Reflectance Spectroscopy - A Useful Technique for Analysing Solid Samples

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The power of spectroscopy lies in its ability to study materials and environments in a variety of different ways. For example, by applying reflectance techniques, spectrometer users can measure light incident on the surface of a solid material that is reflected at an interface; the light not reflected from the sample is absorbed, scattered or transmitted. This interaction reveals information about the materials comprising the sample.

In this article, we'll review some of the basics of reflectance spectroscopy, describe common system components and practices, and share an example application where reflectance spectroscopy is used for quality control.

Specular and Diffuse Reflectance

Very smooth or shiny, mirror-like surfaces have high specular reflectance, in which the incident light reflects in the same direction *(Figure 1)*. Shiny or glossy objects such as optical components, thin film coatings and certain metal products exhibit specular reflectance.

Light incident on rough or matte surface samples including paper, powders and grains, have diffuse reflectance, where the incident light gets scattered in all directions *(Figure 1)*. Other samples - plastic beads, for example - have both specular and diffuse reflectance. This combination is referred to as total reflectance.

Understanding the difference in reflectance characteristics of the samples to be investigated is important when configuring the optimum spectral system to measure those characteristics.

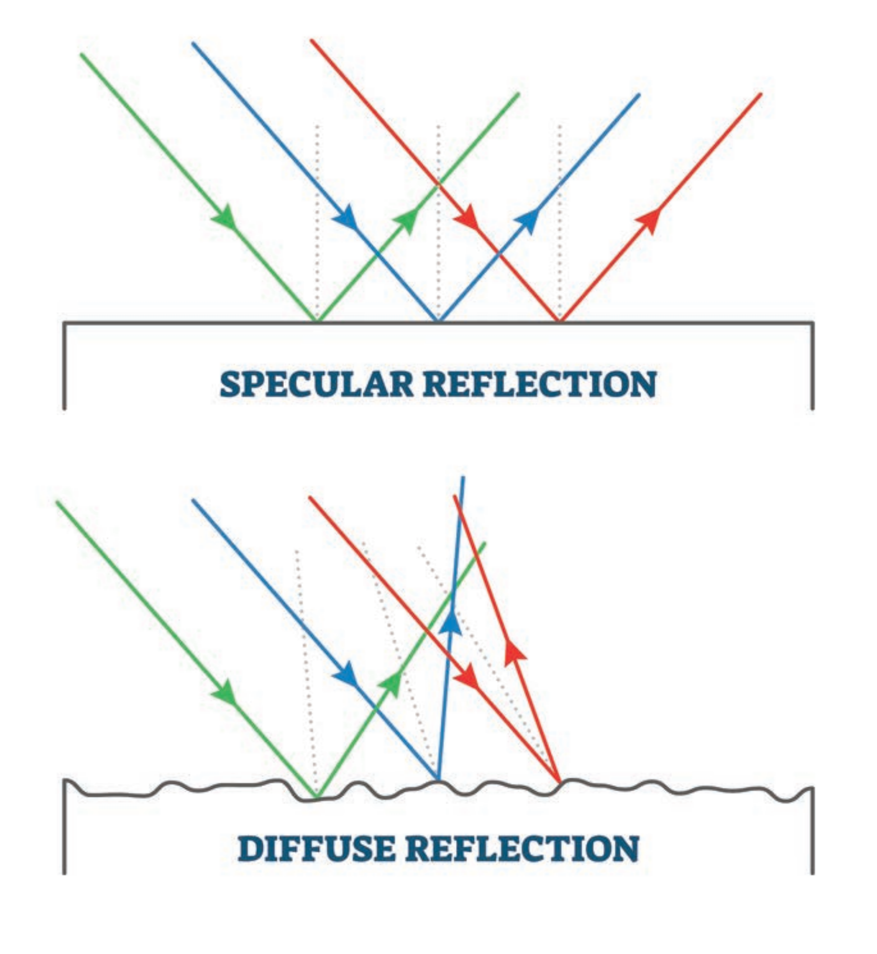

Figure 1. The choice of sampling device in a reflectance measurement setup depends partly on the surface characteristics of the sample, as this illustration demonstrates.

Spectrometer System Setups for Reflectance

Today's modular spectrometer options allow users to mix and match components -spectrometer, light source, sampling optics and more - to optimise reflectance setups for myriad applications *(Figure 2)*. Here are a few criteria to consider:

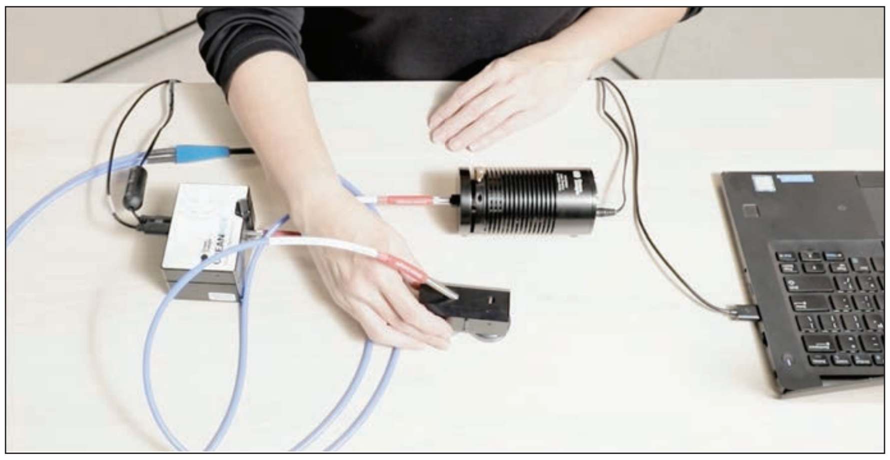

Figure 2. This modular reflectance spectroscopy setup includes a spectrometer, tungsten halogen light source and reflectance probe. Setups like this can be adjusted easily to switch out components depending on measurement needs.

Spectrometers

A general-purpose spectrometer is a good choice for most research applications, although specialty models are available for high-speed applications (sorting components on a process line, for example) and other industrial settings. Also, many miniature spectrometers can be integrated into devices for use in manufacturing as a component, subsystem or complete, automated system.

When selecting a spectrometer, be sure to configure the spectrometer with a detector and grating that matches the wavelength range of interest. Good options are available for UV, visible and NIR reflectance applications.

Light Sources

The most important thing about choosing a light source for reflection is to find one with strong output over the wavelength range of interest. For colour analysis or when making a measurement to mimic the human eye, the light source needs to cover 380-780 nm. For chemical composition of organic material, near-infrared or infrared light will offer more information. Except in a few cases, a narrow light source will not offer enough useful spectral response, so lasers and most LEDs can be ruled out.

For applications in the visible wavelength range, the broad, smoothly varying output of a tungsten halogen light source is ideal for reflectance at visible wavelengths, and for sorting or colour applications. Models that include a high-power bulb and a built-in shutter (handy for taking dark measurements without disturbing your setup) are recommended.

Broader-wavelength light source options are also available. Deuterium-tungsten light sources have a smooth and stable output from 190-2500 nm, and since their output comes from two different bulbs, the UV and visible portions of the spectrum can be used separately. Their output also extends into the NIR, although its intensity decreases at the longer wavelengths.

Regardless of your choice of light source, be sure to warm up the source before taking measurements. Tungsten halogen sources often require 15-20 minutes warm-up time to reach thermal equilibrium, ensuring best results. Also, once the source is warmed up, never turn it off to take a dark measurement; use the shutter on the source instead.

Sampling Optics

The choice of sampling accessory depends on factors including the type of samples to be measured and whether specular, diffuse or both characteristics are desired. Here are some common options:

A reflection probe is great for making quick measurements and for applications where a small spot size needs to be sampled. Reflection probes can measure specular or diffuse reflectance and can be positioned using a probe holder for the optimum angle of incidence. One trade-off is that most reflection probes illuminate and detect from the same direction, so they capture only part of the reflected light. Measurements made with a reflection probe are relative measurements.

An integrating sphere is a good option if the reflectivity of the sample seems to change at different viewing angles. This happens with rough surfaces like brushed metal, grains, and powders. An integrating sphere has a 180° view of the reflected light, giving more accurate (and absolute) reflectance measurements. An integrating sphere can even be used for convex curved surfaces, or to measure the colour of objects that are small enough to fit into the sphere opening.

Integrating spheres have perfectly diffusing interior surfaces. Light enters through a circular input port and is scattered repeatedly by the sphere's inner wall until the light inside the sphere is uniform, regardless of any spatial, angular, or polarisation variations in the input. A fibre placed at 90° to the input port then samples a tiny fraction of the light within the sphere, sending it to the spectrometer. A baffle in front of the fibre port helps block any light rays making their first reflection from the sample port.

Collimating lenses are another reflection sampling option. The lenses can be connected to optical fibres to truly customise the angle of incidence and angle of collection. Specular or diffuse reflectance can be measured this way inexpensively, but much more alignment is needed up front, as is extra fixturing with an optical stage or other accessory. The collimating lenses need to be adjusted carefully to avoid beam divergence and return good signal, making this a more time-consuming sampling method.

Regardless of your choice of sampling optic, be sure to maintain consistent sampling geometry in your setup - i.e., keep the angle and distance from the probe or sphere to the reference and sample the same from measurement to measurement.

Reflectance Standards

Reflectance measurements are a ratio of the reflected light spectrum to the incident light spectrum. Since there is no way to directly collect all of the light incident on a surface, reflectivity is typically measured relative to a reference standard.

Diffuse and specular reflectance standards are widely available, with reflectivity ranging from approximately 88%-98% across UV-Visible-NIR wavelengths. Know that reflectance standards provide absolute measurements only if they come with NIST-traceable calibrated values.

With some diffuse reflectance standards, results are provided only in relative measurements. This means the standard will be nearly equally reflective at all wavelengths, regardless of the angle of collection. Diffuse reflectance standards (Spectralon® and PTFE are common diffusers) are a versatile option for use with probes, lenses or integrating spheres.

If you lose your diffuse reflectance standard or it gets so dirty or damaged that there is no hope of a salvageable reference measurement, do not use a white sheet of paper as a substitute. Paper is not as spectrally white as it appears.

A piece of Styrofoam™ will work much better. It provides diffuse reflection and has relatively even reflectivity across the visible range. Just remember to later measure the Styrofoam against a proper reference standard and correct all your spectra accordingly.

Specular reflectance standards provide a bit more versatility. Options are available for both high-reflectivity samples (very shiny surfaces) and low-reflectivity samples (e.g., anti-reflective coatings, blocking filters and substrates). Specular standards may comprise materials such as glass or an aluminium mirror on a fused glass substrate.

Example Reflectance Application

Ensuring quality in printed circuit boards (PCBs) includes characterising both the specular and diffuse reflectance properties of the black coatings used to absorb light at the board level. This is especially relevant for PCBs in optical instruments, where light reflecting within the instrument can adversely affect system performance.

In theory, a black surface should absorb all wavelengths of light. In reality, there are many variations of black surface coatings with absorbance and reflectance properties that vary as a function of wavelength.

To test this, the reflection spectra for PCBs coated with three types of black paint (flat black, glossy black and black chalkboard paint) were measured using a modular spectrometer (350-1000 nm) with tungsten halogen light source and 400 µm reflection probe.

To account for the relatively low reflectivity of these samples, a grayscale standard was used (~20% diffuse reflectivity) as our reference. Integration times ranged from 20-120 milliseconds, with a boxcar setting of 5 and 20 scans to average. A ring stand supported the reflection probe holder for the measurements and a black hood shielded the setup from direct illumination by ambient light. Measurements were made with the probe arranged at 45° (diffuse reflectance) and 90° (specular reflectance) relative to the coated surface using the reflection probe holder.

The results turned up some unexpected surprises. The specular reflectance data revealed the chalkboard paint to be least reflective, with the glossy black paint ~10x more reflective *(Figure 3)*. The scale may look a little odd (250% reflectivity for the glossy black paint) until we remember that the reference defined as '100%' is actually a 20% diffuse reflectance standard.

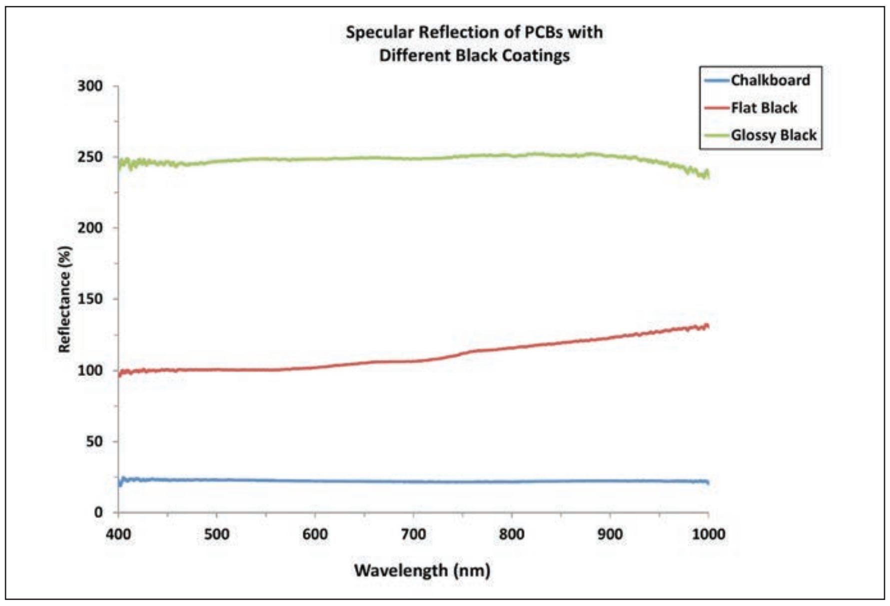

Figure 3. Specular reflectance measurements revealed the PCB with glossy black paint to be most reflective - nearly 10x glossier compared with the chalkboard paint-finished PCB surface.

When examining the diffuse reflectivity data *(Figure 4)*, we observe a similar spectral response in the visible range for the flat and glossy black paints. This suggests these paints would appear very similar in colour to the eye, even if one of the samples is significantly more reflective.

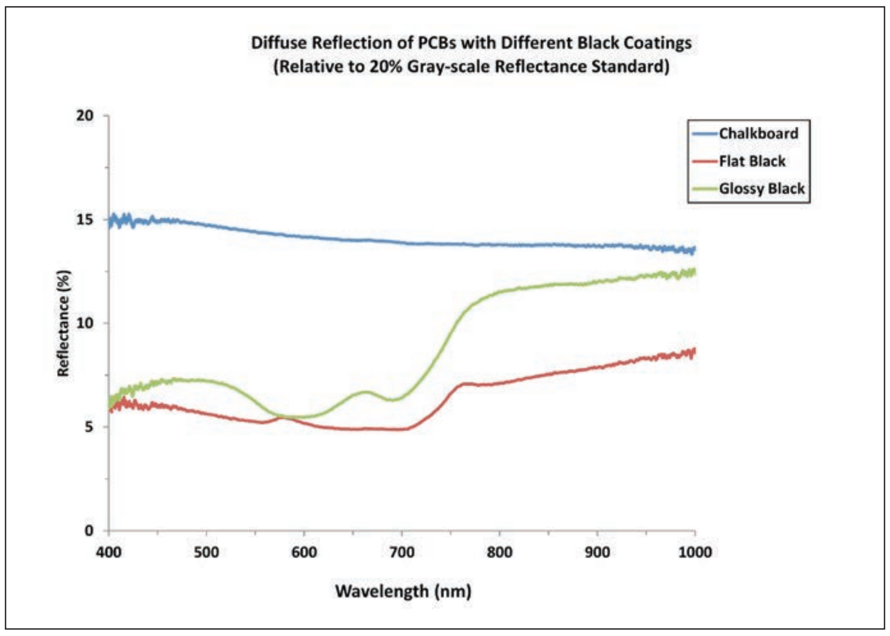

Figure 4. With diffuse reflectance measurements, differences in spectral response for PCBs with different surface coatings become more pronounced at higher wavelengths. Additional analysis could confirm the source of this difference.

The coatings begin to differ in the NIR, suggesting that whatever gives the paint its gloss may be an NIR reflective compound. What is surprising is that the chalkboard paint appears to have a higher diffuse reflectivity than the other black paints. One caveat: The reflection probe is only showing the diffuse reflectivity when illuminated and viewed at 45°.

In this example, a simple modular spectrometer setup measured the diffuse and specular reflectance properties of black coatings on PCBs. The spectral data resulting from these relatively straightforward measurements could be used to select the optimal optical bench coating to reduce stray light and enhance instrument performance.

Because of advances in spectroscopy and its applications, what's exciting about a relatively simple reflection measurement like this is its scalability for similar applications. Indeed, with the versatility of a technique such as reflection spectroscopy, today's modular spectrometer providers can help users across various markets address challenges from research and science applications to industrial-grade and OEM solutions.

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