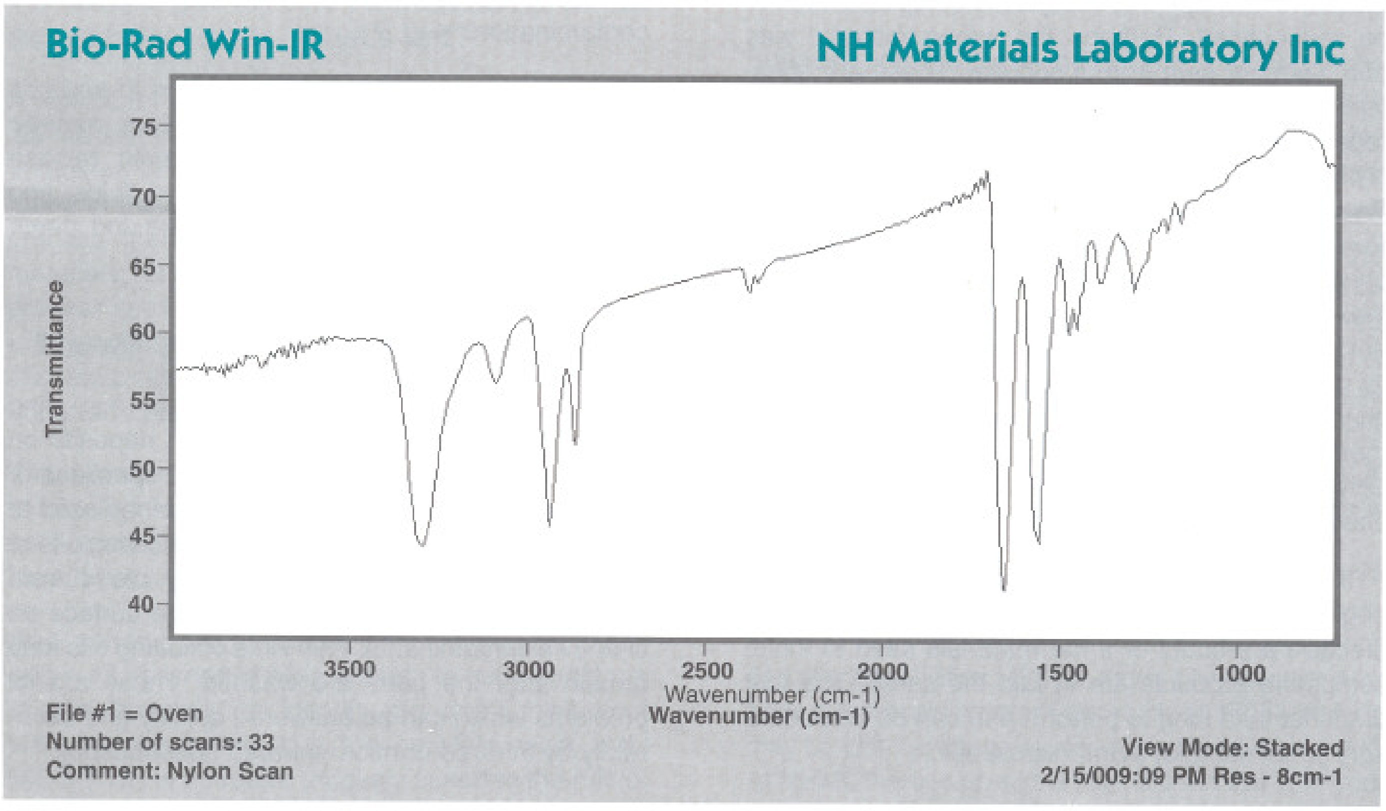

FIGURE 1: Infared spectrum of nylon - the nylon "fingerprint"

The sample graph shows an infrared spectrum of Nylon. The series of "peaks and valleys" is characteristic of the chemical bonds in the Nylon polymer, it can be thought of as the *"nylon fingerprint"*.

Because the infrared spectrum is characteristic of the chemical bonding in the sample, *different chemical compounds will each have their own characteristic absorption spectrum. It is for this reason that Infrared Spectroscopy is such a powerful and useful technique in the identification and comparison of various materials.*

If each material has a unique infrared "fingerprint" then these can be used to identify unknown materials and to determine if two materials are in fact the same. Because of this, infrared spectroscopy finds a wide variety of applications in the materials testing lab. Here are a few questions and how Infrared Spectroscopy can answer them.

A significant portion of our work here at the materials lab involves working with quality managers and engineers to characterize and identify contaminants found in their final product.

"I ordered Polypropylene but it doesn't act like Polypropylene, what is it?"

One of the more common requests is for a simple material identification or verification, the "what is it" question. Perhaps two lots get mixed up and need to be re-identified. Perhaps the wrong material was purchased or sent from a supplier. These are questions that can be readily answered by analysis of a representative sample by FTIR spectroscopy. Nylons, Polyethylene, Polypropylene, Polystyrene, Polyurethanes, Epoxies, Acrylics, Polyesters, Polyamides and Acetyls, to name a few, all have characteristic infrared absorption spectra due to their unique chemical bonding. They all have different infrared "fingerprints" and can therefore be readily distinguished by their infrared spectra! If further characterization of a polymer sample is needed then thermal analysis can also be performed including Differential Scanning Calorimetry (DSC) and Thermo Gravimetric Analysis (TGA).

FTIR spectroscopy is particularly useful for reverse engineering to determine the materials used to manufacture a product or if the materials used to make competing products are in fact the same. The test does not take long to perform and can be done on a very small sample, even microscopic.

My plating didn't take, what's wrong?

Surface preparation is necessary prior to the application of plating or other type of coating. The degree of surface treatment may vary from a simple solvent wipe to a multistage chemical treatment and cleaning requiring substantially more time.

Some processes used for surface cleaning include vapor degreasing, solvent cleaning, acid cleaning, emulsion cleaning, alkaline cleaning, salt bath descaling, and abrasive blast cleaning. The selection of the most appropriate method of surface preparation must be based on the contaminants to be removed as well as on the nature of the material to be cleaned. The purpose of these treatments is to remove any surface contamination which would interfere with proper bonding of the plating or coating to the surface.

If a layer of grease or oil or possibly a film of mold release agent remains after cleaning it will disrupt bonding to the surface. Since these surface cleaning procedures are not 100% effective all of the time there is the possibility of some residual surface contamination.

Infrared spectroscopy is uniquely suited to analysis of thin films on surfaces, especially shiny metallic surfaces. Very small quantities of mold release agents, lubricating oils, greases, cutting fluids, and polishing compounds can all be detected and identified by FTIR.

What's this oily stuff on my relay leaves?

Manufacturers of electronics products may find unknown contaminants, films or other deposits on their product either during routine QC inspections or from parts returned from the field. Machines used to assemble parts may be a source of lubricating oils or other contaminants. Plasticizers may bleed from polymeric materials and end up on the surface as unknown contaminants. Thin films of cutting oils may remain after the parts are washed. These are all problems which can be answered quickly and accurately by infrared or micro-infrared spectroscopy.

My competitors have a superior adhesive, what is it?

In a competitive market there is the reality of continuous improvement, therefore the competition is always looking to find a superior material to enhance the performance of their product. A wide variety of adhesives are available to choose from: Epoxies, Phenolics, Urethanes, Cyanoacrylates, and Silicones to name a few.

Infrared spectroscopy is the method of choice for identification of Adhesives. Some adhesives can be filled with oxide fillers or metallic flakes to enhance thermal or electrical conductivity. This is an example of a problem that requires the use of the different, but complimentary, techniques of FTIR spectroscopy and Energy Dispersive X-Ray Spectroscopy. FTIR is used to identify the type of adhesive and Energy Dispersive X-Ray Spectroscopy (EDS) is used to identify the metallic or inorganic filler. Such an approach is most often taken when analyzing unknown contaminants because they are almost never pure isolated chemical compounds, but are rather mixtures (often complex mixtures!) from several contributing sources.

We didn't get all the contaminants off of our medical parts. What's left?

A significant portion of our work here at the materials lab involves working with quality managers and engineers to characterize and identify contaminants found in their final product. With any manufacturing process there is always the potential for foreign materials, or materials used in the manufacturing process itself, to end up as contaminants in the final product. Some of these contaminants are easily visible while others are not apparent unless observed under an optical microscope during QC inspections.

With advances in FTIR microscopy, now it is possible to obtain infrared spectra of micron sized particles and objects (that's pretty small!), and therefore to identify micro-contaminants by their infrared spectra.

As a general rule of thumb, if it's big enough to see with a microscope, it's big enough to get a spectrum of and hopefully to identify. So don't think your unknown contaminant is too small to work with! Micro-FTIR is therefore uniquely suited to the microelectronics industry as well, where the progressive miniaturization of integrated components on silicon wafers necessitates the capability of analyzing micro-contaminants.

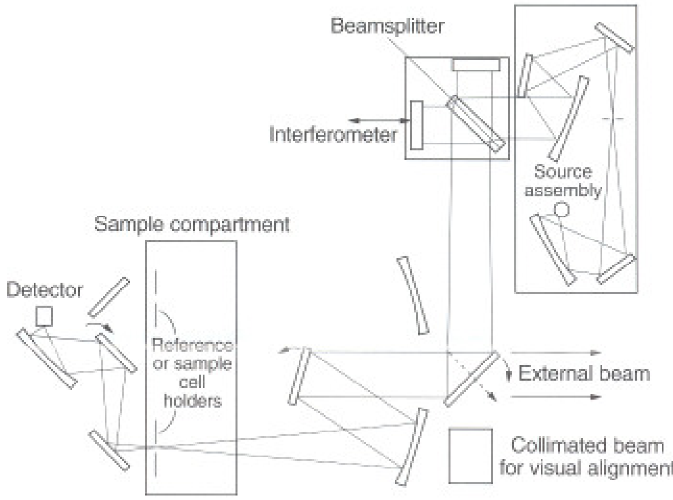

FIGURE 2: Optical diagram of an FTIR spectrometer

Providing a more complete picture

In recent years FTIR has been "coupled" to other methods of analysis to provide a more complete picture of a material's characteristics. One of these is the coupling of FTIR to a Thermo Gravimetric Analyzer (TGA).

Suppose you are the manufacturer of roofing materials and you need to know not only at what temperature the material starts out gassing, but also what gases are being given off over certain temperature ranges. These might be important questions regarding the materials flammability or potential to contribute flammable gasses to a fire. Coupling an FTIR spectrometer to a Thermo Gravimetric Analyzer permits an infrared spectrum (a "fingerprint") to be taken of the gas or gasses which are given off at a particular temperature, and therefore to identify them. FTIR has also been coupled to Gas Chromatography so as to enable identification of the separated components of a mixture.

I hope this brief article has given a flavor for the technique of FTIR spectroscopy, some of its applications and it's capabilities. If you have any questions or have a materials problem for which an FTIR analysis may be just the thing you need, please give us a call. At NHML there is always a scientist familiar with your needed application to talk to you.

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