Guideline for Using Light Microscopy in Forensic Examinations of Tape Components

Scientific Working Group for Materials Analysis (SWGMAT)

1.0 Scope

This document is part of a series of guidelines, prepared by the Scientific Working Group for Materials Analysis (SWGMAT), relating to the forensic analysis of tape and is intended to assist individuals and laboratories that conduct microscopic examinations and comparisons of pressure-sensitive tapes. These methods emphasize the examination and comparison of duct tape (a fabric reinforced tape) and clear polypropylene packing tape (a non-reinforced tape). However, the methods are general and may also be used for other tape types.

2.0 Referenced

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3.0 TERMINOLOGY

Biaxially oriented polypropylene (BOPP): An oriented polypropylene film in which the polymer has been stretched in both the machine direction and cross direction during the manufacturing process. Tapes with such films cannot be torn by hand.

Birefringence: The numerical difference in principle refractive indices for a substance.
Cross direction: The direction of the tape that runs across the width of the tape.

Delusterant: An agent used to alter the light reflected from a fiber causing a dulling effect.

Dispersion staining: A procedure involving central or annular stops in the objective back focal plane to induce colored images of transparent particles mounted in liquids with indices matching the particle at a wavelength in the visible range.

Extinction angle: As it applies to tape, the angle between the machine edge of a clear oriented polymer tape film under crossed polars and the point of extinction (appears dark under crossed polars).

Fill yarns: Yarns in the scrim fabric of reinforced tape that run crosswise, perpendicular to the warp direction. Also called weft yarns.

Machine direction: The direction of the tape that runs the length of the tape.
Monoaxially oriented polypropylene (MOPP): An oriented polypropylene film in which the polymer has been stretched in only one direction during the manufacturing process. Tapes with such films can be torn by hand.

Mounting medium: A liquid, polymer, or resin used to mount specimens for microscopical examinations.

Refractive index: The ratio of the velocity of light in a vacuum to the velocity in some medium.

Retardation: The actual distance of one of the doubly refracted rays behind the other as they emerge from an anisotropic substance. The amount of retardation is dependent on the difference between the two refractive indices and the thickness of the material.

Sign of elongation: A reference to the orientation of the refractive indices in an anisotropic substance as it relates to the elongated direction of a substance. If the slow wave (higher refractive index) is in the elongated direction it has a positive sign of elongation. If the fast wave is in the elongated direction it has a negative sign of elongation.

Warp yarns: Yarns in scrim fabric of reinforced tape that run lengthwise (in the machine direction).

Weft yarns: See fill yarns

4.0 Summary of Guidelines

The following layers of PSA tape will be examined:

- Clear and semi-opaque film backings are examined for optical properties and additives.
- Adhesives are examined for filler components.
- Fibers in fabric-reinforced tape are examined for construction and fiber classification.

Some microscopical examinations of tape involve separation of the layers. After mounting the samples on microscope slides, tapes are compared to determine if they have similar optical properties and morphological features.

5.0 Significance and Use

There is variability in tape films, adhesives, and fibers that can be readily noted with transmitted and polarized light. Some tapes may exhibit microscopic variability that cannot be readily detected in other instrumental or macroscopic examinations. Microscopical examinations of the tape components offer a simple way to assess the similarities and differences between tape products. If differences can be seen by this technique, further tests are not necessary.

6.0 Sample Handling

Other initial examinations, such as physical match, macroscopical/ stereomicroscopical examinations, and the collection of trace evidence such as hair and fibers adhering to the tape, should be completed before proceeding with sample mounting for microscopical examinations. Refer to section 6 of the Physical Characteristics Guideline protocol.

If a questioned tape is submitted in a tangled condition, refer to section 6 of the Physical Characteristics Guideline protocol. Care must be taken with any heat methods to avoid stretching the tape's polymer backing.

7.0 ANALYSIS

Tape samples should be examined first under a stereomicroscope. Areas of the tapes that appear in their original state (i.e., not stretched out of shape and having a clean adhesive area) should be selected for analysis. Fingerprint powders or chemicals should be gently but thoroughly cleaned from the film backing.

7.1 Fabric-Reinforced Tape (Duct tape, Gaffer's tape, Strapping tape)

All three layers of fabric-reinforced tape may be mounted separately for microscopy. The adhesive and reinforcement fibers will have more discriminating microscopic features than the film backing. These methods describe the full examination; however, it is at the discretion of the examiner to choose those methods that are most suitable to the given case. Refer to the SWGMAT *Forensic Fiber Examination Guidelines*, "Microscopy of Textile Fibers" section 7.2 for mounting media recommendations.

7.1.1 Sample Preparation- Areas of the tape free of contamination are selected for analysis. This is best done under a stereomicroscope. Ends should be avoided when cutting samples. The tape is initialed at the site of the cut. The film **backing** is separated from the adhesive and fabric. A suitable solvent (e.g., hexane) may be used if mechanical separation is not feasible. The clean film is mounted in the appropriate medium and cover-slipped.

Microscopical examinations of the **adhesive** are useful only in opaque adhesives. The adhesive is separated from backing by pinching with tweezers and cutting with a scalpel and then transferred to a microscope slide. Care should be taken not to include fibers in this sample. Xylene or a similar solvent may be added to the adhesive sample on the slide to disperse the adhesive's rubber base. After drying, the sample is mounted in a suitable mounting medium and cover-slipped. Most minerals of PSAs can be evaluated in mounting media having refractive indices of 1.66 and 1.55.

Fibers from the scrim fabric can be gently pulled and clipped from the adhesive for mounting. If necessary, the fibers can be rinsed of any adhering adhesive using hexane or other suitable solvent. The warp and fill yarns may be cotton/polyester blends. Therefore, the whole bundle should be loosely mounted on a microscope slide in a mounting medium. Warp and fill yarn fibers are mounted separately.

7.1.2 Microscopical Examination of the Mounted Film Backings

Tape backings with some transparency may be cleaned of adhesive and mounted in a mounting medium. In duct tapes the gray color of polyethylene film backing is due to the presence of aluminum powder. Viewing mounted duct tape films under transmitted light on a comparison microscope may offer some comparative information about the density, size, and dispersion of the aluminum particles in tapes. Note that duct tape backings may be multilayered. A cross section of the duct tape backing should be examined for physical characteristics and chemical composition. In clear and matte backings from strapping tapes and office tapes, additives are looked for and noted in plane polarized and cross polarized light. In addition, clear backings may be suitable for the methods described in section 7.2.

7.1.3 Microscopical Examination of the Mounted Adhesives
The inorganic fillers of PSAs may be examined under transmitted plane and crossed polarized light. Mounting media with refractive indices of 1.66 and 1.55 are suitable for most mineral types that may be found in PSAs. The morphological and optical features of the different inorganic fillers can be noted. These particles are mixed with the elastomer and tackifying resin and include, but are not limited to, kaolinite, calcite, dolomite, rutile, zincite, or talc. Dispersion of adhesive samples first in xylene, as described in section 7.1.1, allows for a better assessment of these fillers. The identity of these minerals may be surmised from their optical properties along with the IR spectra and elemental composition.

7.1.4 Microscopical Examination of the Fiber Reinforcement
Refer to the SWGMAT *Forensic Fiber Examination Guidelines*, "Microscopy of Textile Fibers," for methods of determining the optical properties of the reinforcement fibers of the tape. Using these microscopic methods the following observations should be made separately for the warp and fill fibers:

<u>Fiber class</u> - usually cotton or polyester.
<u>Diameter</u> - of each class of fibers.
<u>Delusterant</u> - either absent, light, medium, or heavy.
<u>Shape</u> - may be round, polygonal, tri-lobal, etc.
<u>Blending</u> - cotton may be blended with polyester.

7.2 Non-Reinforced Tape - Examinations of Oriented Films (Clear Polypropylene Packing Tape)

The methods described in this section are recommended for clear packing tapes; however, they are applicable to other non-reinforced tapes with clear backings. Transparent 1/2" office tapes and some strapping/filament tape may have oriented polymer backings.

The variability in the polymer films in clear packing tape is imparted during the manufacturing process. Controlled heating, cooling and stretching produce films with both amorphous and crystalline areas. Biaxially oriented polypropylene (BOPP) is stretched in two directions with crystalline bundles lining up along the two stretched directions. Monoaxially oriented polypropylene (MOPP) is stretched in one direction. The differences in these two types of packing tapes can be distinguished with polarized light microscopy. Within each of these subclasses of packing tapes, variances may also be noted in the extinction direction with respect to the machine direction of the tape. Differences in interference colors will reveal differences in tape film thickness.

The polypropylene film of packing tapes behaves as an optically biaxial crystal. There are two perpendicular refractive indices in the plane of the film. One runs roughly in the cross direction and the other in the machine direction. The third refractive index runs normal to the plane of the film.

7.2.1 Sample Preparation - Select about an inch of tape that has both machine edges and appears to be in its original state (i.e., has not been damaged by heat, stretching, or contamination). Stick this piece directly onto a clean microscope slide, adhesive side down. An arrow noted on the mounted sample can help keep track of which direction is the machine direction.

There is no need to separate the adhesive from the film for the microscopic examination of clear packing tape. Brown packing tapes with clear film backings and colored adhesive must have the adhesive removed. The cleaned film may be mounted in an appropriate medium for microscopic examinations.

7.2.2 Determination of Polypropylene Film Orientation –
The following polarized light observations presume that the microscope is optimally aligned and illuminated. Refer to the SWGMAT *Forensic Fiber Examination Guidelines*, "Microscopy of Textile Fibers" section 7.3 for further details concerning the determination of optical properties referred to in the following discussions.

The surface of the clear packing tape sample is brought into focus in transmitted light at about a 100X magnification. The polars are crossed, and the extinction position is found. The stage is rotated just off extinction, and patterns are observed in the film. These patterns may be sharpened by refocusing and closing down the aperture diaphragm. A pattern of "X"s is seen in biaxially oriented tapes (BOPP) and shows the bi-directional stretching in the production process. The pattern seen in monoaxially oriented tapes (MOPP) shows the one direction of stretching. Its pattern may be hazy and show more than one interference color that streak in the one direction of the stretch.

The angles of the crosshatches in the BOPP tape pattern described above may vary from one tape film to another but will be consistent throughout a roll of tape. These angles can be determined with an appropriate eyepiece reticule.

7.2.3 Determination of the Extinction Angle Relative to the Machine Direction
The machine direction of the tape relative to the extinction direction may vary from 0 to 15 degrees between different tapes.

The surface of the tape is brought in focus in transmitted light. One of the machine edges of the tape is aligned with the vertical line of the eyepiece graticule. The stage position is noted in degrees. The polars are crossed and the stage is rotated until the tape film is at its nearest full extinction. The stage position is again noted. The difference in degrees is the extinction angle relative to the machine edge. Tape samples from the same roll will show similar extinction angles.

7.2.4 Determination of the Retardation **-** The thickness of the polymer film in clear packing tapes can vary within manufacturers. When the birefringence of the films of the different packing tapes is the same, the variance in the interference color of the films will be a function of the thickness. Slight differences in thickness will show noticeably different interference colors. These interference colors depend only on the tape film thickness, not the total tape thickness (film + adhesive). The clear adhesive layer is isotropic and does not contribute to the interference colors.

Using approximately 400X magnification, the surface of the tape is brought into focus and the polars are crossed. The interference color is noted with the stage rotated to maximum brightness (close to 45 degrees). The fast wave (lower refractive index) is found with one refractive index running roughly across the tape and the other running roughly lengthwise along the tape. The fast wave is aligned parallel to the slow wave of a quartz wedge or Berek compensator. The point of compensation is found and from this, the retardation can be calculated. The experience of the microscopist may dictate the easiest way to arrive at the retardation value.

The birefringence of the polypropylene tape film (BOPP) in the plane of the tape (the difference between the refractive indices of the machine and cross directions of the tape) has been reported in the range of 0.014 - 0.016. (Rappe 1991)

7.2.5 Other Observations - Some clear tape films may have additives that may be visible in transmitted or polarized light and their presence is useful for comparison purposes between tapes. Some assessment of their optical properties should be noted: size, distribution, relative interference colors, etc.

Irregularities in the thickness of the tape film may be observed under crossed polars as multiple interference colors in any given field.

Some tape films may not totally extinguish, or they may show undulose extinction (i.e., areas of lightness and darkness).

7.3 Microscopy of other Tape Classes - Any tape class that has inorganic fillers in the adhesive or backing, reinforcement fibers, or clear or semi-opaque film backings may lend itself to examinations described in these guidelines.

8.0 REPORT DOCUMENTATION

The examiner's notes should contain all descriptions, diagrams, photographs, and calculations that reflect the mcroscopical observations. These observations are only a part of the overall analysis of the tape samples that includes other macroscopical and instrumental exams. For comparative tape examinations, if significant differences are observed in microscopical characteristics, no further testing is necessary, and a report can be issued. If no significant differences are observed, further instrumental examinations should be performed before a report is issued.

9.0 REFERENCES

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