THE USE OF MICROSCOPICAL METHODS FOR CHARACTERIZATION AND FAILURE/CONTAMINATION ANALYSIS OF ADHESIVE TAPES

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Introduction

A variety of techniques involving light microscopy and small particle isolation and identification are available for the analysis of adhesive tapes. These techniques can be applied to both film and adhesive layers of tapes themselves, as well as to any defects or contaminants which may be present and which may result in an unacceptable appearance and/or performance of the product. Defects in different layers of the polymer film, as well as adhesive distribution can be visualized using oblique, transmitted, episcopic, darkfield or fluorescent illumination, as well as contrast enhancement techniques such as phase contrast or Nomarski differential interference contrast microscopy. Additional characterization of layer thickness and adhesion failure, pigment distribution, and distribution of contaminant particles is possible using microtomed cross sections, cut either by hand or mechanically. Particulate contamination down to micron-sized particles can be readily isolated and identified using polarized light microscopy (PLM), infrared microspectroscopy (IR), Raman spectroscopy, and scanning electron microscopy with energy dispersive x-ray spectroscopy (SEM/EDS).

Characterization of Tape Components

In some cases, a project will require as complete a characterization as possible of the normal components of a tape sample, including film layers, fabric, adhesive and pigments/fillers. This is most often needed in cases of reverse engineering of a competitor's product or in forensic science when, for evidentiary purposes, two or more tape samples must be compared to determine their similarity. Identification of the various components of a tape sample can be readily accomplished even on small amounts of material, using a variety of microanalytical techniques.

Characterization of Polymer Film

If the polymer film backing is known to be a single layer, characterization of the organic portion is easily accomplished by shaving off a thin section with a microscalpel and pressing the sample onto a salt plate for analysis by infrared microspectroscopy. On bulk samples, infrared microspectroscopy by attenuated total reflectance (ATR) can also be useful. If multiple layers are present, they can sometimes be separated by freezing or immersion in a suitable solvent, then analyzed separately. More commonly, a cross-section of the polymer film is cut, either by hand or with a microtome. Figure 1 illustrates a typical section of a multilayer polymer film. In some cases, acceptable hand sections can be cut using a Teflon coated double-edged razor blade under a low power stereomicroscope, however mechanically microtomed sections are generally preferable due to their greater uniformity. The thickness and uniformity of the various layers can readily be determined using a microscope equipped with a calibrated eyepiece reticule. In some cases, a contrast enhancement technique such as phase microscopy must be employed to clearly visualize the borders between various layers. Infrared microspectroscopy can also be performed on the individual layers in a cross-section, provided they are not too thin. If a sample contains layers too thin to be resolved by a single IR analysis, an infrared line map across the entire film thickness can provide chemical information about the thinner layers. Alternatively, a section can be cut at an oblique angle, so that each layer presents a wider aspect. In some case, the analysis of very thin layers can also be accomplished by delamination of the sample, followed by ATR spectroscopy of the appropriate surface. In samples when the polymer film is too heavily filled or
pigmented to allow an adequate infrared analysis (as in the case of black electrical tape or conductive carbon tape, for example), or when absorptions due to pigments and fillers overlap important bands in the IR spectrum of the polymer, the organic portion of the film can be isolated by using a micropyrolysis technique. For this analysis, a small portion of the layer of interest is placed in a glass capillary tube with a drawn out tip. Figure 2 shows a capillary pipette loaded with a black polymer particle a few hundred micrometers in size. The sample is heated briefly over an alcohol flame to pyrolyze the organic component. The pyrolysis product is then rinsed from the tube with a suitable solvent, such as methylene chloride, and spotted onto a salt plate. After evaporation of the solvent, the pyrolysis product can be easily analyzed by IR.

**Characterization of Adhesive**

The organic portion of the adhesive layer is also best analyzed by infrared microspectroscopy, either by bringing an ATR crystal into contact with the adhesive layer, or by removing a small amount of the adhesive with a needle and smearing it out on a salt plate for direct IR analysis in transmission mode. When using the latter technique, it is sometimes helpful to partially dissolve the adhesive in a microdrop of a solvent such as xylene or amyl acetate, and then allow it to dry as a thinner, more uniform film on the salt plate. This is also a useful technique when the adhesive is filled (as with a duct tape, for example), as a microscale extraction can be performed directly on the salt plate to separate the soluble organic portion of the adhesive from the insoluble inorganic portion. Inorganic pigments and fillers such as titanium dioxide, calcium carbonate or aluminosilicate clay can be identified by a combination of light microscopy, infrared microspectroscopy and SEM/EDS.

In some cases, it may be of value to characterize the distribution of the adhesive. In the case of an opaque adhesive or backing, this is easiest with oblique lighting and a stereomicroscope. In cases where neither the polymer film nor the adhesive layer are opaque, transmitted illumination or episcopic illumination (light directed at a 90° angle to the surface) are typically more useful for visualizing the contours of the adhesive surface. Figures 3 and 4 depict the adhesive layers of two samples of duct tape, showing noticeably different adhesive distribution.

**Characterization of Fabric/Fibers**

The fiber or fabric reinforcement present in some tapes can also be easily characterized, using stereomicroscopy and PLM. The number of warp (and if present, weft) yarns per inch can be measured using a calibrated eyepiece scale. Individual yarns and fibers can be isolated from the adhesive with an excess of a suitable solvent and mounted in an oil of known refractive index for PLM analysis. Most synthetic and natural fibers can be quickly identified by PLM alone, based on their morphology and optical properties. The most common types of fibers used in adhesive tapes, (cotton, polyester, glass and rayon) can all be distinguished at a glance by PLM. Photomicrographs of these four fibers are included in Figures 5 through 8. Cotton fibers are immediately identifiable by their morphology, appearing as twisted ribbons, while rayon fibers are distinguished by a series of striated lines running parallel to their length. Polyester appears as straight, normally round fibers that have fairly high refractive indices and which are birefringent, appearing bright between crossed polars. Glass fibers also appear as straight rods, but are not birefringent and will appear dark between crossed polars.

**Analysis of Defects and Contaminants**

In addition to being useful for characterizing the normal components of a tape sample, microscopical and microchemical techniques are also valuable for locating, isolating and identifying contaminant particles or defects in the tape itself. In most cases, these methods are quicker and more
readily applicable to contaminant analysis than the macroscale methods used to analyze bulk tape components.

Location of Defects and Contaminants

Defects in the tape itself will most commonly be present in the polymer backing. Depending on the nature and location of the defect, transmitted illumination (brightfield or between crossed polars), oblique illumination or episcopic illumination may be most useful for visualization and photomicrography. In most cases, a defect will be visible to some degree with more than one illumination method. Figures 9 and 10 illustrate typical defects in the polymer film backing of tape samples, viewed with transmitted light. Figures 11 through 13 show defects in the same brown wrapping tape under three different illumination conditions. While the defects are visible in all three cases, episcopic illumination provides by far the best image. If a defect is located within one of the layers of a transparent multilayer film backing, its depth, and the layer it resides in, can be determined by focusing down from the surface of the film until the defect is in clear focus and measuring the change in distance on the fine focus knob of the microscope. This method (called optical sectioning) is faster and easier than manual cross sectioning, and allows all of a defect to be imaged, rather than just what is visible at the edge of a section. The use of crossed polars (with a polarized light microscope) will highlight areas where a polymer has been placed under stress due to either manufacturing flaws or damage. Stressed areas will be more highly oriented than the surrounding polymer and will display different brightness and colors due to differing birefringence. Figure 14 shows a section of a transparent polymer film with numerous stress-related defects, viewed between crossed polars. In the case of more highly oriented polymer films, crossed polars can help to highlight regions of lower orientation, which may be weaker.

Occasionally, specialized illumination and contrast enhancement techniques may be necessary to adequately visualize defects. Phase contrast and Nomarski differential interference contrast can be used to highlight features which are very similar in color and refractive index to the surrounding polymer and which cannot be easily distinguished with ordinary brightfield illumination. Defects too small to be resolved with ordinary transmitted light can be visualized using transmitted darkfield illumination, which will cause the defects to appear as bright spots against a dark background.

Contaminant particles can usually be located using only a low power stereomicroscope, with magnifications of about 5X-60X, and oblique lighting, although extremely small particles may require the higher magnification of a compound microscope. Rarely, fluorescence microscopy may be useful in highlighting contaminants that fluoresce differently than the surrounding matrix, though such particles are typically also visible with normal illumination as well.

Isolation of Contaminants

Contaminant particles in the adhesive layer can typically be removed using fine forceps and needles, with the aid of a stereomicroscope. Larger particles such as fibers or metal flakes can be pulled out of the adhesive layer directly, then rinsed with solvent on a microscope slide to remove residual adhesive. For the removal of smaller particles, it is often helpful to apply a small drop of solvent to the area in order to partially dissolve the adhesive, after which a small blob of softened adhesive containing the particle(s) of interest can be removed with a needle. This sample can then be pressed out on a glass slide under a cover slip for microscopical examination of the particles, with additional solvent being added to further disperse the adhesive, if necessary. If it is necessary to completely isolate small particles from the adhesive for chemical analysis, a small smear of adhesive containing the particles of
interest can be placed on a microscope slide, IR window or SEM stub, after which small droplets of solvent are successively drawn over the sample with a needle until the adhesive matrix has been cleared.

Contaminant particles at or near the surface of the polymer backing can also be easily removed by direct picking with microtools. Particles that are imbedded within the film can often be excised from the polymer by using a sharpened needle and microscalpel. Alternatively, a small section of the polymer containing the particle of interest can be cut out and cross-sectioned (usually by hand) to expose a portion of the particle for IR or SEM analysis. Figures 15 and 16 depict this technique. For particles that are particularly difficult to isolate from the polymer matrix by physical means, dissolution or melting of the polymer or low temperature ashing may also be useful, depending on the nature of the particles and of the matrix.

Identification of Contaminants

Once contaminant particles have been isolated, a variety of microanalytical techniques can be used for their identification. Light microscopy, using a stereomicroscope and polarized light microscope should be the initial techniques in any scheme of analysis and will in many cases be sufficient to identify unknown particles quickly and unambiguously. Fibers, hairs, pigments, food products, pollen grains, spores, glass, plant tissues, combustion products and minerals are just some of the particle types that are readily identifiable with light microscopy alone. A valuable advantage of PLM is that it allows different types of particles with essentially the same chemical makeup to be easily distinguished. Cotton and rayon fibers, for example, are both composed of cellulose and will give very similar spectra if analyzed by IR or SEM, but are distinguishable at a glance under the microscope. Light microscopy also has the advantage that it can sometimes be used to identify particles without the need to isolate them from a matrix.

If additional chemical information about a sample is desired (in the case of polymeric materials or metals, for example), IR and SEM/EDS are typically employed as the next steps in a system of analysis. SEM/EDS can provide information on the elemental composition of metal alloys, minerals, pigments, glasses and other inorganic materials, while IR provides molecular information about organic materials, and can also be used for limited identification of inorganics. Raman spectroscopy is another useful technique that provides chemical information complementary to IR and which can be used to identify both organic and inorganic materials. Raman spectroscopy has the advantages of being effective on smaller particles than IR, as well as sometimes being able to analyze particles in situ. Raman is also useful for the analysis of materials that are opaque (such as graphite) and for distinguishing different crystalline forms of the same compound (such as the rutile and anatase forms of titanium dioxide).

Conclusion

Light microscopy is an extremely powerful tool that can be applied to many types of particle identification and material characterization problems and should be part of the repertoire of any laboratory involved in addressing industrial issues of product failure or contamination. Its speed and versatility in sample evaluation and identification make it an invaluable tool that, in combination with other microanalytical techniques, can lead to solutions of problems that would otherwise be intractable.
Figure 13. Defects in brown wrapping tape–episcopic light

Figure 14. Defects in polymer film–crossed polars

Figure 15. Isolation of defect in polymer film

Figure 16. Cross sectioning of isolated defect